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# *In-Situ/Operando<sup>57</sup>Fe Mössbauer Spectroscopic Technique and* Its Applications in NiFe-based Electrocatalysts for Oxygen Evolution Reaction

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# *In-Situ/Operando<sup>57</sup>Fe Mössbauer Spectroscopic Technique and Its Applications* in NiFe-based Electrocatalysts for Oxygen Evolution Reaction

# Authors

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DOI: 10.132083j.electrochem.210854<br> **Illip://electrochem.xmu.edu.cn**<br> **Cample 1. Rykov**<sup>1</sup>, **Concept Expect Concept and Different Concept** ed 化 学<br>
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, Jun-Hu Wang<sup>1\*</sup><br> *dian Institute of Chemical*<br> *titute of Photoelectronic*<br> *Devices and Technology of*<br> *electronic Technology*, (1. Center for Advanced Missbauer Spectroscopy, Missbauer Spectroscopy (1. Center, Data Center, Name)<br>
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**Absorption of Absorption Cost-Example Cost-Example Cost-effective and The development of Republications in NiFe-based Electrocatalysts for**  $Oxygen$  **Evolution Reaction Jafar Hussain Shah<sup>1</sup>, Qi-Xian Xie<sup>2</sup>, Zhi-Chong Kuang<sup>1</sup> In-Situ/Operando<sup>57</sup>Fe Mössbauer Spectroscopic Technique and<br>
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Jafar Hussain Shah<sup>1</sup>, Qi-Xian Xie<sup>2</sup>, Zhi-Chong Kuang<sup>1</sup>, Ri-Le Ge<sup>1</sup>, Wen-Hu THE SET ASSET AS fts Applications in NiFe-based Electrocatalysts for**<br>**Cypy Concert C** development of in鄄situ/operando characterizations is urgently required to detect key intermediates along with active sites and phases **EXAMPED CONTINUITY NEACTION**<br>
Data Flussain Shah<sup>1</sup>, Qi-Xian Xie<sup>2</sup>, Zhi-Chong Kuang<sup>1</sup>, Ri-Le Ge', Wen-Hui Zhou',<br> *Conter for Adeoneed Massbauer Spectroscopy, Mossbauer Effects Data Center, Dalian Institute of Chemical* Jafar Hussain Shah<sup>1</sup>, Qi-Xian Xie<sup>2</sup>, Zhi-Chong Kuang<sup>1</sup>, Ri-Le Ge<sup>1</sup>, Wen-Hui Zhou<sup>1</sup>,<br>Duo-Rong Liu<sup>1</sup>, Alexandre I. Rykov<sup>1</sup>, Xu-Ning Li<sup>1</sup>, Jing-Shan Luo<sup>2</sup>, Jun-Hu Wang<sup>2</sup><br>(*I. Center for Advanced Mössbauer Spectrosc* Jafar Hussain Shah<sup>1</sup>, Qi-Xian Xie<sup>3</sup>, Zhi-Chong Kuang<sup>1</sup>, Ri-Le Ge<sup>1</sup>, Wen-Hui Zhou<sup>1</sup>,<br>
Duo. Cone Cong Liu<sup>1</sup>, Alexandre I. Rykov<sup>1</sup>, Xu-Ning Li<sup>1</sup>, Iing-Shan Luo<sup>1</sup>, Jun-Hill Wang<sup>1</sup><br>
(*I. Genter for Adeoneed Mashemer* Duo-Rong Liu<sup>1</sup>, Alexandre I. Rykov<sup>1</sup>, Xu-Ning Li<sup>1</sup>, Jing-Shan Luo<sup>2</sup>, Jun-Hu Wang<sup>1</sup><br>(1. Center for Advanced Mössbuser Spectracopy, Mössbuser (Sfleet Data Center, Dolina Institute of Chemical<br>This current status of cat (*I. Center for Advanced Mössbauer Spectroscopy*, Mössbauer-Effect Data Center, Dalian Institute of Chemical Physics, Chinese Academy of Sciences, Dalian 116023, Liaoning, China; 2. Institute of Photoelectronic Thin Film *Physics, Chinese Academy of Sciences, Dairan 116023, Liconing, Chinaz 2. Institute of Photoelectronic Thin Film Detices and Technology, Key Laboratory of Photoelectronic Thin Film Detices and Technology, Taujin, Ministry* Thy and Determining of electrochemic process, assume the other analogues), as a typical model study of Praise for the OER electrocatalyst and Technology of<br>Tanjin, Ministry of Education Engineering Research Center of Thin Fram Fuences and retentioning, is Pranounally of productional transmit Fund Detection Transmit  $T_{G0}$  (Total Thin Thin Photoshectronic Technology,<br>Transm, Ministry of Education Engineering Research Center of Thin Film Ph Reneuvable Energy Conversion and Morage Center, Nankai University, Tranjin 300350, China)<br> **Abstract:** The development of highly efficient and east-effective electrocatalysts for the sluggish oxygen evolution reaction<br>
OC **Abstract:** The development of highly efficient and cost-effective electrocentalysts for the singuish oxygen evolution reaction<br>(OER) remains a significant haring to establish effective utilization of frenewable energy st Examples are experient the control of the state of the control of the state of the state of the better under the state of the better well expansion that the Mission of Mission and the Mission of the Mission and the Missio eyencow or other is considered to the consideration of the subsect of the consideration is the consideration in the mg the relationship between catatytic actroy and the coordinator structure of catatyts. In this tuberature rest tuber tuberature activation is interesting the relation to introduce in detail the knowhow about the turnet st ECs) have been proven as excellent and remarkable candidates for this purpose. But it is critically important to understand the Cs) strate interviewer their decades that influence their activity and underlying necelaation factors that influence their activity and underlying mechanism for the development of state-of-the-art OER catalysts. Therefore, the<br>developmental of *actitogromatio* characterizations is urgently required to detect key i development of *in-sinulay ensuale* draneaterizations is urgently required to detect key intermediates along with active sites and phases<br>responsible for OER. <sup>D</sup>Fe Missobure spectroscopy is one of the appropriate and suit of renewable energy storage systems and water splitting to pro-<br>ER catalysts shows that NiFe-based oxygen evolution catalysts<br>or this purpose. But it is critically important to understand the<br>development of state-of-the-ar Ex causives shows unt Nire-based oxygen evolution cataysts<br>of this purpose. But it is critically important to understand the<br>dedevelopment of state-of-the-art OER catalysts. Therefore, the<br>d to detect key intermediates al able development of state-of-the-art OER catalysts. Therefore, the electroment and development of state-of-the-art OER catalysts. Therefore, the d to detect key intermediates along with active sites and phases sopriate an d to detect key intermediates along with active sites and phases<br>opriate and suitable techniques for determining the phase struc-<br>tifying the active sites, clarifying the catalytic mechanisms, and<br>dination structure of ca

**1 Introduction** solar, wind, and tidal energies)<sup>[1]</sup>. However, owing to

 $\Phi$ <sup>*(k*2)</sup>*E (k*) *Electrochem.*) 2022, 28(3), 2108541 (2 of 31)<br>
ting devices, rechargeable metal-air batteries, polymer formation of an oxygen-oxygen (O-O) bond in which<br>
electrolyte membrane fuel cells (PEMFCs) and the positive electrode (air)<sup>22</sup>. The controllange is the condition of the electrodemic science of electrodesis (OECS), using a non-noble media and water splitting electrodemic for electrode (air)<sup>22</sup>. Therefore, the same **EVALUATION (EXAMPLE 1990)**<br> **EXAMPLE 1990**<br> **EXAMPLE 1990 EXAMPLE 1990 EXAMPLE 1990 EXAMPLE 1990 EXAMPLE 1990 CO2 EXAMPLE 1990 CO2 EXAMPLE 1990 CO2 EXAMPLE 1991 EXAMPLE 1991 CO2 EXAMPLE 1991 E Example 12**<br> **Example 12**<br> **Example as the anomal in the anomal in the anomal in the anomal in the co-bettoomly control and the one with the highest activation energy.<br>
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reduction ting devices, rechargeable metal-air batteries, polymer<br>
clectrochy membrane fuel cells (*PEMF*Cs) and CO<sub>2</sub> canh electrochy email contains a several kinds of novel emergy conver-<br>
electrochemic med cells (*PEMFCs*) and CO electrolyte membrane fuel cells (PEMFCs) and CO<sub>2</sub> each elementary step has its own activation energy.<br>
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And the one with the highest setivation energy is the<br>
reduction reduction are several kinds of novel energy conver-<br>
And the one with the highest activation energy is the<br>
sion and storage technologies<sup>1541</sup>. Foren though the ar-<br>
solver elementary step, making it the rate-dere-<br>
elect sion and storage technologies<sup>2:9</sup>. Even though the ar-<br>
sion-set dementary step, making it the rate-deter-<br>
chirecture of these devices differs, the fundamental miming step in OER<sup>(12)</sup>. Therefore, OER dominates<br>
enheari chiecture of these devices differs, the fundamental mining step in OFR<sup>131</sup>. Therefore, OFR dominates<br>processors are almost the same, In a rechargeable met-<br>**therefore defined in the compression compression of the compres** processes are almost the same. In a rechargeable met-<br>
al-air battre are overall efficiency of the two technologies be-<br>
al-air battre), metal dissolution and deposition on the cause it pertains to the ecation of O-O bonds al-air battery, metal dissolution and deposition on the cause it pertains to the creation of O-O bonds and in-<br>negative cletredot (metal), while the oxygen reduction cludes four proton-coupled electron transfer proces-<br>ne negative electrode (metal), while the oxygen reduction cludes four proton-coupled electron transier process-<br>eaction (ORR) are expaired by ease reading the protocolarity as reading highly OFCs are required to accel-<br>eact on the positive clectrode (air)<sup>19</sup>. For the electrochemi-<br>carte the OER or decrease the overpotential to miti-<br>calc Co, reduction reaction (CO,RR), the OER process aget the energy loss inherent in energy conversion<br>for t eal CO<sub>2</sub> reduction reaction (CO<sub>3</sub>RR), the OER process gate the energy loss inherent in energy conversion<br>can serve as the ander reaction<sup>9</sup>. An umnet challenge technologies.<br>
for this teaction is the lack of efficient o ean serve as the anode reaction<sup>71</sup>. An unmet challenge technologies.<br>
for his reaction is the lack of efficient oxygen coul-<br>  $\frac{1}{2}$  and  $\frac{1}{2}$  and  $\frac{1}{2}$  and  $\frac{1}{2}$  and  $\frac{1}{2}$  and  $\frac{1}{2}$  and  $\frac{1}{2}$  for this reaction is the lack of efficient oxygen evolu-<br>
The innovative combination of materials science<br>
electrocatalyst (OPES), using a non-holb media and water splitting electrochemical year to mix the CO<sub>S</sub>R process tion electrocatalysts (OECs), using a non-noble metal<br>electrochemistry opens new pathe-<br>electrocatalyst to pair with the CO<sub>S</sub>R process for ways for eventage different OECs. Iroy and RuO<sub>2</sub> are<br>high overall energy convers

the the technologies<sup>126</sup>. Electrochem.) 2022, 28(3), 2108541 (2 of 31)<br>ting devices, rechargeable metal-air batteries, polymer formation of an oxygen-oxygen (O-O) bond in which<br>electrolyte membrane fuel cells (PEMFCs) an th(*k<sup>\\*</sup>*(*J. Electrochem.*) 2022, 28(3), 2108541 (2 of 31)<br>
ting devices, rechargeable metal-air batteries, polymer formation of an oxygen-oxygen (O-O) bond in which<br>
electrolyte membrane fuel cells (PEMFCs) and CO<sub>2</sub>  $# \&L^{\omega}(L \&Electrochem.) 2022, 28(3), 2108541 (2 of 31)$ <br>
ting devices, rechargeable metal-air batteries, polymer formation of an oxygen-oxygen (O-O) bond in which<br>
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electrolyte membrane fuel cells (PEMFCs) and CO<sub>2</sub> each elementary step has its own activation energy.  
reduction are several kinds of novel energy conver- And the one with the highest activation energy is the  
sion and storage technologies<sup>[2-6]</sup>. Even though the ar-  
shortective of these devices differs, the fundamental  
mining step in OER<sup>[12]</sup>. Therefore, OER dominates  
processes are almost the same. In a rechargeable met-  
ale-air battery, metal dissolution and deposition on the  
cause$ **EVALUATE (Example 19.13)**<br> **EVALUATE EXECUTE EXECUTE CONSTANT (2013)**<br> **EXECUTE EXECUTE CONSTANT (2013)**<br> **EXECUTE EXECUTE CONSTANT (2013)**<br> **EXECUTE EXECUTE CONSTANT (2014)**<br> **EXECUTE CONSTANT (2014)**<br> **EXECUTE CONSTANT EVALUATION**<br> **EXALUAT EXALUAT THE SECT AND AND THE SECT AND AND THE SECT AND THE SAME SIGN AND THE SAME SIGN AND THE SAME SIGN AND THE SAME SA EVALUAT EXAM (FOLT ALCONS**) and  $\mathbb{R}^{(k+1)}(L)$  *Flectrochem.*) 2022, 28(3), 2108541 (2 of 31)<br>ting devices, rechargeable metal-air batteries, polymer formation of an oxygen-oxygen (O-O) bond in which<br>electrolyte membr **EVALUAT EXAMORE (META)**,  $\mathcal{H}_2(\mathcal{F}_2(\mathcal{F}_3), \mathcal{F}_3(\mathcal{F}_3), 2108541 \text{ (2 of 31)}$ <br>
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electrolyte membrane fuel cel 28(3), 2108541 (2 of 31)<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making it the r 28(3), 2108541 (2 of 31)<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making it the r  $28(3)$ ,  $2108541$  ( $2$  of  $31$ )<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making 28(3), 2108541 (2 of 31)<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making it the r 28(3), 2108541 (2 of 31)<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making it the r 28(3), 2108541 (2 of 31)<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making it the r 28(3), 2108541 (2 of 31)<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making it the r  $28(3)$ ,  $2108541$  ( $2$  of  $31$ )<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making  $28(3)$ ,  $2108541$  ( $2$  of  $31$ )<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making 28(3), 2108541 (2 of 31)<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making it the r 28(3), 2108541 (2 of 31)<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making it the r technologies. (3), 2108541 (2 of 31)<br>mation of an oxygen-oxygen (O-O) bond in which<br>the lementary step has its own activation energy.<br>d the one with the highest activation energy is the<br>west elementary step, making it the rate-deter-<br>i 28(3), 2108541 (2 of 31)<br>
formation of an oxygen-oxygen (O-O) bond in which<br>
each elementary step has its own activation energy.<br>
And the one with the highest activation energy is the<br>
slowest elementary step, making it t 28(3), 2108541 (2 of 31)<br>formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making it the r Excess the straight of the straight of the means of the means and the mean alternative strate and the one with the highest activation energy is the slowest elementary step, making it the rate-determining step in OER<sup>[12]</sup> formation of an oxygen-oxygen (O-O) bond in which<br>each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making it the rate-deter-<br>mining step in each elementary step has its own activation energy.<br>And the one with the highest activation energy is the<br>slowest elementary step, making it the rate-deter-<br>mining step in OER<sup>[12]</sup>. Therefore, OER dominates<br>the overall e 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (2 of 31)<br>
-air batteries, polymer formation of an oxygen-oxygen (O-O) bond in which<br>
s (PEMFCs) and CO<sub>2</sub> each elementary step has its own activation energy.<br>
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And the one with the highest activation energy is the<br>slowest elementary step, making it the rate-deter-<br>mining step in OER<sup>[12]</sup>. Therefore, OER dominates<br>the overall efficiency of the two technologies be-<br>cause it perta slowest elementary step, making it the rate-deter-<br>mining step in OER<sup>[12]</sup>. Therefore, OER dominates<br>the overall efficiency of the two technologies be-<br>cause it pertains to the creation of O-O bonds and in-<br>cludes four p mining step in OER<sup>[12]</sup>. Therefore, OER dominates<br>the overall efficiency of the two technologies be-<br>cause it pertains to the creation of O-O bonds and in-<br>cludes four proton-coupled electron transfer process-<br>es<sup>[13-14]</sup> the overall efficiency of the two technologies be-<br>cause it pertains to the creation of O-O bonds and in-<br>cludes four proton-coupled electron transfer process-<br>es<sup>[13-14]</sup>. As a result, highly OECs are required to accel-<br>e cause it pertains to the creation of O-O bonds and in-<br>cludes four proton-coupled electron transfer process-<br>es<sup>[13-14]</sup>. As a result, highly OECs are required to accel-<br>erate the OER or decrease the overpotential to miticludes four proton-coupled electron transfer process-<br>
es<sup>[13-14]</sup>. As a result, highly OECs are required to accel-<br>
erate the OER or decrease the overpotential to miti-<br>
gate the energy loss inherent in energy conversion<br>  $es^{[13\text{-}14]}$ . As a result, highly OECs are required to accelerate the OER or decrease the overpotential to mitigate the energy loss inherent in energy conversion technologies.<br>The innovative combination of materials sci erate the OER or decrease the overpotential to mitigate the energy loss inherent in energy conversion technologies.<br>The innovative combination of materials science and water splitting electrochemistry opens new pathways f gate the energy loss inherent in energy conversion<br>technologies.<br>The innovative combination of materials science<br>and water splitting electrochemistry opens new path-<br>ways for creating different OECs. IrO<sub>2</sub> and RuO<sub>2</sub> are<br> technologies.<br>
The innovative combination of materials science<br>
and water splitting electrochemistry opens new path-<br>
ways for creating different OECs. IrO<sub>2</sub> and RuO<sub>2</sub> are<br>
considered as benchmark catalysts due to their The innovative combination of materials science<br>and water splitting electrochemistry opens new path-<br>ways for creating different OECs. IrO<sub>2</sub> and RuO<sub>2</sub> are<br>considered as benchmark catalysts due to their low<br>onset potenti and water splitting electrochemistry opens new path-<br>ways for creating different OECs. IrO<sub>2</sub> and RuO<sub>2</sub> are<br>considered as benchmark catalysts due to their low<br>onset potentials for triggering the OER. However,<br>small reser  $\mathbf{I}$ ,



E(*Electrochem.*) 2022, 28(3), 2108541 (3 of 31)<br>
metal carbides<sup>[23]</sup>, and organometallics<sup>[24]</sup>. According processes by which Fe improves the OER activity of<br>
to recent studies, all non-precious metal catalysts Ni-based  $\exists$  the  $\mathbb{R}^{\omega}$  (*L* Electrochem.) 2022, 28(3), 2108541 (3 of 31)<br> **The recent studies, all non-precious metal catalysts**<br>
to recent studies, all non-precious metal catalysts<br>
containing anions other than oxides and  $\#E\#(J. Electron.)$  2022, 28(3), 2108541 (3 of 31)<br>
metal carbides<sup>[23]</sup>, and organometallics<sup>[24]</sup>. According processes by which Fe improves the OER activity of<br>
to recent studies, all non-precious metal catalysts<br>
containing a  $\frac{dE}{dt}$  *Electrochem.*) 2022, 28(3), 2108541 (3 of 31)<br>
metal carbides<sup>[23]</sup>, and organometallies<sup>[24]</sup>. According processes by which Fe improves the OER activity of<br>
to recent studies, all non-precious metal catalysts  $\text{H}(k\#(J, Electrochem.) 2022, 28(3), 2108541 (3 of 31)$ <br>
metal carbides<sup>120</sup>, and organometalies<sup>[24]</sup>. According<br>
to recent studies, all non-precious metal catalysts Ni-based OECs.<br>
containing anions other than oxides and hydroxide contact with the alkaline electrolyte[25,26]. As a result,  $\pm \frac{k^2\gamma}{L}$ . *Electrochem.*) 2022, 28(3), 2108541 (3 of 31)<br>
metal carbides<sup>121</sup>, and organometallies<sup>134</sup>. According processes by which Fe improves the OER activity of<br>
to recent studies, all non-precious metal cataly the external capacity content in the materials in alkaline chectrolytes<sup>(23)</sup>. According processes by which Fe improves the OER activity of<br>to recent studies, all non-precious metal catalysts Ni-based OECs.<br>containing ani **EVALUATION THE REAL CONDUM**<br>
WE THE REAL CONDINE TO SET AND THE SERVICE THE REAL CONDINSTRATION THE SERVICE THE SERVICE THE SERVICE THE CONDUCT SOMETHING A CONDINENTIES and organometrical reachemical real-time constraini **EATAL EXECTS** (*LECTS)* (*LECTS)* (*RECTS)* (*RECTS)* (*RECTS)* (*RECTS)* (*RECTS)* (*RECTS)* (*RECTS)* (*RETS)* **Example 19.1**<br> **Hydroxides and the most potentiallies**<sup>[13]</sup>. According processes by which Fe improves the OER activity of<br>
to recent studies, all non-precious metal eatalysts Ni-based OECs.<br>
Examplement than oxides and  $\frac{1}{2}$  ( $\frac{1}{2}$   $\frac{1}{2}$   $\frac{1}{2}$   $\frac{1}{2}$   $\frac{1}{2}$  and organometallies <sup>[23]</sup>. According **processes by which Fe improves the OER activity of to terest studies, all non-precious metal catalysts Ni-based OECs.<br>
co**  $4k^2$  are  $4k^2$  are  $4k^2$  are  $4k^2$  and organometallies<sup>191</sup>. According processes by which Fe improves the OER activity of<br>to recent studies, all non-precious metal catalysts Ni-bosed OECs.<br>containing anions other tha have shown higher OER activity[28] .

# trocatalyst

mental viese entrowares when the mean one contains when the contained the contained the contains of the particles and **E** in the second in the interpretive hydroxides (was the second the containing of **Fe** Role in Ni-Base **EVENDED THEST (THE STEP INTERTMENT THEST (THE STEP) EXECT<br>
<b>EXECT PROPERS AND UNIONEMISE MANEWERS** matrices makes them hards to be oxi-<br> **ing of Fe Role in Ni-Based Elec-** dized dirther and thus alters the redox electroc **2 Recent Progress and Understand**—<br>
ing of **Fe Role in Ni-Based Elec-** dized further and thus alters the rotox detectrochem-<br> **tractations** tractations is the mass have start and thus alters the rotox detectrolen-<br> **thro ing of Fe Role in Ni-Based Elec-**<br> **into the Ni-Based Electrochem-**<br> **into the Ni(OH)5NiOOH** is the cell volt-based both  $\sim$  Ni(OH)4NiOOH is the since of the incorporated into Ni-based<br>
The most aciree electrocatalyst f **EVALUAL EXECT THE SET THE SE** The most active electroceatalyst for OER was  $\overline{OECs}$ , it causes structural changes, 3) It is well-known<br>thought to be Ni(OH)-NiOOH at the start of the evo-<br>also based Lewis axiel with a strongest ransition metal-<br>hutio thought to be Ni(OH)<sub>/</sub>NiOOH at the start of the evo-<br>
fact that Fe<sup>1</sup> serves as the strongest transition met-<br>
lution of 3d transition metals-based OECs. Subbara-<br>
als-based Levis sciels with a stronger clearephile na-<br> altion of 3d transition metals-based OECs. Subbara<br>man and co-workers suggested that the OER activity<br>man and co-workers suggested that the OER activity<br>three and notably affects the electronic preperties of<br>the order of man and co-workers suggested that the OFR activity<br>
ture and notably affects the electronic properties of<br>
tred of 3d transition metal divalent cations goes in<br>
the other cations in the other cations in the other cations trend of 3d transition metal divalent cations goes in<br>the other cations in which being incorporated; hence<br>the order of Mm<sup>2</sup>  $\leq$  Fe<sup>3</sup>  $\leq$  Co<sup>2</sup>  $\ltimes$  Ni<sup>2</sup><sup>1191</sup>, And it was<br>mation of active with sixes and als in th the order of Mn<sup>2+</sup>  $\ll$  Fe<sup>2+</sup>  $\ll$  Co<sup>2+</sup>  $\ll$  Ni<sup>2+120</sup>). And it was it changes the Ni oxidation states and aids in the for-<br>believed to be true and accepted widely until it was mation of active Ni<sup>4</sup> sizes, and 4) Fe<sup></sup> believed to be true and accepted widely until it was<br>
fround that a trace amount of Fe ( $\sim$  1 ppm) largely on-<br>
hife-based OEES with a 1:0.33 ratio dependent<br>
harened the OER activity of Ni<sup>+</sup> based electrocation-<br>
hyste found that a trace amount of Fe  $(-1$  ppm) largely en-<br>hanced OFCs with a 1:0.33 ratio depicts a su-<br>hanced the OER activity of Ni<sup>2+</sup> based electrocata-<br>percechange magnetic contact that improves OER<br>bytsi<sup>28:31</sup>. In the hanced the OER activity of Ni<sup>2</sup> based electrocata-<br>perexchange magnetic contact that improves OER<br>hysts<sup>2018</sup>. In the late 1980s, the extraordinary OER per-<br>clertotroatally is by allowing electrons to hop.<br>Tomaces of Ni(

电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (3 of 31)<br>
etallics<sup>[24]</sup>. According processes by which Fe improves the OER activity of<br>
rivisions metal catalysts Ni-based OECs.<br>
stides and hydroxides Without a doubt, Fe inco

the  $(2\frac{m}{2}$  ( $L$   $R$  lectrochem.) 2022, 28(3), 2108541 (3 of 31)<br>metal carbides<sup>(21)</sup>, and organometallies(2<sup>31</sup>. According processes by which Fe improves the OER activity of<br>to recent studies, all non-precious metal metal carbides<sup>1911</sup>, and organometallies<sup>191</sup>, According processes by which Fe improves the OER activity of<br>to recent studies, all non-precious metal eachlysts Ni-based OECs.<br>containing anions other than oxides and hydro al carbides<sup>[23]</sup>, and organometallies<sup>[23]</sup>. According processes by which Fe improves the OER activity of<br>cent studies, all non-process metal catalysts Ni-based OECs.<br>
studies anime amons other than oxides and hydroxides The most control of the most activity of the most actual to the significant of the significant of the significant of the most activities were<br>progression in the significant of the significant of the most activities were<br>p the production of suitable construction, resulting in any in order behind the significant OEK improvements are the evo-<br>the production of oxides/hydroxides on the surface in metric can be a first of DER activities were<br>co the production of oxtates-based and subsects of the state in meth sense to the state in the contact virus auxine electrocopy<sup>-----</sup>. As a result, grade KOH electrocity so int. OER suggested to be the materials without alkaline circumstances, their (oxy)hydroxides are all-<br>regardless of the materials without alka regardless of the maleral surface of the metal surface of the metal surface of the metal surface in the metal surface of the station metal (oxylyhydroxides metals). Nife-(oxy) and surface transition metal (oxylyhydroxides EVALUAT THE CONDIVIDENT CONDUCT THE TRANSPORTED THANGE THE AND THANGE SIMPLE THANGE THANGE THANGE THAN was the reaction entails downly the reaction of the control in the state of the state of Nicolar states of Nicolar States of Technomical proportion of the true is and the state of Nicolar States of Te in the matrix of Nitransition metallooxynythooties muchanis, Nire-book Dick, Based Office, Hassed Office, Hassed Offices and the finding in the matrix of Ni-based Offices and the matrix of Ni-based of the most physical state in the matrix o pyroxides have been proved to be the measl potential is seen published in literatures detailing the electrocatalysts, while their potents are not computed in Figure 2012 (CFCs, important roles stated in literatures with e materials makes in adaptation stration of Fe in the matrix of Ni-based<br>
studies, pure Ni and Pe oxides/hydroxides are not cf-<br>
DECs. Important roles stated in literatures with ex-<br>
frient OER electrocatalysts, while their students, pure Ni anore occuses provides are note.<br>
Figure Ni anore occuses provident OER electrocatalyses, while their complexes<br>
From place of Ni in their respective hydroxides (oxy)<br> **2 Recent Progress and Understand** (28(3), 2108541 (3 of 31)<br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in u (3), 2108541 (3 of 31)<br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in unpu (3), 2108541 (3 of 31)<br>
vecesses by which Fe improves the OER activity of<br>
based OECs.<br>
Without a doubt, Fe incorporation has been the<br>
ving force behind the significant OER improve-<br>
nt seen with Ni-based OECs in unpurifi 28(3), 2108541 (3 of 31)<br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in un 28(3), 2108541 (3 of 31)<br>
processes by which Fe improves the OER activity of<br>
Ni-based OECs.<br>
Without a doubt, Fe incorporation has been the<br>
driving force behind the significant OER improve-<br>
ment seen with Ni-based OECs (28), 2108541 (3 of 31)<br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in unp 28(3), 2108541 (3 of 31)<br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in un (28(3), 2108541 (3 of 31)<br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in u  $(28(3), 2108541 (3 of 31))$ <br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in un  $(283), 2108541 (3 of 31)$ <br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in unp  $\frac{1}{28(3)}$ ,  $\frac{2108541}{3}$  of 31)<br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-bas  $28(3)$ ,  $2108541$  (3 of 31)<br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs  $(28(3), 2108541 (3 of 31))$ <br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in un  $(28(3), 2108541 (3 of 31))$ <br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in un , 28(3), 2108541 (3 of 31)<br>processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment scen with Ni-based OECs in **EXECTS FROM THET SETT CONSULTS CONSULTS CONSULTS (NOTE)**<br>
Ni-based OECs.<br>
Without a doubt, Fe incorporation has been the<br>
driving force behind the significant OER improve-<br>
ment seen with Ni-based OECs in unpurified, rea processes by which Fe improves the OER activity of<br>Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in unpurified, reagent<br>grade Ni-based OECs.<br>Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in unpurified, reagent<br>grade KOH electrolyte so far. OER activities were<br>being m Without a doubt, Fe incorporation has been the<br>driving force behind the significant OER improve-<br>ment seen with Ni-based OECs in unpurified, reagent<br>grade KOH electrolyte so far. OER activities were<br>being massively reporte driving force behind the significant OER improvement seen with Ni-based OECs in unpurified, reagent grade KOH electrolyte so far. OER activities were being massively reported for these materials without realizing that Fe i ment seen with Ni-based OECs in unpurified, reagent<br>grade KOH electrolyte so far. OER activities were<br>being massively reported for these materials without<br>realizing that Fe in concentration as low as 1 ppm<br>may significant grade KOH electrolyte so far. OER activities were<br>being massively reported for these materials without<br>realizing that Fe in concentration as low as 1 ppm<br>may significantly change the OER activities of<br>Ni-based OECs. Based being massively reported for these materials without<br>realizing that Fe in concentration as low as 1 ppm<br>may significantly change the OER activities of<br>Ni-based OECs. Based on these findings, much re-<br>search has been publi realizing that Fe in concentration as low as 1 ppm<br>may significantly change the OER activities of<br>Ni-based OECs. Based on these findings, much re-<br>search has been published in literatures detailing the<br>various functions o may significantly change the OER activities of<br>Ni-based OECs. Based on these findings, much re-<br>search has been published in literatures detailing the<br>various functions of Fe in the matrix of Ni-based<br>OECs. Important role Ni-based OECs. Based on these findings, much re-<br>search has been published in literatures detailing the<br>various functions of Fe in the matrix of Ni-based<br>OECs. Important roles stated in literatures with ex-<br>perimental pie search has been published in literatures detailing the<br>various functions of Fe in the matrix of Ni-based<br>OECs. Important roles stated in literatures with ex-<br>perimental pieces of evidence are: 1) Incorporation of<br>Fe in pl various functions of Fe in the matrix of Ni-based<br>OECs. Important roles stated in literatures with ex-<br>perimental pieces of evidence are: 1) Incorporation of<br>Fe in place of Ni in their respective hydroxides/(oxy)<br>hydroxid Cs. Important roles stated in literatures with ex-<br>imental pieces of evidence are: 1) Incorporation of<br>in place of Ni in their respective hydroxides/(oxy)<br>droxides matrices makes them harder to be oxi-<br>ed further and thus perimental pieces of evidence are: 1) Incorporation of<br>Fe in place of Ni in their respective hydroxides/(oxy)<br>hydroxides matrices makes them harder to be oxi-<br>dized further and thus alters the redox electrochem-<br>istry of Fe in place of Ni in their respective hydroxides/(oxy)<br>hydroxides matrices makes them harder to be oxi-<br>dized further and thus alters the redox electrochem-<br>istry of Ni, 2) When Fe is incorporated into Ni-based<br>OECs, it c hydroxides matrices makes them harder to be oxidized further and thus alters the redox electrochemistry of Ni, 2) When Fe is incorporated into Ni-based OECs, it causes structural changes, 3) It is well-known fact that  $Fe^{$ dized further and thus alters the redox electrochemistry of Ni, 2) When Fe is incorporated into Ni-based OECs, it causes structural changes, 3) It is well-known fact that Fe<sup>3+</sup> serves as the strongest transition met-<br>als istry of Ni, 2) When Fe is incorporated into Ni-based<br>OECs, it causes structural changes, 3) It is well-known<br>fact that Fe<sup>3+</sup> serves as the strongest transition met-<br>als-based Lewis acid with a stronger electrophilic na-

OECs, it causes structural changes, 3) It is well-known<br>fact that Fe<sup>3+</sup> serves as the strongest transition met-<br>als-based Lewis acid with a stronger electrophilic na-<br>ture and notably affects the electronic properties of fact that Fe<sup>3+</sup> serves as the strongest transition met-<br>als-based Lewis acid with a stronger electrophilic na-<br>ture and notably affects the electronic properties of<br>the other cations in which being incorporated; hence<br>it als-based Lewis acid with a stronger electrophilic na-<br>ture and notably affects the electronic properties of<br>the other cations in which being incorporated; hence<br>it changes the Ni oxidation states and aids in the for-<br>mat ture and notably affects the electronic properties of<br>the other cations in which being incorporated; hence<br>it changes the Ni oxidation states and aids in the for-<br>mation of active Ni<sup>4+</sup> sites, and 4) Fe<sup>3+</sup> ions in<br>NiFethe other cations in which being incorporated; hence<br>it changes the Ni oxidation states and aids in the for-<br>mation of active Ni<sup>4+</sup> sites, and 4) Fe<sup>3+</sup> ions in<br>NiFe-based OECs with a 1:0.33 ratio depicts a su-<br>perexchan it changes the Ni oxidation states and aids in the for-<br>mation of active Ni<sup>4+</sup> sites, and 4) Fe<sup>3+</sup> ions in<br>NiFe-based OECs with a 1:0.33 ratio depicts a su-<br>perexchange magnetic contact that improves OER<br>electrocatalysi mation of active Ni<sup>4+</sup> sites, and 4) Fe<sup>3+</sup> ions in<br>NiFe-based OECs with a 1:0.33 ratio depicts a su-<br>perexchange magnetic contact that improves OER<br>electrocatalysis by allowing electrons to hop.<br>Almost all researches in NiFe-based OECs with a 1:0.33 ratio depicts a su-<br>perexchange magnetic contact that improves OER<br>electrocatalysis by allowing electrons to hop.<br>Almost all researches involving Ni-based OECs<br>and Fe incorporation have revea perexchange magnetic contact that improves OER<br>electrocatalysis by allowing electrons to hop.<br>Almost all researches involving Ni-based OECs<br>and Fe incorporation have revealed that the most ap-<br>propriate and active composi

 $#E\# (J. Electrochem.) 2022, 28(3), 2108541 (4 of 31)$ activity despite a reduction in effective conductivity.<br>
As a result, it is apparent that the observed increase<br>
As a result, it is apparent that the observed increase<br>
could not  $\text{H}(k\#(J. Electron.) 2022, 28(3), 2108541 (4 of 31)$ <br>activity despite a reduction in effective conductivity.<br>As a result, it is apparent that the observed increase<br>could not be attributed only to the change in conduc-<br>peak when age the  $\mathbb{R}E^*$  (*J. Electrochem.*) 2022, 28(3), 2108541 (4 of 31)<br>activity despite a reduction in effective conductivity. idation<sup>psq</sup>. Boettcher and co-workers observed compa-<br>As a result, it is apparent that the observ the  $W^2(X, Electrochem, 2022, 28(3), 2108541 (4 of 31)$ <br>activity despite a reduction in effective conductivity. idation<sup>[95]</sup>. Boettcher and co-workers observed compa-<br>As a result, it is apparent that the observed increase rable ano **EVACAL Example 10**<br> **EVACAL Example 1002**<br> **EXECUTE ACCES AND ACCES AS a result, it is apparent that the observed incompareable and<br>
could not be attributed on EVALUATION**<br> **EXALUATION 1999**<br> **EXALUATION 1999**<br> **EXALUATION ACCOMPTED AS a result, it is apparent that the observed increase rable and<br>
<b>EXALUATION AS a result, it is a pracent that the observed increase rable and<br>
c**  $\pm$  *i Electrochem.*) 2022, 28(3), 2108541 (4 of 31)<br>
activity despite a reduction in effective conductivity. idation<sup>89</sup>. Boettcher and co-workers observed compa-<br>
As a result, it is apparent that the observed increa **Example 19**<br> **Example 3**<br> **Example 1**<br> **Example 1**<br> **Example 1**<br> **Example 1**<br> **Example 1**<br> **Example 1**<br> **Example 3**<br> **Example 3**  $\pm$  *Wether a* eduction in effective conductivity.<br>  $\pm$  *Wether and co-workers observed comparison* and the dependent of the observed increase in the dependent of Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH oxidation could not be attributed o **EVALUATIVE THE CO-EXECTS INTERT AMORE THE CO-EXECTS IN A SURFACT CONDITED SOLUTION (INCRED SOLUTION) IN EXAMPLE CONDITIONS (INCRED SOLUTION IN THE SIGN OF CONDITIONS AND RESPONSIBLE THE VALUATION OF THE SIGN OF THE SIGN** (https://www.com/common-2022, 28(3), 2108541 (4 of 31)<br>
and the conductivity despite a reduction in effective conductivity. ideation<sup>195</sup>. Boettcher and co-workers observed compar-<br>
As a result, it is apparent that the ob the  $\mathcal{L}^{\mu}$  is the remotential phase of the reader of a result in the reduction of the reduction in effective conductivity. Identical and co-workers observed comparison as a result, it is apparent that the observed i **EVALUATION**<br> **EVALUATION**  $\# \{\&\cong (L \to U, \&Eleurochem.)\ 2022, 28(3), 2108541 \ (4 of 31) \}$ activity despite a reduction in effective conductivity. idation<sup>181</sup>. Roetcher and co-workers observed compa-<br>As a result, it is apparent that the observed increase ra the  $\frac{d_1}{dx}$  of  $\frac{d_2}{dx}$  ( $\frac{d_3}{dx}$  are a detection in effective conductivity. idation<sup>18</sup>, Boettcher and co-workers observed comparies a result, it is apparent that the observed increase — mble anodic shifts of N activity despite a reduction in effective conductivity.<br>
activity despite a reduction in effective conductivity.<br>
As a result, it is apparent that the observed increase<br>
rable anodic shifts of Ni(OH)<sub>2</sub> -> NiOOH oxidation activity despite a reduction in effective conductivity. idition<sup>188</sup>. Boettcher and co-workers observed compar-<br>
Cas a result, it is apparent that the observed increase and herease and<br>
could not be attributed only to the Ni-based OECs. The fact that Fe in FeOOH is insu-<br>
KOH( $\sim$ 1 ppm Fe) for 1 hour (Fi<br>
lating, which is a big contradiction. In this way, how<br>
the Fe concentration reached 5%<br>
could it improve the conductivity of NiOOH when



 $^{-1}$  Fe(NO<sub>3</sub>)<sub>3</sub> in a 1 mol  $\cdot$  L<sup>-1</sup> aqueous KOH <sup>1 CH</sup> 1 CH <sup>1</sup>

As a result, it is apparent that the observed increase<br>
reals anodic shifts of Ni(OH)<sub>2</sub> → NiOOH osidiation<br>
could not be atrivated only to the change in conduc-<br>
peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>
Nicould not be attributed only to the change in conduc-<br>
trivity that occurs when aged the clectrodeposited Ni(OH)<sub>1</sub> film in<br>
Wi-based OECs. The fact that Fe in incoporated into TraceSelect KOH ( $\leftarrow$  3 by the phe Fe) and r tivity that occurs when Fe is incorporated into TraceSclect KOH (~ 36 ppb Fe) and reagent grade<br>Ni-based OECs. The fact that Fe in FeOOH is insu-<br>Kang, which is a hige contradiction. In this way, how the Fe concentration ing, which is a big contradiction. In this way, how<br>
the Fe concentration reached 5%, they found a simi-<br>
dud it improve the conductivity of NiOOH when<br>
the range in the NiCos Hendre in the reachesis of Fin in pure<br>
whine could it improve the conductivity of NiOOH when<br>
lare change in the Ni:Fe co-deposited film in pure<br>
combined? Bell and co-workers proposed a plausible<br>
emchanism in which Fe<sup>y</sup> ions were incorporated into<br>
diation Shiff combined? Bell and co-workers proposed a plausible<br>
ECHI (Fe free). However, there was no such and<br>
enchanism in which Fe' ions were incorporated into shift in Ni(OH),  $\sim$  NiOOH orientation peak in the<br>
g-NiOOH lattices mechanism in which Fe<sup>31</sup> ions were incorporated into<br>
shift in Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH coidation peak in the<br>
deNitection is an earlier announts and exhibited hire to edeposited film with 25% Fe contract. The<br>
an extraordin B-NiOOH lattices in smaller amounts and exhibited NiF-Fe co-deposited film with 25% Fe content. The<br>m extraordinary shrunken Fe-O bond length, result- most intriguing result of this research was the reduc-<br>ong in Fe' site 28(3), 2108541 (4 of 31)<br>idation<sup>[35]</sup>. Boettcher and co-workers observed compa-<br>rable anodic shifts of Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH oxidation<br>peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>TraceSelect KOH (~ 36 ppb Fe) an 28(3), 2108541 (4 of 31)<br>idation<sup>[35]</sup>. Boettcher and co-workers observed compa-<br>rable anodic shifts of Ni(OH)<sub>2</sub> → NiOOH oxidation<br>peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>TraceSelect KOH (~ 36 ppb Fe) and r 28(3), 2108541 (4 of 31)<br>idation<sup>[35]</sup>. Boettcher and co-workers observed compa-<br>rable anodic shifts of Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH oxidation<br>peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>TraceSelect KOH ( $\sim$  36 ppb Fe) 28(3), 2108541 (4 of 31)<br>
idation<sup>[35]</sup>. Boettcher and co-workers observed compa-<br>
rable anodic shifts of Ni(OH)<sub>2</sub> → NiOOH oxidation<br>
peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>
TraceSelect KOH (~ 36 ppb Fe) a 28(3), 2108541 (4 of 31)<br>
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idation<sup>[35]</sup>. Boettcher and co-workers observed compa-<br>
rable anodic shifts of Ni(OH)<sub>2</sub> → NiOOH oxidation<br>
peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>
TraceSelect KOH (~ 36 ppb Fe) 28(3), 2108541 (4 of 31)<br>
idation<sup>[35]</sup>. Boettcher and co-workers observed compa-<br>
rable anodic shifts of Ni(OH)<sub>2</sub> → NiOOH oxidation<br>
peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>
TraceSelect KOH (~ 36 ppb Fe) 28(3), 2108541 (4 of 31)<br>idation<sup>[35]</sup>. Boettcher and co-workers observed compa-<br>rable anodic shifts of Ni(OH)<sub>2</sub> → NiOOH oxidation<br>peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>TraceSelect KOH (~ 36 ppb Fe) and r 28(3), 2108541 (4 of 31)<br>idation<sup>[58]</sup>. Boettcher and co-workers observed compa-<br>rable anodic shifts of Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH oxidation<br>peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>TraceSelect KOH (~ 36 ppb Fe) an 28(3), 2108541 (4 of 31)<br>
idation<sup>[59]</sup>. Boettcher and co-workers observed compa-<br>
rable anodic shifts of Ni(OH)<sub>2</sub> → NiOOH oxidation<br>
peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>
TraceSelect KOH (~ 36 ppb Fe) 28(3), 2108541 (4 of 31)<br>
idation<sup>[85]</sup>. Boettcher and co-workers observed compa-<br>
rable anodic shifts of Ni(OH)<sub>2</sub> → NiOOH oxidation<br>
peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>
TraceSelect KOH (~ 36 ppb Fe) a 28(3), 2108541 (4 of 31)<br>idation<sup>[59]</sup>. Boettcher and co-workers observed compa-<br>rable anodic shifts of Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH oxidation<br>peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>TraceSelect KOH (~ 36 ppb Fe) an 28(3), 2108541 (4 of 31)<br>
idation<sup>[35]</sup>. Boettcher and co-workers observed compa-<br>
rable anodic shifts of Ni(OH)<sub>2</sub> → NiOOH oxidation<br>
peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>
TraceSelect KOH (~ 36 ppb Fe) a Let  $\mathbb{R}^{3}$ . Boettcher and co-workers observed comparable anodic shifts of Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH oxidation peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in TraceSelect KOH ( $\sim$  36 ppb Fe) and reagent grade KOH (idation<sup>[35]</sup>. Boettcher and co-workers observed comparable anodic shifts of Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH oxidation peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in TraceSelect KOH (~ 36 ppb Fe) and reagent grade KOH (~ 1 pp rable anodic shifts of Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH oxidation<br>peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>TraceSelect KOH (~ 36 ppb Fe) and reagent grade<br>KOH (~ 1 ppm Fe) for 1 hour (Figure 3(A-E))<sup>p6</sup>. When<br>the Fe conc peak when aged the electrodeposited Ni(OH)<sub>2</sub> film in<br>TraceSelect KOH (~ 36 ppb Fe) and reagent grade<br>KOH (~ 1 ppm Fe) for 1 hour (Figure 3(A-E))<sup>p6</sup>. When<br>the Fe concentration reached 5%, they found a simi-<br>lar change in TraceSelect KOH (~ 36 ppb Fe) and reagent grade<br>
KOH (~ 1 ppm Fe) for 1 hour (Figure 3(A-E))<sup>pg</sup>. When<br>
the Fe concentration reached 5%, they found a simi-<br>
lar change in the Ni:Fe co-deposited film in pure<br>
KOH (Fe free) KOH ( $\sim$  1 ppm Fe) for 1 hour (Figure 3(A-E))<sup>pg</sup>. When<br>the Fe concentration reached 5%, they found a simi-<br>lar change in the Ni:Fe co-deposited film in pure<br>KOH (Fe free). However, there was no such anodic<br>shift in Ni( In Fe concentration reached 5%, they found a similar change in the Ni:Fe co-deposited film in pure KOH (Fe free). However, there was no such anodic shift in Ni(OH)<sub>2</sub> → NiOOH oxidation peak in the Ni:Fe co-deposited film KOH (Fe free). However, there was no such anodic<br>shift in Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH oxidation peak in the<br>Ni:Fe co-deposited film with 25% Fe content. The<br>most intriguing result of this research was the reduc-<br>tion in the stre shift in Ni(OH)<sub>2</sub> → NiOOH oxidation peak in the<br>Ni:Fe co-deposited film with 25% Fe content. The<br>most intriguing result of this research was the reduc-<br>tion in the strength of the Ni(OH)<sub>2</sub> → NiOOH oxida-<br>tion peak when Ni:Fe co-deposited film with 25% Fe content. The<br>most intriguing result of this research was the reduc-<br>tion in the strength of the Ni(OH)<sub>2</sub> → NiOOH oxida-<br>tion peak when aged in Fe-free KOH and the devel-<br>opment of a n most intriguing result of this research was the reduction in the strength of the Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH oxidation peak when aged in Fe-free KOH and the development of a new peak at a high overpotential, which was ascribed t tion in the strength of the Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH oxida-<br>tion peak when aged in Fe-free KOH and the devel-<br>opment of a new peak at a high overpotential, which<br>was ascribed to the production of Ni<sup>4+</sup> ions. They also<br>discov tion peak when aged in Fe-free KOH and the devel-<br>opment of a new peak at a high overpotential, which<br>was ascribed to the production of Ni<sup>+</sup> ions. They also<br>discovered that OER activity was reduced with ag-<br>ing time and opment of a new peak at a high overpotential, which<br>was ascribed to the production of Ni<sup>+</sup> ions. They also<br>discovered that OER activity was reduced with ag-<br>ing time and attributed it to the reduction in struc-<br>tural f is ascribed to the production of Ni<sup>+</sup> ions. They also<br>covered that OER activity was reduced with ag-<br>g time and attributed it to the reduction in struc-<br>al flaws in Ni(OH)<sub>2</sub>. However, Ni(OH)<sub>2</sub> films aged<br>TraceSelect KO discovered that OER activity was reduced with ag-<br>ing time and attributed it to the reduction in struc-<br>tural flaws in Ni(OH)<sub>2</sub>. However, Ni(OH)<sub>2</sub> films aged<br>in TraceSelect KOH (~ 36 ppb Fe) and reagent grade<br>KOH (~ 1 p ing time and attributed it to the reduction in structural flaws in Ni(OH)<sub>2</sub>. However, Ni(OH)<sub>2</sub> films aged<br>in TraceSelect KOH ( $\sim$  36 ppb Fe) and reagent grade<br>KOH ( $\sim$  1 ppm Fe) showed a substantial increase in<br>OER ac tural flaws in Ni(OH)<sub>2</sub>. However, Ni(OH)<sub>2</sub> films aged<br>in TraceSelect KOH ( $\sim$  36 ppb Fe) and reagent grade<br>KOH ( $\sim$  1 ppm Fe) showed a substantial increase in<br>OER activity. This suggests that incorporation of Fe<br>boost in TraceSelect KOH ( $\sim$  36 ppb Fe) and reagent grade<br>
KOH ( $\sim$  1 ppm Fe) showed a substantial increase in<br>
OER activity. This suggests that incorporation of Fe<br>
boosts activity, although the anodic shift of Ni(OH)<sub>2</sub><br> KOH ( $\sim$  1 ppm Fe) showed a substantial increase in<br>OER activity. This suggests that incorporation of Fe<br>boosts activity, although the anodic shift of Ni(OH)<sub>2</sub><br>→ NiOOH oxidation peak in these cases is opposite<br>to what 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (4 of 31)<br>
ffective conductivity. idation<sup>[35]</sup>. Boettcher and co-workers observed compa-<br>
the observed increase rable anodic shifts of Ni(OH)<sub>2</sub> → NiOOH oxidation<br>
the change

OER activity. This suggests that incorporation of Fe<br>boosts activity, although the anodic shift of Ni(OH)<sub>2</sub><br>→ NiOOH oxidation peak in these cases is opposite<br>to what was anticipated. Furthermore, aging had no<br>effect on boosts activity, although the anodic shift of Ni(OH)<sub>2</sub><br>  $\rightarrow$  NiOOH oxidation peak in these cases is opposite<br>
to what was anticipated. Furthermore, aging had no<br>
effect on the co-deposited NiFe-(oxy)hydroxide films.<br>
The  $\rightarrow$  NiOOH oxidation peak in these cases is opposite<br>to what was anticipated. Furthermore, aging had no<br>effect on the co-deposited NiFe-(oxy)hydroxide films.<br>They concluded that the OER activity is intrinsic to<br>NiFe-(oxy) to what was anticipated. Furthermore, aging had no<br>effect on the co-deposited NiFe-(oxy)hydroxide films.<br>They concluded that the OER activity is intrinsic to<br>NiFe-(oxy)hydroxide sheets and not linked to struc-<br>tural flaws effect on the co-deposited NiFe-(oxy)hydroxide films.<br>They concluded that the OER activity is intrinsic to<br>NiFe-(oxy)hydroxide sheets and not linked to struc-<br>tural flaws in Ni<sub>075</sub>Fe<sub>025</sub>OOH. However, the observed<br>anodic They concluded that the OER activity is intrinsic to NiFe-(oxy)hydroxide sheets and not linked to structural flaws in Ni<sub>075</sub>Fe<sub>025</sub>OOH. However, the observed anodic shift of Ni(OH)<sub>2</sub> → NiOOH oxidation peak in their rese NiFe-(oxy)hydroxide sheets and not linked to structural flaws in  $Ni_{0.75}Fe_{0.25}OOH$ . However, the observed anodic shift of Ni(OH)<sub>2</sub> → NiOOH oxidation peak in their research could not be fully explained.<br>Later on, Strasse Figure 2 Steady-state constant in the competent in the state of the conductivity of NiOH), and a<br>
state of NiORHy or the state effect on the co-deposited NiFe-(oxylhydroxide films<br>
tistics of NiORHy of the state effect on Since of NiO(UI<sub>1</sub>). In the notic profession of the relation of the college incorporation of the NiO(UI1). In the DR activity is intrinsic to the observed from the Silfung electrochemical water ox-<br>
They concluded that th go of the Nt(OH)<sub>3</sub> oxida-<br>
lectrochemical water ox-<br>
They concluded that the OER activity is intrinsic to<br>
NiFe-(oxy)hydroxide films.<br>
Wind the OER activity is intrinsic to<br>
NiFe-(oxy)hydroxide films.<br>
In this concluded (IOH), oxida-<br>
effect on the co-deposited NiFe-(oxy)hydroxide films.<br>
ical water ox-<br>
They concluded that the OER activity is intrinsic to<br>
NiFe-(oxy)hydroxide films.<br>
Nicolarid at the OER activity is intrinsic to<br>
tural tion peak to NiOOH during electrochemical water ox-<br>
Weild (and the OER activity is intrinsic to structure of NiCOH)  $\frac{1}{2}$ . The control in the strength of NiCOH)  $\frac{1}{2}$  and the strength condition peak in the streng NiFe-(oxy)hydroxide sheets and not linked to strain<br>  $\frac{1}{2}$ <br>  $\frac$ 



Copyright©2014 American Chemical Society. Reproduced with permission. (color on line) **During O<sub>2</sub>** evolution may be responsible for insignifi-<br>
Numering Occurs and West the set of CV-R and the set of the set of the set of the content of the set of the Controlled the metal state of Circuit ( $\Delta E_{\rm tot} = 2$  metal detected metal and the state of  $\Delta E_{\rm tot} = 2$  metal and  $\Delta E_{\rm tot} = 2$  metal detected in the state of  $\Delta E_{\rm tot} = 2$  metal and  $\Delta E_{\rm tot} = 2$  metal and  $\Delta E_{\rm tot} = 2$ There was no Fe or very little Fe or very **Example 12**<br> **Example 12** 

**Example the main of Continuous Co Example 18.** The metal reduction phase during on the metal reduction phase during on the metal reduction phase during O2 evolution phase during O2 evolution phase during O2 evolution phase during O2 evolution phase durin From a computer metallic of the substantial of the substantial of the substantial of the substantial of  $\delta$  for the substantial of  $\delta$  for the substantial  $\delta$  of  $\delta$  for the substantial  $\delta$  of  $\delta$  or  $\delta$  for the su **Example 19**<br>
Note the **Concertification**  $\frac{d^2}{dx^2} = 2m\sqrt{\frac{2m\pi}{\pi}} = 2m\sqrt{\frac{2m\pi}{$ **Example 1.**  $\frac{1}{4}$  **Constrained Constrained C Eighree 3** Cyclic volumme pearals in ( $\eta$ ) = **R**<br> **Eighree 3** Cyclic volumme grans were taken during the uging of films in various partices of KOII. A total of 13 CV seems are<br>
shown for each sample: one for the initial **Figure 3** Cyclic voltanmograns were taken during the aging of films in various purities of KOH. A total of 13 CV scans are shown for each system from the case of the minia as-deposited flim (the dark purity)e), and one a shown for each sample: one for the initial as-deposited flim (the dark parple), and one additional sean after each 5 min aging period<br>up to a total of hour of aging (the dark roll). The changes in the amolie are lost step np to a total of 1 hour of sging (the dark red). The changes in the anodic and cathodic peak positions  $(\Delta E_{\rm so}$  and  $\Delta E_{\rm so}$ ) are labeled for custom for the Nix-Fax-no(10%, cold,  $(\Delta E_{\rm so}$  vanish in the next in the N each set of CVs. ( $\Delta E_n$ , value is abova for the Ni<sub>i</sub>-Fe<sub>an</sub>(OH), as the oxidation peak is partially obscured by the OER current.)<sup>301</sup><br>Copyright@2014 American Chemical Society. Reproduced with permission. (color on line) Copyright 20014 American Chemical Society. Reproduced with permission. (color on line)<br>
during O<sub>2</sub> evolution may be responsible for insignifi-<br>
portance of Fe concentration, electrolyte strength,<br>
can detectable high val during O<sub>2</sub> cvolution may be responsible for insignifi-<br>portance of Fe concentration, clectrolyte strength,<br>cant detectable high valence metal deposition<sup>281</sup>. When and support material during catalyst fabrication. Al-<br>th during O<sub>2</sub> evolution may be responsible for insignification of Fe concentration, electrolyte strength,<br>cant decreasted high valuence metal deposition<sup>59</sup>. When and support material during etally<br>the evas no Fe or very lit cant detectable high valence metal deposition<sup>93</sup>. When and support material during eatalyst fabrication. Althore was no Fe or very little Fe (less than 10%), the though there is an explanation for why andic shift knetice there was no Fe or very little Fe (less than 10%), the though there is an explanation for why anodic shift kin<br>competition competition preferred metal oxidation, but and intensity drop oecur in Ni-based OECs with im-<br>kwhe

etivity<br>  $\begin{pmatrix}\n\overline{e} & 0.3 & 0.4 & 0.5 & 0.6 \\
0.3 & 0.4 & 0.5 & 0.6\n\end{pmatrix}$ <br>
determined by  $\overline{e}$ <br>
determined by  $\overline{e}$ <br>  $\overline{e}$ <br> E<br>
and the subset of  $\frac{5}{2}$ <br>  $\frac{25}{2}$ terivity<br>  $\frac{2}{5}$ <br>  $\frac{2}{5}$ <br>
Elms in various purities of KOH. A ctivity  $\frac{2}{3}$ <br>  $\frac{2}{3}$ <br>
Ellins in various purities of KOH. A total of 13 CV scans are<br>
purified KOH<br>
in t Example and Manuscript Manuscript Care is a component of Nie-<br>
Fe contential,  $\eta(\mathbf{V})$  and  $\mathbf{R}_{\text{p},\epsilon} = -2 \text{ mV}$  Ni<sub>9,75</sub>Fe  $\mathbf{e}_{\alpha,3}(\mathbf{O}H)$ , purified KOH<br>
Durified KOH<br>
Expressed on the contential,  $\eta(\mathbf{V})$  do defined the subset of Niegral and September in a subject of Alberta Correlation in the subsetion of  $\frac{1}{2}$  is still and cathodic peak positions ( $\Delta E_{pa}$  and  $\Delta E_{pa}$ ) are labeled for original and cathodic peak posi  $\sum_{k=1}^{n}$   $\Delta E_{p,q} = -2 \text{ mV} \frac{\mathbf{M}_{0,pq} \mathbf{F}_{0,2pq}(\text{OH})_2}{\text{partial KO}}$ <br>  $\Delta \mathbf{F}_{0,1} = -2 \text{ mV} \frac{\mathbf{M}_{0,pq} \mathbf{F}_{0,2pq}(\text{OH})_2}{\text{partial KO}}$ <br>  $\Delta \mathbf{F}_{0,2}$ <br>  $\Delta \mathbf{F}_{0,3}$ <br>  $\Delta \mathbf{F}_{0,4}$  and  $\Delta \mathbf{F}_{0,5}$  and  $\Delta \mathbf{F}_{0,6}$  ar ble findings in a study of NiFe-LDH (LDH: layered

do **Containery**<br> **Configured Consisting**<br> **Consponential**,  $\eta(\mathbf{0} \cdot \mathbf{R}_{\theta})$ <br> **Consponential**,  $\eta(\mathbf{0} \cdot \mathbf{R}_{\theta})$ <br>
diffus and cathodic peak positions ( $\Delta E_{\theta\theta}$  and  $\Delta E_{\theta\theta}$ ) are labeled for<br>
e oxidation peak **Conditionally Conditionally Conditional**<br> **Compositerists**,  $\mathbf{\hat{r}}(\mathbf{M}) \cdot \mathbf{R}_{\alpha}$ <br> **Conditional scan after each** 5 min aging period<br>
dic and cathodic peak positions ( $\Delta E_{\gamma,\alpha}$  and  $\Delta E_{\alpha,\beta}$ ) are labeled for films in various purities of KOH. A total of 13 CV scans are<br>purple), and one additional scan after each 5 min aging period<br>dic and cathodic peak positions  $(\Delta E_{p,a} \text{ and } \Delta E_{p,b})$  are labeled for<br>e oxidation peak is partial purple), and one additional scan after each 5 min aging period<br>dic and cathodic peak positions  $(\Delta E_{p,a} \text{ and } \Delta E_{p,b})$  are labeled for<br>e oxidation peak is partially obscured by the OER current.)<sup>[36]</sup><br>mission. (color on lin dic and cathodic peak positions ( $\Delta E_{\rm ps}$  and  $\Delta E_{\rm nb}$ ) are labeled for<br>e oxidation peak is partially obscured by the OER current.)<sup>[36]</sup><br>mission. (color on line)<br>portance of Fe concentration, electrolyte strength,<br>an is partially obscured by the OER current.)<sup>[36]</sup><br>ission. (color on line)<br>portance of Fe concentration, electrolyte strength,<br>and support material during catalyst fabrication. Al-<br>though there is an explanation for why ano insision. (color on line)<br>portance of Fe concentration, electrolyte strength,<br>and support material during catalyst fabrication. Al-<br>though there is an explanation for why anodic shift<br>and intensity drop occur in Ni-based portance of Fe concentration, electrolyte strength,<br>and support material during catalyst fabrication. Al-<br>though there is an explanation for why anodic shift<br>and intensity drop occur in Ni-based OECs with in-<br>creasing Fe portance of Fe concentration, electrolyte strength,<br>and support material during catalyst fabrication. Al-<br>though there is an explanation for why anodic shift<br>and intensity drop occur in Ni-based OECs with in-<br>creasing Fe and support material during catalyst fabrication. Al-<br>though there is an explanation for why anodic shift<br>and intensity drop occur in Ni-based OECs with in-<br>creasing Fe content, but enhancing mechanism by re-<br>dox inactive though there is an explanation for why anodic shift<br>and intensity drop occur in Ni-based OECs with in-<br>creasing Fe content, but enhancing mechanism by re-<br>dox inactive Fe<sup>3+</sup> ions is still a subject of ambiguity.<br>Gray and



 $K$ -edges<sup>[37]</sup>, Copyright<sup>©</sup>2016 American Chemical Society, Reproduced with permission, (color on line)

**For OCE 1998**<br> **Example 3.5**<br> **Example 3.5**<br> **Example 3.6**<br> **Example 3 EVALUATION CONTROV**<br> **EVALUATION CONTROV**<br> **EVALUATION CONTROV**<br> **EVALUATION CONTROV**<br> **EXECUTED CON EVALUATION THE CONTROVERT CONTROVER CONTROVER CONTROVERTIES WELL AS THE CONTROVERT CONTROVERT CONTROVERT (SOPTIBOT THE CONTROVERT CONTROVERT CONTROVERT CONTROVERT CONTROVERT CONTROVERT CONTROVERT CONTROVERT CONTROVERT CO EVALUAT:** This  $\frac{1}{2}$  or  $\frac{1}{2}$  or  $\frac{1}{2}$   $\frac{1}{2}$ **EVALUATE 1999** Energy /eV<br>
E lution.

OFR intermediates have a weaker adsorption contact (b) Fe<sup>3</sup> ions have a 6-percent shorter Fe-O bond length<br>which welve they ceal to be collent M-O bonds, in NiOOH matrices, suggesting that Fe<sup>14</sup> ions may<br>which weld a la with Ni where they create more covalent M-O bonds,<br>
which need a larger potential to break in order-<br>
order aleady brighter orderivations states, and erives the proferable reduced as<br>
welop O<sub>2</sub>. This is why, in the absen which need a larger potential to break in order to de-<br>nearby higher oxidation states, and active sites in<br>velop  $O_2$ . This is why, in the absence of Fe, Ni(OH)<sub>2</sub> both Nife systems are anticipated to be Fe<sup>3</sup>. The<br>needs velop O<sub>2</sub>. This is why, in the absence of Fe, Ni(OH)<sub>2</sub> both NiFe systems are anticipated to be Fe<sup>32</sup>. The meets a greater overpotential to start the oxygen evo-<br>
and cotive medium needed for Fe<sup>33</sup> to analyze OER<br>
Inti

**Energy / EV Energy / EV And the state upon Fe incorporation. It was also discovered cause the only f Figure 4** *In-singipermedo* XAS of the NiFe OER eatalysts with varying eatalyst composition Ni<sub>ne</sub>, Fe, (A) Fe *K*-edges and (B) Ni<br> *K*-edges<sup>19</sup>. Copyright<sup>62</sup>2016 American Chemical Society. Reproduced with permission. **ngure** 4 *m*-anoperamic AAS or the sure curst causuas win vaping campast como relugies. (A) re A-eages and (B) start  $\epsilon$ -deges<sup>30</sup>. Copyright@2016 American Chemical Society. Reproduced with permission. (color on line)<br> te upon Fe incorporation. It was also discovered cause the only first monolayer of FeOOH is OFR ac-<br>t the highly active Ni,Fe<sub>2</sub>,OOH catalyst has two tive and electrolyte-permeable at lower overpoten-<br>ive sites with remar state upon Fe incorporation. It was also discovered cause the only first monolayer of FeOOH is OER ac-<br>that the highly acity Ni<sub>J</sub>Fe<sub>1</sub>,OOH cealsly has two tive and electrolyt-permeable at lower overpoten-<br>eactive sites w that the highly active Ni<sub>F</sub>e<sub>1</sub>,OOH catalyst has two tive and electrolyte-permeable at lower overpoten-<br>active sites with remarkable difference in OFR rate tails;<sup>421</sup>, 3) The active sites are Fe'<sup>1</sup> ions that replaced<br>c active sites with remarkable difference in OER rate inls<sup>te(1</sup>, 4) The active sites are Fe<sup>3</sup> ions that replaced<br>constants, and the precentage of fast sites matched Ni<sup>7</sup> ions in NiOOH matrices at the edges, comers,<br>well constants, and the percentage of fast sites matched Ni<sup>2+</sup> ions in NiOOH matrices at the edges, corners, well with the fraction of Fe in the catalyst<sup>19</sup>. These and defects itss. Those who are deeply embedded in findings well with the fraction of Fe in the catalyst<sup>30</sup>. These and defect sites. Those who are deeply embedded in finding schrifted that Fe and Ni, bot har a active sites. The majority of NiOOH do not take part in OFRPs<sup>26</sup>. 5) findings clarified that Fe and Ni, both are active sites the majority of NiOOH do not take part in OER<sup>(16</sup>, 5)<br>
for OER, with Fe sites being aginificantly faster. This The active Ni<sup>2</sup> ions are simply stabilized by Fe<sup>3</sup> for OER, with Fe sites being significantly faster. This The active Ni\* ions are simply stabilized by Fe<sup>3</sup>, as in-<br>may be due to the optimal bond carrgics of interme-dicated by the anoids shift of the Ni(Oft)<sub>2</sub> -> NiOOH<br> may be due to the optimal bond energies of interme-<br>dicated by the anodic shift of the Ni(OH)<sub>2</sub> → NiOOH<br>OER intermediates when corollande with Pc. On the other hand, coxidation peak with the decreased integrated change®, diates when coordinated with Fe. On the other hand,<br>
oxidation peak with the decreased integrated charge<sup>[26]</sup><br>
OER intermediates lawe a weaker adsoption conduct (b) Fe<sup>+</sup> into layer a 6-percent shorter Fe-O bond length<br> Ni<sup>+2</sup><br>
XANES<br>
Ning catalyst composition Ni<sub>LG+</sub>Fe<sub>x</sub>: (A) Fe *K*-edges and (B) Ni<br>
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cause the only first mon N<sup>1+2</sup><br>
XANES<br>
2330 8350 8370 8390<br>
Energy /eV<br>
ving catalyst composition Ni<sub>va-Fe</sub>: (A) Fe *K*-edges and (B) Ni<br>
d with permission. (color on line)<br>
cause the only first monolayer of FeOOH is OER ac-<br>
tive and electrolyt XANES<br>
8330 8350 8370 8390<br>
Energy /eV<br>
ving catalyst composition  $Ni_{100}$ , Fe,: (A) Fe *K*-edges and (B) Ni<br>
d with permission. (color on line)<br>
cause the only first monolayer of FeOOH is OER ac-<br>
tive and electrolyte-pe XANES<br>
S330 8350 8370 8390<br>
Energy /eV<br>
ving catalyst composition  $Ni_{10x}$ ,  $Fe_{x}$ : (A) Fe *K*-edges and (B) Ni<br>
d with permission. (color on line)<br>
cause the only first monolayer of FeOOH is OER ac-<br>
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Energy /eV<br>
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tive and electrolyte-permeable at lowe ing catalyst composition  $Ni_{100}$ -Fe<sub>3</sub>: (A) Fe K-edges and (B) Ni<br>
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tials<sup>[43]</sup>. 4) The active sites are Fe<sup>3+</sup> ions that replaced<br>
Ni<sup>3+</sup> ions in N cause the only first monolayer of FeOOH is OER ac-<br>tive and electrolyte-permeable at lower overpoten-<br>tials<sup>[42]</sup>. 4) The active sites are Fe<sup>3+</sup> ions that replaced<br>Ni<sup>3+</sup> ions in NiOOH matrices at the edges, corners,<br>and cause the only first monolayer of FeOOH is OER ac-<br>tive and electrolyte-permeable at lower overpoten-<br>tials<sup>[42]</sup>. 4) The active sites are Fe<sup>3+</sup> ions that replaced<br>Ni<sup>3+</sup> ions in NiOOH matrices at the edges, corners,<br>and tive and electrolyte-permeable at lower overpoten-<br>tials<sup>(a3)</sup>. 4) The active sites are Fe<sup>3+</sup> ions that replaced<br>Ni<sup>3+</sup> ions in NiOOH matrices at the edges, corners,<br>and defect sites. Those who are deeply embedded in<br>the tials<sup>(a3)</sup>. 4) The active sites are Fe<sup>3+</sup> ions that replaced<br>Ni<sup>3+</sup> ions in NiOOH matrices at the edges, corners,<br>and defect sites. Those who are deeply embedded in<br>the majority of NiOOH do not take part in OER<sup>[34]</sup>. 5 Ni<sup>3+</sup> ions in NiOOH matrices at the edges, corners,<br>and defect sites. Those who are deeply embedded in<br>the majority of NiOOH do not take part in OER<sup>[34]</sup>. 5)<br>The active Ni<sup>2+</sup> ions are simply stabilized by Fe<sup>3+</sup>, as in and defect sites. Those who are deeply embedded in<br>the majority of NiOOH do not take part in OER<sup>[34]</sup>. 5)<br>The active Ni<sup>2+</sup> ions are simply stabilized by Fe<sup>3+</sup>, as in-<br>dicated by the anodic shift of the Ni(OH)<sub>2</sub> → NiOO the majority of NiOOH do not take part in OER<sup>[34]</sup>. 5)<br>The active Ni<sup>2+</sup> ions are simply stabilized by Fe<sup>3+</sup>, as in-<br>dicated by the anodic shift of the Ni(OH)<sub>2</sub> → NiOOH<br>oxidation peak with the decreased integrated char is eactive Ni<sup>2+</sup> ions are simply stabilized by Fe<sup>3+</sup>, as in-<br>ated by the anodic shift of the Ni(OH)<sub>2</sub> → NiOOH<br>idation peak with the decreased integrated charge<sup>[38]</sup>.<br>Fe<sup>3+</sup> ions have a 6-percent shorter Fe-O bond leng dicated by the anodic shift of the Ni(OH)<sub>2</sub>  $\rightarrow$  NiOOH<br>oxidation peak with the decreased integrated charge<sup>188</sup>.<br>6) Fe<sup>3+</sup> ions have a 6-percent shorter Fe-O bond length<br>in NiOOH matrices, suggesting that Fe<sup>3+</sup> ions may oxidation peak with the decreased integrated charge<sup>188</sup>.<br>
6) Fe<sup>3\*</sup> ions have a 6-percent shorter Fe-O bond length<br>
in NiOOH matrices, suggesting that Fe<sup>3\*</sup> ions may<br>
have partly higher oxidation states, and active site 6) Fe<sup>3+</sup> ions have a 6-percent shorter Fe-O bond length<br>in NiOOH matrices, suggesting that Fe<sup>3+</sup> ions may<br>have partly higher oxidation states, and active sites in<br>both NiFe systems are anticipated to be Fe<sup>3+</sup>. The<br>cond in NiOOH matrices, suggesting that Fe<sup>3+</sup> ions may<br>have partly higher oxidation states, and active sites in<br>both NiFe systems are anticipated to be Fe<sup>3+</sup>. The<br>conductive medium needed for Fe<sup>3+</sup> to catalyze OER<br>is provid have partly higher oxidation states, and active sites in<br>both NiFe systems are anticipated to be Fe<sup>3+</sup>. The<br>conductive medium needed for Fe<sup>3+</sup> to catalyze OER<br>is provided by NiOOH matrices<sup>[43]</sup>. 7) Fe<sup>3+</sup> in the con-<br>d both NiFe systems are anticipated to be Fe<sup>3+</sup>. The<br>conductive medium needed for Fe<sup>3+</sup> to catalyze OER<br>is provided by NiOOH matrices<sup>[48]</sup>. 7) Fe<sup>3+</sup> in the con-<br>ductive Fe<sub>2</sub>S<sub>s</sub> phase is OER active in the same way as<br>F

 $\frac{\text{#L}\#(J. \text{Electrochem.}) 2022, 28(3), 2108541 (7 of 31)}{\text{during a redox reaction, Corrigan et al. conducted an}}$ The presence of Fe<sup>4+</sup> in the OER was first clearly<br> *in-situ*<sup>5</sup>Fe Mössbauer spectroscopic study<sup>469</sup>. They said (b) observed in 2015 when Stahl et al. then the tectrochem.) 2022, 28(3), 2108541 (7 of 31)<br>
during a redox reaction, Corrigan et al. conducted an<br>
in-situ <sup>37</sup>Fe Mössbauer spectroscopic study<sup>148</sup>. They said<br>
the electronic changes at Fe site are not due to<br>  $\frac{\text{d} \xi(\frac{1}{2} \xi(L \text{ *Electrochem.}) 2022, 28(3), 2108541 (7 of 31)}{\text{during a redox reaction, Corrigan et al. conducted an}}*$ <br>The presence of Fe<sup>+</sup> in the OER was first clearly<br>*in-situ*<sup>57</sup>Fe Mössbauer spectroscopic study<sup>361</sup>. They said observed in 2015 when Stahl e **the redox process in Fe**, but attributed to the host lat-<br>  $\mathbb{R}E^{\text{int}}(J. Electrochem.)$  2022, 28(3), 2108541 (7 of 31)<br>  $\mathbb{R}E^{\text{int}}$  are redox reaction, Corrigan et al. conducted an The presence of Fe<sup>4+</sup> in the OER was fi  $\pm \frac{1}{2}$  (*J. Electrochem.*) 2022, 28(3), 2108541 (7 of 31)<br>
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during a redox reaction, Corrigin et al. conducted an The presence of Fe<sup>t</sup> in the OER was first clearly<br>
in-situ<sup>5</sup>Fe Mössbauer spectroscopic study<sup>164</sup>. They usad in 20  $\mathbb{R}E\#(J. Electron, Corrigan et al. conducted an  
\n $\mathbb{R}E\#(J. Electron, Corrigan et al. conducted an  
\n $\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{R}E\#(\mathbb{$$$ **EXAMPLE 12**<br> **EXAMPLE 12** either of the single component hydroxides. It was **EXALLATE 1999**<br> **EXALLATE 1999 EXALLATE 19** *in-situ*<sup>31</sup>Fe Mössbauer spectroscopic study<sup>16</sup>. They said observed in 2015 when Stahl et al. investigated NiFe-<br>that the electronic changes at Fe site are not due to LDM electrocatalyst during OER<sup>(p)</sup>. They used *an-i* that the electronic changes at Fe site are not due to LDH electrocatalyst during OER<sup>[46]</sup>. They used *in-situl*<br>the redox process in Fe, but attributed to the host dat-<br>oriential. Moreover, an intimate orientation state the redox process in Fe, but attributed to the host lat-<br>
operando <sup>5</sup>Te Mössbauer technique to track the F<br>
tice in a composite material. Moreover, an intimate<br>
condinions tate in 3:1 NiFe-LDH and Fe oxide cat<br>
than the tice in a composite material. Moreover, an intimate oxidation state in 3:1 NiFe-LDH and Fe oxide eata-<br>combination of Fe and Ni on an atomic scale rather  $\frac{1}{2}$  sts, while polarizing them in the OER zone. hurt-<br>than th combination of Fe and Ni on an atomic scale rather<br>
than the mixture of Ni(OH)<sub>2</sub> and FeOOH particles<br>
estingly, as shown in Figure 5, the existence with the mixture of Ni(OH)<sub>2</sub> and FeOOH particles<br>
exhibited better perf

**Example 19**<br> **Example 19**  $\frac{\ln\{k\mathcal{P}(f, klecmcben, 2022, 28(3), 2108541 (7 \text{ or } 31)}{\ln\{k\}}$ <br>
during a redox reaction, Corrigan et al. conducted an The presence of Fe<sup>t+</sup> in the OER was first clearly<br>
tin-siza<sup>2</sup>Fe Müssbauer spectroscopic study<sup>68</sup>. Th **in the set of the SER was first clearly<br>in-sine in redox reaction, Corrigen et al. conducted an<br>in-sine in the OER was first clearly<br>that the electronic changes at F**  $4E^2(L)E(extrocheen.) 2022, 28(3), 2108541 (7 of 31)$ <br>during a redox reaction, Corrigan et al. conducted an The presence of Fe<sup>ti</sup> in the OER was first clearly<br>that the electronic changes at fe site are not due to  $LDI$  letcherocally th(*E\*F*(*L Electrochom.*) 2022, 28(3), 2108541 (7 of 31)<br>
tharing a redox reaction, Corrigan et al. conducted an The presence of Fe<sup>\*</sup> in the OFR was first clearly<br>
in-sini<sup>7</sup>Fe Mössbauer spectroscopic study<sup>64</sup>. They during a redox reaction, Corrige and at conducted and more resumed to the interaction of  $\mu$  and  $\mu$  and  $\mu$  as the sites for  $\mu$  and  $\$ during a redox reaction, Corrigan et al. conducted an<br>
The presence of Fe<sup>t+</sup> in the OER was first electry<br>
in-site <sup>2Fe</sup> Mossbauer spectroscopie study<sup>56</sup>. They staid observed in 2015 when Stahl et al. twentigated NiFe-<br> to the host lat-<br>
operando <sup>57</sup>Fe Mössbauer technique to track the Fe<br>
r., an intimate coidation state in 3:1 NiFe-LDH and Fe oxide cata-<br>
ic scale rather lysts, while polarizing them in the OER zone. Inter-<br>
OOH particle (3), 2108541 (7 of 31)<br>The presence of Fe<sup>4+</sup> in the OER was first clearly<br>served in 2015 when Stahl et al. investigated NiFe-<br>DH electrocatalyst during OER<sup>[47]</sup>. They used *in-situl*<br>*erando* <sup>57</sup>Fe Mössbauer technique (28(3), 2108541 (7 of 31)<br>The presence of Fe<sup>4+</sup> in the OER was first clearly<br>observed in 2015 when Stahl et al. investigated NiFe-<br>LDH electrocatalyst during OER<sup>[47]</sup>. They used *in-situl*<br>operando <sup>57</sup>Fe Mössbauer tech  $(28(3), 2108541 (7 of 31))$ <br>
The presence of Fe<sup>4+</sup> in the OER was first clearly<br>
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The presence of Fe<sup>4+</sup> in the OER was first clearly<br>
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The presence of Fe<sup>4+</sup> in the OER was first clearly<br>
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The presence of Fe<sup>4+</sup> in the OER was first clearly<br>
observed in 2015 when Stahl et al. investigated NiFe-<br>
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operando <sup>57</sup>Fe Mössbauer t . 28(3), 2108541 (7 of 31)<br>
The presence of Fe<sup>4+</sup> in the OER was first clearly<br>
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The presence of Fe<sup>4+</sup> in the OER was first clearly<br>
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The presence of Fe<sup>4+</sup> in the OER was first clearly<br>
observed in 2015 when Stahl et al. investigated NiFe-<br>
LDH electrocatalyst during OER<sup>[47]</sup>. They used *in-situl*<br> *operando* <sup>57</sup>Fe Mössba 28(3), 2108541 (7 of 31)<br>
The presence of Fe<sup>4+</sup> in the OER was first clearly<br>
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The presence of Fe<sup>4+</sup> in the OER was first clearly<br>
observed in 2015 when Stahl et al. investigated NiFe-LDH electrocatalyst during OER<sup>[47]</sup>. They used *in-situl*<br> *operando* <sup>57</sup>Fe Mössbauer The presence of Fe<sup>4+</sup> in the OER was first clearly<br>observed in 2015 when Stahl et al. investigated NiFe-<br>LDH electrocatalyst during OER<sup>[47]</sup>. They used *in-situl*<br>operando <sup>57</sup>Fe Mössbauer technique to track the Fe<br>oxid The presence of Fe<sup>4+</sup> in the OER was first clearly<br>observed in 2015 when Stahl et al. investigated NiFe-<br>LDH electrocatalyst during OER<sup>[47]</sup>. They used *in-situl*<br>operando <sup>57</sup>Fe Mössbauer technique to track the Fe<br>oxid observed in 2015 when Stahl et al. investigated NiFe-<br>LDH electrocatalyst during OER<sup>[47]</sup>. They used *in-situl*<br>operando <sup>57</sup>Fe Mössbauer technique to track the Fe<br>oxidation state in 3:1 NiFe-LDH and Fe oxide cata-<br>lysts and the divergence of the signify-<br>
and Fe intervals and the signify-<br>
dechnique to track the Fe<br>
-LDH and Fe oxide cata-<br>
in the OER zone. Inter-<br>
e 5, the existence of Fe<sup>4+</sup><br>
rization and under OER<br>
with increasing pot LDH electrocatalyst during OER<sup>[67]</sup>. They used *in-situl*<br>operando <sup>57</sup>Fe Mössbauer technique to track the Fe<br>oxidation state in 3:1 NiFe-LDH and Fe oxide cata-<br>lysts, while polarizing them in the OER zone. Inter-<br>esting g OER<sup>[47]</sup>. They used *in-situl*<br>technique to track the Fe<br>e-LDH and Fe oxide cata-<br>m in the OER zone. Inter-<br>arization and under OER<br>d with increasing potential.<br>Fe oxide did not show any<br>demonstrating that Fe<sup>4+</sup> can<br>D *operando* <sup>57</sup>Fe Mössbauer technique to track the Fe oxidation state in 3:1 NiFe-LDH and Fe oxide cata-<br>lysts, while polarizing them in the OER zone. Inter-<br>estingly, as shown in Figure 5, the existence of Fe<sup>4+</sup><br>was con oxidation state in 3:1 NiFe-LDH and Fe oxide cata-<br>lysts, while polarizing them in the OER zone. Inter-<br>estingly, as shown in Figure 5, the existence of Fe<sup>4+</sup><br>was confirmed upon polarization and under OER<br>conditions, whi lysts, while polarizing them in the OER zone. Inter-<br>estingly, as shown in Figure 5, the existence of Fe<sup>4+</sup><br>was confirmed upon polarization and under OER<br>conditions, which increased with increasing potential.<br>They found 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (7 of 31)<br>
un et al. conducted an The presence of Fe<sup>4+</sup> in the OER was first clearly<br>
ppic study<sup>[46]</sup>. They said observed in 2015 when Stahl et al. investigated NiFe-<br>
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 $\# \&\#(J. \text{Electrochem.}) 2022, 28(3), 2108541 (8 of 31)$ <br>
-0.27 mm ·s<sup>-1</sup> appeared, which indicates 12% of the potential active locations<br>
Fe sites shown in Figure 5(D). Further increasing the give any clear understand<br>
potential  $\text{H}(k\#(J. \text{Electrochem.}) 2022, 28(3), 2108541 (8 of 31)$ <br>-0.27 mm ·s<sup>-1</sup> appeared, which indicates 12% of the potential active locations if they exist, but could not<br>Fe sites shown in Figure 5(D). Further increasing the give any **ELET ELET THE EXECUTE CONDUCE 1.5 V (ELET THE EXECUTE THE SET ON A SSET ON A STRONGET PEAK AS THE SOLUTION OF** (*Electrochem.*) 2022, 28(3), 2108541 (8 of 31)<br>  $-0.27 \text{ mm} \cdot \text{s}^{-1}$  appeared, which indicates 12% of the<br>
potential active locations if they exist, but could not<br>
Fe sites shown in Figure 5(D). Further increasing the<br> Fe (Figure 5(E). After 48 hours winducture applied potential active locations if they exist, but could not<br>Fe sites shown in Figure 5(D). Further increasing the give any clear understanding about the role of Fe dur-<br>poten **EVALUATE 1.19 V** Electrochem.) 2022, 28(3), 2108541 (8 of 31)<br> **1.927** mm ·s<sup>1</sup> appeared, which indicates 12% of the potential active locations if they exist, but could not<br>
Fe sites shown in Figure 5(D). Further increa

a doublet  $(\delta = 0.36 \sim 0.37$  mm·s<sup>-1</sup>,  $\Delta = 0.64 \sim 0.67$  whether Ni is active sites or Fe is active sites.  $mm \cdot s^{-1}$ ). The significant feature of NiFe catalyst was potential to 1.76 V generates a stronger peak for Fe ing OER. XAS results showed that the loxidation corresponding to almost 21% of the total Fe-O in NiFe-based catalysts was signife (Figure 5(E). After bringing back the oxidation corresponding to almost 21% of the total<br>
Fe (Figure 5(E)). After bringing back the potential to<br>
Fe (Figure 5(E)). After bringing back the potential to<br>
the than that of Fe<sup>3</sup><sup>-</sup>O, which indi<br>
1.49 V, the curre Fe (Figure 5(Fi)). After hringing hack the potential to<br>
are than that of Fe<sup>3</sup>-O, which indicated that Fe should<br>
1.49 V, the current density dropped to bassline, but the in higher value<br>
Fe oxidation peak appeared in Mi 1.49 V, the current density dropped to bascline, but<br>
be in higher valence states during the OER process,<br>
Fe coxidation peak appeared in Mossbaucr spectrum and supported the result of *tin-situalogramada* Moss-<br>
accounti Fe oxidation peak appeared in Mossbauer spectrum and supported the results of *in-situ/operando* Miss-<br>encounting for almost - 20% of total Fe as shown in bauer suddes<sup>59</sup>. This work gave a strong and clear in-<br>Figure 5(F accounting for almost ~ 20% of total Fe as shown in<br>
Figure 5(<sup>50</sup>). This work gave a strong and clear in-<br>
Figure 5(F). After 48 houst without any applied potential correle existence of high values ein to which<br>
Figure 5 Figure 5(F). After 48 hours without any applied poten-<br>
tigation for the existence of high valance iron which<br>
tial the Fe oxidation peak disappeared (Figure 5(C)). survely presents in the system and may have a enciral<br>
c tial, the Fe oxidation peak disappeared (Figure 5(G)). surely presents in the system and may have a crucial<br>
On the other hand, in the case of <sup>P</sup>Fe-carinched hy-<br>
role in OER, but not much clear. The results of xy-<br>
drou On the other hand, in the case of "Fe-enriched hy-<br>role in OER, but not much clear. The results of oxy-<br>throw is equided to to show to the sheavier such a behavior gen intermediates such a loss ousported the presence<br>in M drous Fe oxide, they did not observe such a behavior<br>
in intermediates study also supported the presence<br>
in Missbauer spectra under all the conditions as orbigh-valent iron<sup>691</sup>. However, there is still debate on<br>
in Sim in Mössbauer spectra under all the conditions as<br>so frigh-valent iron<sup>tio</sup>! However, there is still debate on<br>shown in Figure 5(11-1). And the spectra showed only the mechanism of Nie'e catalyst during OER reaction,<br>a dou shown in Figure 5(H-J). And the spectra showed only<br>
the mechanism of NiFe catalyst during OER reaction,<br>
a double ( $\delta = 0.36 - 0.37$  mm · s<sup>-1</sup>,  $\Delta = 0.64 - 0.67$  whether Ni is active sites. The is active sites.<br>
mm · s<sup>-1</sup> a doublet ( $\delta = 0.36 - 0.37$  mm ·s',  $\Delta = 0.64 - 0.67$  whether Ni is active sites or Fe is active sites.<br>
mm ·s'). The significant fracture of NiFe catalyst was<br>
the appearance of Fe oxidation peak under an applied (NiFe-PB mm •s<sup>+</sup>). The significant feature of NiFe catalyst was<br>
that papearance of Fe oxidation peak was fitted cithe<sup>2</sup>PBAs) were suited for OER through  $\hat{m} \cdot \sin \hat{m}$ <br>
peatences of Fe oxidation peak was fitted cither as  $\omega_{$ the appearance of Fe oxidation peak under an applied (NiFe-PBAs) were studied for OER through *in-situ/*<br>potential ( $\simeq 1.5$  V). This rew peak was fitted citier as *operarado* XAS technique<sup>48</sup>. It was observed that NiFe potential ( $\geq 1.5$  V). This new peak was fitted either as *operando* XAS technique<sup>ns</sup>. It was observed that NiFe-<br>a singlet ( $\beta = 0.27$  rm  $\cdot$ s;  $\gamma$ ) or as a double ( $\beta = 0.0$ ) PBA was first transformed into Ni(OfF), a singlet ( $\delta = -0.27$  mm ·s") or as a doublet ( $\delta = 0.0$  PBA was first transformed into Ni(OH)<sub>2</sub> and then into<br>and  $\Delta = 0.58$  mm ·s"), and is consistent with the exi-<br>bifolofl<sub>5</sub>, under the applied potential due to depro and  $\Delta = 0.58$  mm -s"), and is consistent with the exi-<br>
dizzed Fe species as Fe<sup>1</sup>. This was the first direct evi-<br>
direct div-<br>
dizzed Fe species as Fe<sup>1</sup>. This was the first direct evi-<br>
denote of the Fe<sup>4+</sup> formation dized Fe species as Fe\*. This was the first direct evi-<br>nation, which showed an excellent activity at very<br>denote of the Fe<sup>+</sup> formation under reaction conditions. I low overpotential. They called the deprotonation pro-<br>F dence of the Fe<sup>6</sup> formation under reaction conditions. low overpotential. They called the deprotonation pro-<br>Furthermore, the presence of these fe<sup>4</sup> species seen as a reversible process which partially generated<br>differ Furthermore, the presence of these Fe<sup>4+</sup> species even cess a reversible process which partially generated<br>after lowering the potential where the activity Wi<sup>7</sup> as a result of charge compensation due to depro-<br>dropped to after lowering the potential where the activity  $N_i^{th}$  as a result of charge compensation due to deproduct<br>dropped to baseline indicated that these species were contaion. In other words,  $\hat{m}_{\text{eff}}$  are<br>not directly res dropped to haseline indicated that these species were<br>
tonation. In other words, *in-sinu* generated catalyst<br>
polaring a key role in enhancing the OER activity with NOOH<sub>2</sub>, during OER was considered as responsible<br>
play not directly responsible for the observed activity but<br>
DiOOH<sub>2*a*</sub> during OFR was considered as responsible<br>
playing a key role in rehancing the OBR activity. For higher activity which can be transformed reverse-<br>
This d playing a key role in enhancing the OFR activity. for higher activity which can be transformed reverse-<br>This discovery showed that Fe<sup>+</sup> in Nife-LDH lattice  $|V|$  D Ni(OH), This indicated that the structure of the<br>is redo

<sup>-1</sup> appeared, which indicates 12% of the potential active locations if they exist, but could not when in Figure 5(D). Further increasing the give any clear understanding about the role of Fe dur-<br>1.76 V generates a stron H( $E\mathcal{L}(L\left|Electrochem. \right)$  2022, 28(3), 2108541 (8 of 31)<br>
-0.27 mm ·s<sup>-1</sup> appeared, which indicates 12% of the potential active locations if they exist, but could not<br>
Fe sites shown in Figure 5(D). Further increasing the g **EVALUATION 1989**<br> **EVALUAT THE SECT ASSAME AND SET ASSAME AND SET ASSAMED THE SET ASSAMED THE SET ASSAMEL AND AND SET ASSAMEL AND THE SET ASSAMEL AND SET ASSAMEL AND SET AS SET AND SET AND SET AND SET AND SET AND SET AND** the  $\mathcal{H}_2^{\text{eff}}(L)$  *Electrochem.*) 2022, 28(3), 2108541 (8 of 31)<br>
-0.27 mm ·s<sup>1</sup> appeared, which indicates 12% of the potential active locations if they exist, but could not<br>
Fc sites shown in Figure 5(D). Further in **HE OF (***L* Electrochem.) 2022, 28(3), 2108541 (8 of 31)<br> **The Fe sites shown** in Figure 5(D). Further increasing the give any clear trace for they exist, but could not<br>
Fe sites shown in Figure 5(D). Further increasing  $\frac{1}{2}$ We  $\frac{1}{2}$  ( $\frac{1}{2}$   $\frac{1}{2$ the  $\mathcal{Z}(L$  Electronobem.) 2022, 28(3), 2108541 (8 of 31)<br>  $\Phi(E\mathcal{Z}(L)E(1),E(2))$  and  $\Phi(E)$  are points of the points of the points of the such **in the spectra unit of the spectra unit of the condition of the spectra unit of the spectra unit and the spectra unit of the spectra unit of**  $4E \approx (LE \times (E \times (E \times (E \times E)))$ <br>
4.0.27 mm ·s<sup>-1</sup> appeared, which indicates 12% of the potential active locations if they exist, but could not<br>
Fe sites shown in Figure 5(D). Further increasing the give any clear understanding a **EVALUATION THE (** $E/T$ , *D. Electrochem*, 2022, 28(3), 2108541 (8 of 31)<br>
4.0.27 mm ·s<sup>1</sup> appeared, which indicates 12% of the potential active locations if they exist, but cours Fe sites shown in Figure 5(D). Further inc  $\frac{126}{12}$  *Chechelem*.) 2022, 28(3), 2108541 (8 of 31)<br>
icates 12% of the potential active locations if they exist, but could not<br>
ther increasing the give any clear understanding about the role of Fe dur-<br>
onger peak mm •s' appeared, which indicates 12% of the potential acity correspondent in they exist, but could not as shown in Figure 5(D). Further increasing the pive any clear values in the pive any clear of NiFe catalyst was signi -0.27 mm •s<sup>1</sup> appeared, which indicates 12% of the potential active locations if they exist, but could not existles shown in Figure 3(D). Further increasing the give any clear anderstanding about the role of Fe durescent Fe sites shown in Figure 5(D). Further increasing the give any clear understanding about the role of Fe dur-<br>potential to 1.76 V generates a stronger peak for Fe ing OER. XAS results showed that the botal consideration or ates a stronger peak for Fe ing OER. XAS results showed that the bond length of<br>
almost 21% of the total Fe-O in NiFe-based catalysts was significantly short-<br>
arging back the potential to erthan that of Fe<sup>3</sup>-O, which in ding to almost 21% of the total<br>
Fe-O in NiFe-based catalysts was significantly short-<br>
ther bringing back the potential to er than that of Fe<sup>3</sup>-O, which indicated that Fe should<br>
density droped to baseline, but be in hi 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was 28(3), 2108541 (8 of 31)<br>potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was Let the strainer of DER. XAS results showed that the bond length of -O in NiFe-based catalysts was significantly short-<br>than potential active locations if they exist, but could not<br>give any clear understanding about the role of Fe dur-<br>ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was significantly short-<br>er t give any clear understanding about the role of Fe during OER. XAS results showed that the bond length of Fe-O in NiFe-based catalysts was significantly shorter than that of Fe<sup>3+</sup>-O, which indicated that Fe should be in h ing OER. XAS results showed that the bond length of<br>Fe-O in NiFe-based catalysts was significantly short-<br>er than that of Fe<sup>3+</sup>-O, which indicated that Fe should<br>be in higher valence states during the OER process,<br>and su Fe-O in NiFe-based catalysts was significantly shorter than that of Fe<sup>3+</sup>-O, which indicated that Fe should be in higher valence states during the OER process, and supported the results of *in-situloperando* Möss-bauer s er than that of Fe<sup>3+</sup>-O, which indicated that Fe should<br>be in higher valence states during the OER process,<br>and supported the results of *in-situloperando* Möss-<br>bauer studies<sup>[43]</sup>. This work gave a strong and clear in-电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (8 of 31)<br>indicates 12% of the potential active locations if they exist, but could not<br>Further increasing the give any clear understanding about the role of Fe dur-<br>stronger pe

be in higher valence states during the OER process,<br>and supported the results of *in-situloperando* Möss-<br>bauer studies<sup>[43]</sup>. This work gave a strong and clear in-<br>dication for the existence of high valance iron which<br>su and supported the results of *in-situ/operando* Möss-<br>bauer studies<sup>[48]</sup>. This work gave a strong and clear in-<br>dication for the existence of high valance iron which<br>surely presents in the system and may have a crucial<br>r bauer studies<sup>(45</sup>). This work gave a strong and clear in-<br>dication for the existence of high valance iron which<br>surely presents in the system and may have a crucial<br>role in OER, but not much clear. The results of oxy-<br>ge dication for the existence of high valance iron which<br>surely presents in the system and may have a crucial<br>role in OER, but not much clear. The results of oxy-<br>gen intermediates study also supported the presence<br>of high-v surely presents in the system and may have a crucial<br>role in OER, but not much clear. The results of oxy-<br>gen intermediates study also supported the presence<br>of high-valent iron<sup>[48]</sup>. However, there is still debate on<br>th role in OER, but not much clear. The results of oxy-<br>gen intermediates study also supported the presence<br>of high-valent iron<sup>[48]</sup>. However, there is still debate on<br>the mechanism of NiFe catalyst during OER reaction,<br>whe gen intermediates study also supported the presence<br>of high-valent iron<sup>1481</sup>. However, there is still debate on<br>the mechanism of NiFe catalyst during OER reaction,<br>whether Ni is active sites or Fe is active sites.<br>Later of high-valent iron<sup>tag</sup>. However, there is still debate on<br>the mechanism of NiFe catalyst during OER reaction,<br>whether Ni is active sites or Fe is active sites.<br>Later on in 2018, NiFe Prussian blue analogues<br>(NiFe-PBAs) the mechanism of NiFe catalyst during OER reaction,<br>whether Ni is active sites or Fe is active sites.<br>Later on in 2018, NiFe Prussian blue analogues<br>(NiFe-PBAs) were studied for OER through *in-situl*<br>operando XAS techniq whether Ni is active sites or Fe is active sites.<br>
Later on in 2018, NiFe Prussian blue analogues<br>
(NiFe-PBAs) were studied for OER through *in-situl*<br>
operando XAS technique<sup>[49]</sup>. It was observed that NiFe-<br>
PBA was fir Later on in 2018, NiFe Prussian blue analogues<br>(NiFe-PBAs) were studied for OER through *in-situl*<br>operando XAS technique<sup>[69]</sup>. It was observed that NiFe-<br>PBA was first transformed into Ni(OH)<sub>2</sub> and then into<br>NiOOH<sub>2</sub><sub>a</sub> (NiFe-PBAs) were studied for OER through *in-situl*<br>operando XAS technique<sup>1695</sup>. It was observed that NiFe-<br>PBA was first transformed into Ni(OH)<sub>2</sub> and then into<br>NiOOH<sub>2<sup>x</sup></sub> under the applied potential due to deproto-<br>n *operando* XAS technique<sup>469</sup>. It was observed that NiFe-<br>PBA was first transformed into Ni(OH)<sub>2</sub> and then into<br>NiOOH<sub>2<sup>*m*</sub> under the applied potential due to deproto-<br>nation, which showed an excellent activity at very<br></sub></sup> A was first transformed into Ni(OH)<sub>2</sub> and then into OOH<sub>2<sub>2</sub> under the applied potential due to deproto-<br>ion, which showed an excellent activity at very<br>v overpotential. They called the deprotonation pro-<br>ss a reversible</sub> NiOOH<sub>2x</sub> under the applied potential due to deproto-<br>nation, which showed an excellent activity at very<br>low overpotential. They called the deprotonation pro-<br>cess a reversible process which partially generated<br>Ni<sup>4+</sup> as nation, which showed an excellent activity at very<br>low overpotential. They called the deprotonation pro-<br>cess a reversible process which partially generated<br>Ni<sup>4+</sup> as a result of charge compensation due to depro-<br>tonation low overpotential. They called the deprotonation process a reversible process which partially generated Ni<sup>++</sup> as a result of charge compensation due to deprotonation. In other words, *in-situ* generated catalyst NiOOH<sub>2*</sub>* cess a reversible process which partially generated Ni<sup>4+</sup> as a result of charge compensation due to deprotonation. In other words, *in-situ* generated catalyst NiOOH<sub>2</sub><sup>-</sup> aduring OER was considered as responsible for hi Ni<sup>++</sup> as a result of charge compensation due to deprotonation. In other words, *in-situ* generated catalyst NiOOH<sub>2*x*</sub> during OER was considered as responsible for higher activity which can be transformed reversely to N tonation. In other words, *in-situ* generated catalyst NiOOH<sub>2x</sub> during OER was considered as responsible for higher activity which can be transformed reverse-ly to Ni(OH)<sub>2</sub>. This indicated that the structure of the cata NiOOH<sub>2*x*</sub> during OER was considered as responsible<br>for higher activity which can be transformed reverse-<br>ly to Ni(OH)<sub>2</sub>. This indicated that the structure of the<br>catalyst cannot be stabilized under reaction condition<br>a

for higher activity which can be transformed reverse-<br>ly to Ni(OH)<sub>2</sub>. This indicated that the structure of the<br>catalyst cannot be stabilized under reaction condition<br>and could be changed by applying potential. In fact,<br>t

# Electrocatalysts

OER catalyst. The stability of the structure under OER *erando* techniques can provide infor<br>conditions is also very important because the stability<br>structural and electronic states of  $\uparrow$ <br>of the catalyst structure unde



 $\frac{\text{#L}\#(J. \text{Electrochem.}) 2022, 28(3), 2108541 (9 of 31)}{\text{failed to fully explain the underlying mechanism for}}$ failed to fully explain the underlying mechanism for monitor the OER process in order to identify reaction<br>
OER. Moreover, we believe that the higher activity  $\frac{\text{tL} \left(\frac{1}{2} \mathcal{L}_2 \mathcal{L}_3 \mathcal{L}_4 \mathcal{L}_5 \mathcal{L}_5 \mathcal{L}_6 \mathcal{L}_7 \mathcal{L}_7 \mathcal{L}_8 \mathcal{L}_7 \mathcal{L}_8 \mathcal{L}_8 \mathcal{L}_9 \mathcal{L}_1 \mathcal{L}_2 \mathcal{L}_2 \mathcal{L}_3 \mathcal{L}_3 \mathcal{L}_1 \mathcal{L}_3 \mathcal{L}_2 \mathcal{L}_3 \mathcal{L}_3 \mathcal{L}_1 \mathcal{L}_2 \mathcal{L}_3 \mathcal{L}_2 \mathcal{L}_3 \mathcal{L}_1$  $\exists t \in \mathcal{H}$ . *Electrochem.*) 2022, 28(3), 2108541 (9 of 31)<br>
failed to fully explain the underlying mechanism for<br>
OER. Moreover, we believe that the higher activity is<br>
intermediates and electrochemical activity on the  $\frac{d}{dt}\mathcal{L}^{\infty}(J. Electrochem.)$  2022, 28(3), 2108541 (9 of 31)<br>
failed to fully explain the underlying mechanism for<br>
OER. Moreover, we believe that the higher activity is<br>
of monitor the OER process in order to identify reacti **EVALUATE 1988**<br> **EVALUATE 1988**<br> **EVALUATE 1999**<br> **EVALUATE 1999**<br> **EVALUATE 1999**<br> **EVALUATE 1999**<br> **EVALUATE 1999**<br> **OER.** Moreover, we believe that the higher activity is<br> **INCOTENTE 1999**<br> **OER** conditions is also ve  $\pm \frac{\mu_0 \pm \frac{\mu_0 \pm \mu_0}{2}}{2}$ <br>
failed to fully explain the underlying mechanism for<br>
for the catalyst streme<br>
of the catalyst structure intervals and electrochemical activity on the sur-<br>
crucial, but it should not be th **EVALUATE (EXECT)**<br> **EVALUATE CONSTER (EXECT)** 19022, 28(3), 2108541 (9 of 31)<br> **EVALUATE CONSTER CONSTER APPLICATE:**<br> **EVALUATE CONSTER CONSTER CONSTER CONSTERNATE APPLICATE:**<br> **CER** catalyst The stability of the structu  $\pm$  The General applications and **CEP CHACE CORP CORP CORP**<br> **Experimentation** of the Section of the GEN process in order to identify reaction<br>
OER. Moreover, we believe that the higher activity is intermediates and elec  $\pm$  (*E* Electrochem.) 2022, 28(3), 2108541 (9 of 31)<br>
d to fully explain the underlying mechanism for<br>
content to CER process in order to identify reaction<br>
2. Moreover, we believe that the higher activity is<br>
intermedi the *C*  $E^2$  (*L Electrochem.*) 2022, 28(3), 2108541 (9 of 31)<br>
and to fully explain the underlying mechanism for<br>
R. Moreover, we believe that the higher activity is<br>
intermediates and electrochemical activity on the the paster of the particular method is the paster of the particular of the paster such as Tafel slopes and electrochemical impedance  $\frac{4}{3}E^2\pi J. Electrochem > 2022, 28(3), 2108541 (9.631) \n$ \nfinite to fully explain the underlying mechanism for  
\nOER. Moreover, we believe that the higher activity is  
\nintermediates and electrochemical activity on the sur-  
\nericial, but it should not be the only concern for any  
\nfree of OFCs during the OFRE<sup>28,231</sup>. These in-stubip-  
\nOER has the ability of the structure under OER containing the  
\nconditions is also very important because the stability  
\nstubipertaut and electroton states of Nife-based elec-  

finited to fully explain the understandant someonic someonic control.<br>
DER. Moreover, we believe that the higher activity is intermediates and electrochemical activity on the sur-<br>
critical, but it should not be the only failed to fully explain the underlying mechanism for<br>
colffic. Moreover, we belicut that higher activity is intermediates and electrochermic articuly<br>
credit to the mechanism of the mechanism of the summer of of OECs duri (28(3), 2108541 (9 of 31)<br>monitor the OER process in order to identify reaction<br>intermediates and electrochemical activity on the sur-<br>face of OECs during the OER<sup>[52-53]</sup>. These *in-situ/op-*<br>*erando* techniques can prov (28(3), 2108541 (9 of 31)<br>
monitor the OER process in order to identify reaction<br>
intermediates and electrochemical activity on the sur-<br>
face of OECs during the OER<sup>[52-53]</sup>. These *in-situ/op-*<br> *erando* techniques can  $f(28(3), 2108541 (9 of 31))$ <br>monitor the OER process in order to identify reaction<br>intermediates and electrochemical activity on the sur-<br>face of OECs during the OER<sup>[52-53]</sup>. These *in-situ/op-*<br>*erando* techniques can provi  $(28(3), 2108541 (9 of 31))$ <br>
monitor the OER process in order to identify reaction<br>
intermediates and electrochemical activity on the sur-<br>
face of OECs during the OER<sup>[52-53]</sup>. These *in-situ/op-*<br> *erando* techniques can pr  $(28(3), 2108541 (9 of 31))$ <br>monitor the OER process in order to identify reaction<br>intermediates and electrochemical activity on the sur-<br>face of OECs during the OER<sup>[52-53</sup>]. These  $in-situ/op-$ <br>*erando* techniques can provide in  $(28(3), 2108541 (9 of 31))$ <br>monitor the OER process in order to identify reaction<br>intermediates and electrochemical activity on the sur-<br>face of OECs during the OER<sup>[52-53]</sup>. These  $in-situ/op-$ <br>*erando* techniques can provide in  $(28(3), 2108541 (9 of 31))$ <br>monitor the OER process in order to identify reaction<br>intermediates and electrochemical activity on the sur-<br>face of OECs during the OER<sup>[52-53]</sup>. These *in-situlop-*<br>erando techniques can provide  $(28(3), 2108541 (9 of 31))$ <br>
monitor the OER process in order to identify reaction<br>
intermediates and electrochemical activity on the sur-<br>
face of OECs during the OER<sup>[52-53]</sup>. These *in-situlop-*<br> *erando* techniques can pr 28(3), 2108541 (9 of 31)<br>
monitor the OER process in order to identify reaction<br>
intermediates and electrochemical activity on the sur-<br>
face of OECs during the OER<sup>(58,58)</sup>. These *in-situlop-*<br> *erando* techniques can p mechanism<sup>[54]</sup>.  $\frac{8(3)}{2}$ , 2108541 (9 of 31)<br>
onitor the OER process in order to identify reaction<br>
ermediates and electrochemical activity on the sur-<br>
ce of OECs during the OER<sup>[353]</sup>. These *in-situlop-*<br> *ndo* techniques can provi  $(28(3), 2108541 (9 of 31))$ <br>monitor the OER process in order to identify reaction<br>intermediates and electrochemical activity on the sur-<br>face of OECs during the OER<sup>[23:83]</sup>. These *in-situlop-*<br>*erando* techniques can provid  $(28(3), 2108541 (9 of 31))$ <br>monitor the OER process in order to identify reaction<br>intermediates and electrochemical activity on the sur-<br>face of OECs during the OER<sup>[25,53]</sup>. These *in-situ/op-*<br>*erando* techniques can provid  $(28(3), 2108541 (9 of 31))$ <br>
monitor the OER process in order to identify reaction<br>
intermediates and electrochemical activity on the sur-<br>
face of OECs during the OER<sup>(32,51</sup>). These *in-situlop-*<br> *erando* techniques can pr **Electrochemical systems** under the GER process in order to identify reaction<br>intermediates and electrochemical activity on the sur-<br>face of OECs during the OER<sup>(25,5)</sup>. These *in-situ/op-*<br>*erando* techniques can provide monitor the OER process in order to identify reaction<br>intermediates and electrochemical activity on the sur-<br>face of OECs during the OER<sup>(32-53)</sup>. These *in-situ/op-*<br>*erando* techniques can provide information about the<br> 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (9 of 31)<br>
rlying mechanism for monitor the OER process in order to identify reaction<br>
the higher activity is intermediates and electrochemical activity on the sur-<br>
only conce

Figure 6 Selected in-situ/operando spectroscopy (APXPS),<br>
miques includes two regions: X-ray absorption near the degreessure X-ray photoelectron spectroscopy (APXPS),<br>
Missishaure represented Service (XANES) and extended The mathemative control of the structure (XANES) structure (XANES) and extended X-ray absorption<br>pressure X-ray photoelectron spectroscopy, (APXPS). structure (KXAFS). Combing with the in-sinulation<br>or the structure the st The street of the systems and the section spectroscopy (DV-KVEN) structure (XANES) and extended X-ray absorption<br>
Mosshauer spectroscopy, DEMS, and surface interrogation scanning operator  $N_{\text{He}}S_{\text{He}}$ COH cetallytic in Mossbauer spectroscopy, UV-visible (UV-visi spectroscopy in structure (FXAFS). Combing with the *in-sinul* troscopy, DFMS, and surface interrogation scanning  $\delta_{\text{perc}}$  and  $\delta_{\text{H}}$ . Complement and a  $\gamma$ -NiOOH structu Eigene (Electrochanical microscopy) (SI-SFCM) were used to  $\frac{1}{2}$  a low potential and a y-NiOOH structure at a high the electrochanical microscopy (SI-SFCM) structure as a low potential and a y-NiOOH structure at a high OER. Moreover, we believe that the higher activity is<br>
intermediates and electrochemical activity on the sur-<br>
critical, but it should not be the only concern for any  $\alpha$  from the COEC during the OFR(BE size in structura erucial, but it should not be the only concern for any<br>
once of OECs during the OER<sup>103.51</sup>. These *in-situlop*-<br>
OER cealiby state through or the state of the state of the calibative state of the calibative state of the conditions is also very important because the stability<br>of the catalyst structural and electronic states of NiFe-based elec-<br>of the catalyst structure under OER conditions is erru-<br>trooks. Figure 6 graphically shows sever of the catalyst structure under OER conditions is eru-<br>
trodes. Figure 6 graphically shows several selected<br>
cial for commercial applications.<br> **3 The General Introduction of**  $In_2$  minis we been such the been successfull cial for commercial applications.<br> **3 The General Introduction of**  $In-$  niques which have been successfully applied to cap-<br> **5***itul0perando* **Techniques for Cha-** have been successively applied to cap-<br> **Electrocatalysts 3 The General Introduction of**  $I_n$  inques which have been successfully applied to cap-<br> **Situl/Operando Techniques for Cha**<br>
are species, investigating the OER pathways and<br> **racterizing the NiFe-Based OER**<br>
In the past **Situ/Operando Techniques for Cha-**<br> **Examplementary and the NiFe-Based OER**<br> **Examplementary and the NiFe-Based OER**<br> **Electrocatalysts Examplementary could be used to observe vi-<br>
In the past, traditional electrochem Example 10.1** and **CEC Example 10.1 and Example 10.1 and** *M* **Expectises, investigating the OER pathways and <b>Electrocatalysts** momanim spectroscopy could be used to observe vi-<br>
In the past, traditional electrochemic **TREETIZING THE NIFE-Based OEK**<br> **Encorations** concerned technismi<sup>164</sup>.<br> **Encorational subset of the confident interrogation scanne interrogation scanne and the NiFe catalysts<sup>101</sup>. The** *in-situloperando* **Raman spe-<br>
suc Electrocatalysts**<br>
En the past, traditional electrochemical techniques<br>
In the past, traditional electrochemical impedance<br>
In the past reading that of the intermediates durangery modes of<br>
such as Tafel slopes and elect intermediates and electrochemical activity on the sur-<br>face of OECs during the OER<sup>(32-83)</sup>. These *in-situ/op-*<br>*erando* techniques can provide information about the<br>structural and electronic states of NiFe-based elec-<br>t face of OECs during the OER<sup>[32,53</sup>]. These *in-situ/op-*<br> *erando* techniques can provide information about the<br>
structural and electronic states of NiFe-based elec-<br>
trodes. Figure 6 graphically shows several selected<br> *erando* techniques can provide information about the<br>structural and electronic states of NiFe-based elec-<br>trodes. Figure 6 graphically shows several selected<br>in-situ/operando spectroscopic characterization tech-<br>niques wh structural and electronic states of NiFe-based electrodes. Figure 6 graphically shows several selected in-situ/operando spectroscopic characterization techniques which have been successfully applied to capture the NiFe-bas trodes. Figure 6 graphically shows several selected<br>in-situ/operando spectroscopic characterization tech-<br>niques which have been successfully applied to cap-<br>ture the NiFe-based OER electrocatalytic intermedi-<br>ate species in-situ/operando spectroscopic characterization techniques which have been successfully applied to capture the NiFe-based OER electrocatalytic intermediate species, investigating the OER pathways and mechanism<sup>[54]</sup>. Rama miques which have been successfully applied to cap-<br>ture the NiFe-based OER electrocatalytic intermedi-<br>ate species, investigating the OER pathways and<br>mechanism<sup>[54]</sup>.<br>Raman spectroscopy could be used to observe vi-<br>brat ture the NiFe-based OER electrocatalytic intermediate species, investigating the OER pathways and mechanism<sup>[64]</sup>.<br>
Raman spectroscopy could be used to observe vi-<br>
bration, rotational, and other low frequency modes of th ate species, investigating the OER pathways and<br>mechanism<sup>[54]</sup>.<br>Raman spectroscopy could be used to observe vi-<br>bration, rotational, and other low frequency modes of<br>the NiFe catalysts<sup>155</sup>. The *in-situloperando* Raman mechanism<sup>[54]</sup>. Raman spectroscopy could be used to observe vibration, rotational, and other low frequency modes of the NiFe catalysts<sup>[55]</sup>. The *in-situloperando* Raman spectroscopy could monitor the intermediates duri Raman spectroscopy could be used to observe vi-<br>bration, rotational, and other low frequency modes of<br>the NiFe catalysts<sup>[58]</sup>. The *in-situloperando* Raman spectroscopy could monitor the intermediates during the<br>electroc bration, rotational, and other low frequency modes of<br>the NiFe catalysts<sup>1851</sup>. The *in-situloperando* Raman spectroscopy could monitor the intermediates during the<br>electrochemical systems under the applying test volt-<br>ag the NiFe catalysts<sup>551</sup>. The *in-situloperando* Raman spectroscopy could monitor the intermediates during the electrochemical systems under the applying test voltage in aqueous media, which could provide real-time reactio etroscopy could monitor the intermediates during the<br>electrochemical systems under the applying test volt-<br>age in aqueous media, which could provide real-time<br>reaction information. This technique helps to under-<br>stand how electrochemical systems under the applying test volt-<br>age in aqueous media, which could provide real-time<br>reaction information. This technique helps to under-<br>stand how an electrochemically driven reaction oc-<br>curs. The age in aqueous media, which could provide real-time<br>reaction information. This technique helps to under-<br>stand how an electrochemically driven reaction oc-<br>curs. The *in-situloperando* XAS could serve as a<br>valuable techni reaction information. This technique helps to under-<br>stand how an electrochemically driven reaction oc-<br>curs. The *in-situloperando* XAS could serve as a<br>valuable technique for studying the electronic struc-<br>ture and loca stand how an electrochemically driven reaction oc-<br>curs. The *in-situloperando* XAS could serve as a<br>valuable technique for studying the electronic struc-<br>ture and local geometric structure of catalyst materials<br>under wor curs. The *in-situ/operando* XAS could serve as a<br>valuable technique for studying the electronic struc-<br>ture and local geometric structure of catalyst materials<br>under working conditions. The XAS technique in-<br>cludes two r valuable technique for studying the electronic struc-<br>ture and local geometric structure of catalyst materials<br>under working conditions. The XAS technique in-<br>cludes two regions: X-ray absorption near the edge<br>structure ( ture and local geometric structure of catalyst materials<br>under working conditions. The XAS technique in-<br>cludes two regions: X-ray absorption near the edge<br>structure (XANES) and extended X-ray absorption<br>fine structure (E under working conditions. The XAS technique in-<br>cludes two regions: X-ray absorption near the edge<br>structure (XANES) and extended X-ray absorption<br>fine structure (EXAFS). Combing with the *in-situl*<br>operando XAS technique cludes two regions: X-ray absorption near the edge<br>structure (XANES) and extended X-ray absorption<br>fine structure (EXAFS). Combing with the *in-situl*<br>operando XAS techniques, Friebel et al. found that the<br>Ni<sub>a/35</sub>Fe<sub>a25</sub> structure (XANES) and extended X-ray absorption<br>fine structure (EXAFS). Combing with the *in-situl*<br>operando XAS techniques, Friebel et al. found that the<br> $Ni_{0.25}Fe_{0.25}OOH$  catalyst has an  $\alpha$ -NiOOH structure at a high<br> fine structure (EXAFS). Combing with the *in-situl*<br>operando XAS techniques, Friebel et al. found that the<br>Ni<sub>0.75</sub>Fe<sub>0.25</sub>OOH catalyst has an  $\alpha$ -Ni(OH)<sub>2</sub> structure at<br>a low potential and a  $\gamma$ -NiOOH structure at a hi *operando* XAS techniques, Friebel et al. found that the  $Ni_{0.25}Fe_{0.25}OOH$  catalyst has an  $\alpha$ -Ni(OH)<sub>2</sub> structure at a low potential and a  $\gamma$ -NiOOH structure at a high potential. With the potential increasing, the sh

 $\#E\#(J. Electron) 2022, 28(3), 2108541 (10 of 31)$ <br>
synchrotron radiation can overcome the limitations of study of NiFe-based OER catalysts towards practical<br>
UHV conditions<sup>[56]</sup>. Ali-Leytty et al. conducted *in-situ* applications  $\frac{\text{d} \mathcal{H}^{\#}(J. \text{Electrochem.}) 2022, 28(3), 2108541 (10 of 31)}{\text{synchrotron radiation can overcome the limitations of study of Nife-based OER catalysts towards practical  
\nUHV conditions<sup>[56]</sup>. Ali-Lcyty et al. conducted *in-situ* applications.\nAPXPS experiments on Nife based catalysts under OER conditions. They found that when the electric potential is increased from 0 to 0.3 V (vs. Ag/AgCl),  
\nthe chic, Ni(OH), is oxidized to NiOOH, the O/OH ratio in \frac{d}{dx}$   $\frac{d}{dx}$   $\frac{d}{dx}$  **EVALUATE 1988**<br> **EVALUATE 1988**<br> **EVALUATE 1998**<br> **EVALUATE 1999**<br> **E** *I*E *Electrochem.*) 2022, 28(3), 2108541 (10 of 31)<br>
synchrotron radiation can overcome the limitations of study of NiFe-based OER catalysts towards practical<br>
UHV conditions<sup>[56]</sup>. Ali-Leytty et al. conducted *in-s* **EV**<sup>\ther</sup>(*J. Electrochem.*) 2022, 28(3), 2108541 (10 of 31)<br> **synchrotron radiation can overcome the limitations of** study of NiFe-based OER catalysts towards practical<br>
UHV conditions<sup>(sq</sup>. Ali-Leytty et al. conducte **EVALUATION**<br> **EVALUAT EXAMORET SET ASSES AND A CONSECT AND A PAPER CONSECT AND A PAPER CONSECT AND A PAPER CONSECT AND INTEGRALLY SURPORTION (I.E., Ni Example 19**<br> **Example 19 Synchrotron radiation can overcome the limitations of study of NiFe-based OER catalysts towards practical<br>
UHV conditions<sup>286</sup>. Ali-Leytty et al. conducted** *in-situ* **applications.<br>
APXPS experime**  $\frac{d\mathbb{E}/$ **EVALUAT Example 1988**<br> **EVALUAT EXAMORE SET THE UNITERED SET AND SYSTEM CONDUCT CONDUCT CONDUCT AND CONDUCT AND SYSTEM CONDUCT CONDUCT OF THE METAL CONDUCT OF THE METAL ORIGINAL ORDER CONDUCT OF THE DAMORE CONDUCT ON DEV Example 10. Electrochem.** (and the minitions of study of NiFe-based OER catalysts towards practical<br>UHV conditions<sup>591</sup>. Ali-Leytty et al. conducted *in-situ* applications.<br>
APNPS experiments on NiFe based catalysts u **THEP (***L. Electrochem.*) 2022, 28(3), 2108541 (10 of 31)<br> **Synchrotron radiation can overcome the limitations of** study of NiFe-based OFR catalysts towards practical<br>
CIIV conditions. They issued catalysts under **4 In EVALUATION THE SET INTO A SUCK A CONSECT AND SUCK A SUCK A SUCK A SUCK A SUCK AND SUCK CONDITIONS. A PAYS experiments on NiFe based catalysts**  $#i\ell\neq (L\ Ekerneken.) 2022, 28(3), 2108541 (10 of 31)$ <br>
synchrotron radiation can overcome the limitations of study of NiFe-based OER catalysts towards practical<br>
APYPS experiments on NiFe based catalysts under a **All Introduction** 4( $E\mathscr{F}(LElectroekom.)$  2022, 28(3), 2108541 (10 of 31)<br>synchrotron radiation can overcome the limitations of study of NiFe-based OER catalysts towards practical<br>UHV conditions<sup>564</sup>. Ali-Leytty et al. conducted *in-situ* app synchrotron radiation can overcome the limitations of<br>
UIIV conditions<sup>570</sup>. Ali-Leytty et al. conducted *in-situ*<br>
upplications.<br>
APXPS experiments on NiFe based catalysts under<br> **4 Introduction of <sup>57</sup>Fe Mössbauer**<br>
O synchrotron radiation can overcome the limitations of<br>
UHV conditions of DiFic-based OER catalysts towards practical<br>
UHV conditions.<sup>561</sup>, Mi-Leytty et al. conducted *in*-situations.<br>
OER conditions. They found that when UIIV conditions<sup>168</sup>. Ali-Leytty et al. conducted *in-sinu* applications of <sup>57</sup>**Fe Mössbauer**<br>
OER conditions. They found that when the electric **4 Introduction** of <sup>57</sup>**Fe Mössbauer**<br>
DOER conditions. They found t APXPS experiments on NiFe based catalysts under<br>
OER conditions. They found that when the electric<br>
oeconomic function is increased from 0 to 0.3 V (vs. Ag/AgCl),<br>
i.e., Ni(OH)<sub>2</sub> is oxidized to NiOOH, the O/OH ratio incr OFR conditions. They found that when the electric<br>
poetcroscopy<br>
potential is increased from 0 to 0.3 V ( $v_s$  Ag/AgCl),<br>
i.e., Ni(OH), is oxidized to NiOOH, the O/OH ratio in<br>
creases significantly. In addition, the Ni an potential is increased from 0 to 0.3 V (vs. Ag/AgCl).<br>
Les Coloritation to NOO Fraction in a standard technique for analyzing eatalysis processes<br>
tecnses significantly. In addition, the Ni and Fe 2p<br>
Erenses significantl ereases significantly. In addition, the Ni and Fe 2p<br>soon after Moskshaure effect was discovered<sup>199</sup>. This is<br>NPS spectra also show the oxidation of Ni and Fe<br>main golish methods of Ni and Fe<br>smallence of the main golish XPS spectra also show the oxidations of Ni and Fe<br>
unique for revealing the "black box" of catalysis be-<br>
durique for revealing the "black box" of catalysis be-<br>
durique for revealing the "black box" of catalysis be-<br>
spe during OER process<sup>[57]</sup>. The *in-situdopermalo* UV-vis cause of its *in-situdopermalo* application capabilities<sup>860</sup>.<br>
Secretorsocopy is used to track the metal oxidation pro-<br>
cess that cocurs in Ni(OH). Givin et al. us spectroscopy is used to track the metal oxidation pro-<br>
spectroscopy is used to track the metal oxidation pro-<br>
The main applications of Mössbauer spectroscopy in<br>
operarodo UV-vis spectroscopy to track the metal oxi-<br>
or cess that occurs in Ni(OH)<sub>2</sub>. Görlin et al. used *in-situl* catalysis research are: 1) identification of<br>operardo UV-vis spectroscopy to track the metal oxi-<br>or active phases for catalytic processes; 2<br>databy moreoves in *operando* UV-vis spectroscopy to track the metal oxi-<br>
or a crive phases for catalytic processes; 2) investiga-<br>
droin process in bilfe hased electrocatally.t. It was<br>
Simply performance; cat 3) sharedscrained of cat-<br>
N found that Fe inhibited the oxidation form Ni<sup>2</sup> to<br>
vertally tie performance; and 3) characterization of eat-<br>  $N_1^{12}$  during the OFR process. Combing the *m*-sin/<br>
or during the CR process combing the institution that NP<sup>-46</sup> during the OFR process. Combing the in-situ/ alysts during reaction and deactivation un-<br>operardo UV-vis spectroscopy, they concluded that<br>doe that also the situng prearmino conditions. There<br>of EMs carried out th operardo UV-vis spectroscopy, they concluded that<br>
OETR was carried variative and/or in-situ/operardo conditions. There<br>
OERR was carried out at the center of Ni<sup>2</sup><sup>2</sup> in the center of Ni<sup>2</sup><sup>2</sup> in the center of Ni<sup>2</sup><sup>2</sup> ( OFR was carried out at the center of Ni<sup>2+</sup> in the<br>
sure limited numbers of Massbauer-active clements<br>
Nife-baned catalys!". Moreover, Gotin et al. used in<br>  $m = \frac{1}{2}$ , the change distribution from the addition of the ca

applications. 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (10 of 31)<br>
ome the limitations of study of NiFe-based OER catalysts towards practical<br>
t al. conducted *in-situ* applications.<br>
based catalysts under **4 Introduction of <sup>57</sup>Fe** 

# 28(3), 2108541 (10 of 31)<br>study of NiFe-based OER catalysts towards practical<br>applications.<br>**4 Introduction of <sup>57</sup>Fe Mössbauer**<br>**Spectroscopy** Spectroscopy

i.e., Ni(OH), is oxidized to NiOOH, the O/OH ratio in-<br>
a standard technique for analyzing catalysis processes<br>
experimently. In addition, the Ni and Fe 2p<br>
orea after Mossbauer effect was discovered<sup>fly</sup>. This is<br>
2KPs s 28(3), 2108541 (10 of 31)<br>
study of NiFe-based OER catalysts towards practical<br>
applications.<br> **4 Introduction of <sup>57</sup>Fe Mössbauer<br>
Spectroscopy**<br>
Mössbauer spectroscopy has progressively become<br>
a standard technique for (3), 2108541 (10 of 31)<br>
Idy of NiFe-based OER catalysts towards practical<br>
plications.<br> **Introduction of <sup>57</sup>Fe Mössbauer**<br> **Spectroscopy**<br>
Mössbauer spectroscopy has progressively become<br>
tandard technique for analyzing 28(3), 2108541 (10 of 31)<br>
study of NiFe-based OER catalysts towards practical<br>
applications.<br> **4 Introduction of <sup>57</sup>Fe Mössbauer**<br> **Spectroscopy**<br>
Mössbauer spectroscopy has progressively become<br>
a standard technique fo 28(3), 2108541 (10 of 31)<br>study of NiFe-based OER catalysts towards practical<br>applications.<br>**4 Introduction of <sup>57</sup>Fe Mössbauer**<br>**Spectroscopy**<br>Mössbauer spectroscopy has progressively become<br>a standard technique for anal 28(3), 2108541 (10 of 31)<br>
study of NiFe-based OER catalysts towards practical<br>
applications.<br> **4 Introduction of <sup>57</sup>Fe Mössbauer**<br> **Spectroscopy**<br>
Mössbauer spectroscopy has progressively become<br>
a standard technique fo 28(3), 2108541 (10 of 31)<br>study of NiFe-based OER catalysts towards practical<br>applications.<br>**4 Introduction of <sup>57</sup>Fe Mössbauer**<br>**Spectroscopy**<br>Mössbauer spectroscopy has progressively become<br>a standard technique for anal 28(3), 2108541 (10 of 31)<br>
study of NiFe-based OER catalysts towards practical<br>
applications.<br> **4 Introduction of <sup>57</sup>Fe Mössbauer<br>
Spectroscopy**<br>
Mössbauer spectroscopy has progressively become<br>
a standard technique for 28(3), 2108541 (10 of 31)<br>study of NiFe-based OER catalysts towards practical<br>applications.<br>**4** Introduction of <sup>57</sup>Fe Mössbauer<br>Spectroscopy<br>Mössbauer spectroscopy has progressively become<br>a standard technique for analyz 28(3), 2108541 (10 of 31)<br>
study of NiFe-based OER catalysts towards practical<br>
applications.<br> **4 Introduction of <sup>57</sup>Fe Mössbauer<br>
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Mössbauer spectroscopy has progressively become<br>
a standard technique for 28(3), 2108541 (10 of 31)<br>
study of NiFe-based OER catalysts towards practical<br>
applications.<br> **4 Introduction of <sup>57</sup>Fe Mössbauer<br>
Spectroscopy**<br>
Mössbauer spectroscopy has progressively become<br>
a standard technique for 28(3), 2108541 (10 of 31)<br>
study of NiFe-based OER catalysts towards practical<br>
applications.<br> **4 Introduction of <sup>57</sup>Fe Mössbauer**<br> **Spectroscopy**<br>
Mössbauer spectroscopy has progressively become<br>
a standard technique fo 28(3), 2108541 (10 of 31)<br>
study of NiFe-based OER catalysts towards practical<br>
applications.<br> **4 Introduction of <sup>57</sup>Fe Mössbauer**<br> **Spectroscopy**<br>
Mössbauer spectroscopy has progressively become<br>
a standard technique fo study of NiFe-based OER catalysts towards practical<br>applications.<br>**4 Introduction of <sup>57</sup>Fe Mössbauer<br>Spectroscopy**<br>Mössbauer spectroscopy has progressively become<br>a standard technique for analyzing catalysis processes<br>so study of NiFe-based OER catalysts towards practical<br>applications.<br>**4 Introduction of** <sup>57</sup>**Fe Mössbauer**<br>**Spectroscopy**<br>Mössbauer spectroscopy has progressively become<br>a standard technique for analyzing catalysis processe applications.<br> **4 Introduction of <sup>57</sup>Fe Mössbauer<br>
Spectroscopy**<br>
Mössbauer spectroscopy has progressively become<br>
a standard technique for analyzing catalysis processes<br>
soon after Mössbauer effect was discovered<sup>[59]</sup>. **4 Introduction of <sup>57</sup>Fe Mössbauer<br>
Spectroscopy**<br>
Mössbauer spectroscopy has progressively become<br>
a standard technique for analyzing catalysis processes<br>
soon after Mössbauer effect was discovered<sup>1591</sup>. This is<br>
uniqu **Spectroscopy**<br>Mössbauer spectroscopy has progressively become<br>a standard technique for analyzing catalysis processes<br>soon after Mössbauer effect was discovered<sup>[59]</sup>. This is<br>unique for revealing the "black box" of catal Mössbauer spectroscopy has progressively become<br>a standard technique for analyzing catalysis processes<br>soon after Mössbauer effect was discovered<sup>[59]</sup>. This is<br>unique for revealing the "black box" of catalysis be-<br>cause a standard technique for analyzing catalysis processes<br>soon after Mössbauer effect was discovered<sup>[39]</sup>. This is<br>unique for revealing the "black box" of catalysis be-<br>cause of its *in-situloperando* application capabiliti soon after Mössbauer effect was discovered<sup>[59]</sup>. This is<br>unique for revealing the "black box" of catalysis be-<br>cause of its *in-situ/operando* application capabilities<sup>[69]</sup>.<br>The main applications of Mössbauer spectrosco unique for revealing the "black box" of catalysis be-<br>cause of its *in-situ/operando* application capabilities<sup>[60]</sup>.<br>The main applications of Mössbauer spectroscopy in<br>catalysis research are: 1) identification of active cause of its *in-situ/operando* application capabilities<sup>[60]</sup>.<br>The main applications of Mössbauer spectroscopy in<br>catalysis research are: 1) identification of active sites<br>or active phases for catalytic processes; 2) inv The main applications of Mössbauer spectroscopy in<br>catalysis research are: 1) identification of active sites<br>or active phases for catalytic processes; 2) investiga-<br>tion of correlations between catalyst structure and<br>cata talysis research are: 1) identification of active sites<br>active phases for catalytic processes; 2) investiga-<br>n of correlations between catalyst structure and<br>alytic performance; and 3) characterization of cat-<br>zsts during or active phases for catalytic processes; 2) investiga-<br>tion of correlations between catalyst structure and<br>catalytic performance; and 3) characterization of cat-<br>alysts during reaction activation and deactivation un-<br>der tion of correlations between catalyst structure and<br>catalytic performance; and 3) characterization of cat-<br>alysts during reaction activation and deactivation un-<br>der *ex-situ* and/or *in-situloperando* conditions. There<br>a catalytic performance; and 3) characterization of cat-<br>alysts during reaction activation and deactivation un-<br>der *ex-situ* and/or *in-situ/operando* conditions. There<br>are limited numbers of Mössbauer-active elements<br>such alysts during reaction activation and deactivation un-<br>der *ex-situ* and/or *in-situ/operando* conditions. There<br>are limited numbers of Mössbauer-active elements<br>such as iron (<sup>57</sup>Fe), tin (<sup>195</sup>Sn), antimony (<sup>121</sup>Sb), g der *ex-situ* and/or *in-situ/operando* conditions. There<br>are limited numbers of Mössbauer-active elements<br>such as iron (<sup>57</sup>Fe), tin (<sup>195</sup>Sn), antimony (<sup>21</sup>Sb), gold<br>(<sup>97</sup>Au), nickel (<sup>6</sup>Ni), ruthenium (<sup>29</sup>Nu), iridi

Nife-based eatalyst<sup>(19)</sup>. Moreover, Girlin et al. used *in*-<br>such as iron (<sup>19</sup>Es), tin (<sup>19</sup>Sn), antimony (<sup>19</sup>Sh), gold<br>tirulation erand the DNMS to track the Franday charge dis-<br>elition from the dathition of the cenda *situloper ando* DEMS to track the Faraday charge dis-<br> *tivistion from* the addition of the catalyst to the prod-<br>
tribution from the addition of the catalyst to the prod-<br>
traction from the addition of the catalyst is t tribution from the addition of the catalyst to the prod-<br>
und <sup>183</sup>H<sub>/</sub>), and neptunium (<sup>39</sup>Np) etc., which can be<br>
ter formation and the redox process of the catalyst.<br>
The lower metal redox chance indicates that the av uct formation and the redox process of the eatalyst.<br>
Similal The lower metal redox charge indicates that the aver-<br>
The lower metal redox charge indicates that the aver-<br>
sign value of Ni in the NiFe-based catalyst is lo The lower metal redox charge indicates that the aver-<br>
tion is due to several criteria such as suitable lifetime<br>
of the interNic-based catalyst is lower of fuld in the Nic-based catalysts due to the metal state, transico than that of nickel oxide catalysts. They concluded<br>
inter Fe inhibits the woodchor of Ni during OER<br>
and the Fe inhibits the oxident of Ni during OER<br>
and seems to have a stabilizing effect on low valent<br>
Numerable and s that the Fe inhibits the oxidation of Ni-during OER<br>
ments, <sup>*n*</sup>Fe is the most studied and well-known Misss-<br>
and secons to have a stabilizing effect on low valent<br>
Ni, thereby promoting OFR<sup>191</sup>.<br>
Among several other *i* and scems to have a stabilizing effect on low valent<br>
Ni, thereby promoting OER<sup>ID</sup>, Microby promoting CRE<sup>ID</sup>, Microby and the mis-<br>
Ni, thereby promoting CRE<sup>ID</sup>, a and the integration of the system radio<br>
<sup>23</sup>The Mössba Ni, thereby promoting OER<sup>1*m*</sup>,<br>
Missbauer spectroscopy, which involves the emis-<br>
Among soveral other *in-stutopermado* techniques,<br>
"The Missbauer spectroscopy is a strong method with<br>
tion of y-rays from radioactive n are limited numbers of Mössbauer-active elements<br>such as iron (<sup>57</sup>Fe), tin (<sup>195</sup>Sn), antimony (<sup>121</sup>Sb), gold<br>(<sup>497</sup>Au), nickel (<sup>6</sup>Ni), ruthenium (<sup>29</sup>Nu), iridium (<sup>93</sup>Ir<br>and <sup>193</sup>Ir), and neptunium (<sup>297</sup>Np) etc., wh such as iron (<sup>37</sup>Fe), tin (<sup>19</sup>Sn), antimony (<sup>21</sup>Sb), gold<br><sup>(397</sup>Au), nickel (<sup>69</sup>Ni), ruthenium (<sup>39</sup>Nu), iridium (<sup>39</sup>Ir<br>and <sup>193</sup>Ir), and neptunium (<sup>297</sup>Np) etc., which can be<br>studied using Mössbauer spectroscopy. T (<sup>397</sup>Au), nickel (<sup>69</sup>Ni), ruthenium (<sup>99</sup>Ru), iridium (<sup>93</sup>Ir<br>and <sup>193</sup>Ir), and neptunium (<sup>237</sup>Np) etc., which can be<br>studied using Mössbauer spectroscopy. This limita-<br>tion is due to several criteria such as suitable and <sup>193</sup>Ir), and neptunium (<sup>237</sup>Np) etc., which can be studied using Mössbauer spectroscopy. This limitation is due to several criteria such as suitable lifetime of nuclear excited state, transition energy, easy accessi studied using Mössbauer spectroscopy. This limita-<br>tion is due to several criteria such as suitable lifetime<br>of nuclear excited state, transition energy, easy acces-<br>sibility, and handling. Among these different ele-<br>ment standed analysis *machinality* in the method of  $\frac{1}{2}$  is the method of inclusive furthermore, is the most studied infering of nuclear excited state, transition energy, easy accessibility, and handling. Among these dif of nuclear excited state, transition energy, easy accessibility, and handling. Among these different elements, <sup>57</sup>Fe is the most studied and well-known Mössbauer nuclide.<br>
Mössbauer spectroscopy, which involves the emiss sibility, and handling. Among these different elements, <sup>57</sup>Fe is the most studied and well-known Mössbauer nuclide.<br>
Mössbauer spectroscopy, which involves the emission of  $\gamma$ -rays from radioactive nuclei of the same el bauer nuclide.<br>
Mössbauer spectroscopy, which involves the emission of  $\gamma$ -rays from radioactive nuclei and the absorption of these  $\gamma$ -rays by other nuclei of the same element, is a powerful technique for studying mate Mössbauer spectroscopy, which involves the emis-<br>sion of  $\gamma$ -rays from radioactive nuclei and the absorp-<br>tion of these  $\gamma$ -rays by other nuclei of the same ele-<br>ment, is a powerful technique for studying materials<br>by m

The Mossbauer effect is the absorption and emison of the first excited state of <sup>37</sup>Co has the mean life-<br>
Independent (3) and receiver the absorption and emission of  $\gamma$ -rays from a recoilless nuclear resonance<br>
The Mos **EVALUATION 1999**<br> **EVALUATION**<br> **EVALUATION**<br> **EVALUATION**<br> **EVALUATION**<br> **EVALUATION**<br> **EVALUATIONS FOR PROCESS.**<br> **EVALUATION**<br> **EVALUATION**<br> **EVALUATION**<br> **EVALUATION**<br> **EVALUATION**<br> **EVALUATION**<br> **EVALUATION**<br> **EVALU EVALUATION**<br> **EXAMORE THENOM THENOM CONDUCE 2022, 28(3), 2108541 (11 of 31)**<br> **ETHENOM CONTAMORE CONTAMORE CORPORATION**<br> **EXAMORE THENOM CONTAMORE CORPORATION**<br> **4.1 Principle of Mössbauer Spectroscopy**<br> **4.1 Principle o** the same for the same for the same for the same for the same frequency of the same frequency of the same frequency for the same frequency from the same frequency of Mössbauer effect some and the Napply in The Missbauer ef **Example 19**<br> **set of the sender of the Shander Spectroscopy<br>
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link between OER behavior and Fc structural charac-<br>
rays, named as Müssbauer effect, soon after he was<br>
teristics in Fe-based catalysts during the OER proce **EXAMPLE 12**<br> **EXAMPLE 12** (*Electrochem.*) 2022, 28(3), 2108541 (11 of 31)<br>
link between OER behavior and Fe structural charac-<br>
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link between OER behavior and Fe structural charac-rays, named as Mossbauer effect, soon after he was<br>
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teristics in Fe-based catalysts during the OER process. \quad \text{awarded the Nobel prize in physics 1961, may be  
the Missbauer effect is the absorption and emission of  $\gamma$ -rays from a recoless nuclear resonance. \quad \text{Figure 7(A) descriptor in physics 1961, known as the Lan-  
sinon of  $\gamma$ -rays from a recoless nuclear resonance of  $\gamma$ -flow. The Missbauer fraction<sup>6D</sup>. \quad \text{Step 2(A) descriptor in$ the  $\frac{1}{2}(L)$  Electrobera, 2022, 28(3), 2108541 (11 of 31)<br>
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mays, named as Mössbauer effect, soon after he was<br>
teristics in Fe-based catalysts during the OFR process.<br> **4.1 Principle of Mössbauer Spectroscopy**<br> **4.1 Principle o** link between OER behavior and Fe structural charac-<br>
teristies in Fe-based catalyst during the OER process.<br> **Altherity:** Findery definite absorption and emiss-<br> **4.1 Principle of Müssbauer Spectroscopy**<br> **4.41 Principle** teristics in Fe-based catalysts during the OFR process<br> **All Principle of Nobsorption** (abso<mark>sbouter Spectroscopy)</mark><br>
teted only for this proportion, known as the Lamb-<br> **Principle of Mössbauer Spectroscopy**<br>
information a **4.1 Principle of Mössbauer Spectroscopy** tected only for this propotion, known as the Lamb-<br>The Massbauer effect is the absorption and emiss-<br>**Masshauer freedtor in the control** and emission of y-rays from a receilles nu The Mossbauer effect is the absorption and emiss-<br>
Mossbauer fraction<sup>901</sup>.<br>
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phenomenon, analogous to the acoustic resonance of the par phenomenon, analogous to the acoustic resonance of<br>
phenomena in which the mean lifetime of the excited<br>
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the state of the parameter of state of the parameter two tuning forks with the same frequency  $f = f$ , for<br>scate of the parent nuclei is very important. For exam-<br>scale (s) and recoire (s). A nucleus with cneegy  $E_x$  be, the first excited state of <sup>5</sup>Co has the mean life-<br>sca sender (a) and receiver (r). A nucleus with energy  $E_s$  ple, the first excited state of <sup>3</sup>Co has the mean life-<br>at an excited state (with Z proton and N nucleum) un-<br>time of around 100 ns which is normally being used<br>dee at an excited state (with Z proton and N neutron) un-<br>
dengens transitions to the ground state with energy  $R_c$ . for <sup>3</sup>Fe Missbaure experiment. As a result of the ra-<br>
depresence instrictions to the ground state, with en dergoes transitions to the ground state with energy  $E_x$  for <sup>37F</sup>e Mössbauer experiment. As a result of the ra-<br>by producing  $\gamma$ -rays are crossing  $E_x$ . The  $\gamma$ -rasy may be dioactive decay, the excited state of Co deca by producing  $\gamma$ -rays of energy  $E_{\nu}$ . The  $\gamma$ -rays may be<br>
absorbed by another nucleus of the same type (dicani-<br>  $\gamma$ <sup>-rec</sup>e excited state by electron capture<br>
cal  $Z$  and  $N$ ) in its ground state, resulting in a tr absorbed by another nucleus of the same type (identi-<sup>3</sup><sup>2</sup><sup>3</sup>Fe excited state by electron capture process. The ex-<br>coil 2 and A) in is ground state, resulting in a transi-<br>include is throssbeaner nucleus decoses and come cal Z and N) in its ground state, resulting in a transi-<br>
circl state of Mossbauer nucleus decays and comes to<br>
troin to the excited state of energy  $E_r$ . This phesime ground state. The decay of <sup>6</sup>Co generates two excite tion to the excited state of energy  $E_v$ . This phecaeure absorption is called  $\gamma$ -rays recoilless resonance absorption as solenation in Figure 7(A). Reserved in solenation and by the solenation as operator of the mission enon is called  $\gamma$ -rays recoilless resonance absorbates of <sup>57</sup>Fe where almost 99% decay occurs for the<br>one as schematically shown in Figure 7(A). Res. lowest energy level which is 14.4 keV and is known<br>one ashorption ca sorption as schematically shown in Figure 7(A). Res-<br>
consume absorption can only be secn if the cmission as the first excited state from ground state f<sup>7</sup>Fc.<br>
and absorption lines overlap enough. When  $\gamma$ -rays The ground

 $\# \&\#$  (*I. Electrochem.*) 2022, 28(3), 2108541 (11 of 31)<br>
link between OER behavior and Fe structural charac-<br>
rays, named as Mössbauer effect, soon after he was<br>
awarded the Nobel prize in physics 1961, may be de-<br> the  $\# \# \langle I. \text{Electrochem.} \rangle$  2022, 28(3), 2108541 (11 of 31)<br>
link between OER behavior and Fe structural charac-<br>
teristics in Fe-based catalysts during the OER process.<br> **4.1 Principle of Mössbauer Spectroscopy**<br>
The Mössba 4.1 Principle of M觟ssbauer Spectroscopy 28(3), 2108541 (11 of 31)<br>rays, named as Mössbauer effect, soon after he was<br>awarded the Nobel prize in physics 1961, may be de-<br>tected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>[63]</sup>.<br>Figure 7(A) des 28(3), 2108541 (11 of 31)<br>
rays, named as Mössbauer effect, soon after he was<br>
awarded the Nobel prize in physics 1961, may be de-<br>
tected only for this proportion, known as the Lamb-<br>
Mössbauer fraction<sup>[63]</sup>.<br>
Figure 7( 28(3), 2108541 (11 of 31)<br>rays, named as Mössbauer effect, soon after he was<br>awarded the Nobel prize in physics 1961, may be de-<br>tected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>[63]</sup>.<br>Figure 7(A) de 28(3), 2108541 (11 of 31)<br>
rays, named as Mössbauer effect, soon after he was<br>
awarded the Nobel prize in physics 1961, may be de-<br>
tected only for this proportion, known as the Lamb-<br>
Mössbauer fraction<sup>[63]</sup>.<br>
Figure 7( 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (11 of 31)<br>d Fe structural charac-<br>uring the OER process. awarded the Nobel prize in physics 1961, may be de-<br>**termscopy** tected only for this proportion, known as the Lamb-<br>cha

*Electrochem.*) 2022, 28(3), 2108541 (11 of 31)<br>
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ted only for this proportion, known as the Lamb-<br>
issbauer fraction<sup>[63]</sup>.<br>
Figure 7(A) desc 28(3), 2108541 (11 of 31)<br>rays, named as Mössbauer effect, soon after he was<br>awarded the Nobel prize in physics 1961, may be de-<br>tected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>[63]</sup>.<br>Figure 7(A) de 28(3), 2108541 (11 of 31)<br>rays, named as Mössbauer effect, soon after he was<br>awarded the Nobel prize in physics 1961, may be de-<br>tected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>[63]</sup>.<br>Figure 7(A) de 28(3), 2108541 (11 of 31)<br>
rays, named as Mössbauer effect, soon after he was<br>
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tected only for this proportion, known as the Lamb-<br>
Mössbauer fraction<sup>[63]</sup>.<br>
Figure 7( 28(3), 2108541 (11 of 31)<br>
rays, named as Mössbauer effect, soon after he was<br>
awarded the Nobel prize in physics 1961, may be de-<br>
tected only for this proportion, known as the Lamb-<br>
Mössbauer fraction<sup>[63]</sup>.<br>
Figure 7( 28(3), 2108541 (11 of 31)<br>rays, named as Mössbauer effect, soon after he was<br>awarded the Nobel prize in physics 1961, may be de-<br>tected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>(69)</sup>.<br>Figure 7(A) de 28(3), 2108541 (11 of 31)<br>rays, named as Mössbauer effect, soon after he was<br>awarded the Nobel prize in physics 1961, may be de-<br>tected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>[63]</sup>.<br>Figure 7(A) de 28(3), 2108541 (11 of 31)<br>rays, named as Mössbauer effect, soon after he was<br>awarded the Nobel prize in physics 1961, may be de-<br>tected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>[6]</sup>.<br>Figure 7(A) des 28(3), 2108541 (11 of 31)<br>rays, named as Mössbauer effect, soon after he was<br>awarded the Nobel prize in physics 1961, may be de-<br>tected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>[63]</sup>.<br>Figure 7(A) de 28(3), 2108541 (11 of 31)<br>
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tected only for this proportion, known as the Lamb-<br>
Mössbauer fraction<sup>[63]</sup>.<br>
Figure 7( 28(3), 2108541 (11 of 31)<br>
rays, named as Mössbauer effect, soon after he was<br>
awarded the Nobel prize in physics 1961, may be de-<br>
tected only for this proportion, known as the Lamb-<br>
Mössbauer fraction<sup>[63]</sup>.<br>
Figure 7( Example and Solution of the two states and as Mössbauer effect, soon after he was awarded the Nobel prize in physics 1961, may be detected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>[63]</sup>. Figure 7(A) rays, named as Mössbauer effect, soon after he was<br>awarded the Nobel prize in physics 1961, may be de-<br>tected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>[63]</sup>.<br>Figure 7(A) describes the overall Mössba awarded the Nobel prize in physics 1961, may be de-<br>tected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>[63]</sup>.<br>Figure 7(A) describes the overall Mössbauer effect<br>phenomena in which the mean lifetime of tected only for this proportion, known as the Lamb-<br>Mössbauer fraction<sup>[63]</sup>.<br>Figure 7(A) describes the overall Mössbauer effect<br>phenomena in which the mean lifetime of the excited<br>state of the parent nuclei is very impor Mössbauer fraction<sup>[63]</sup>.<br>
Figure 7(A) describes the overall Mössbauer effect<br>
phenomena in which the mean lifetime of the excited<br>
state of the parent nuclei is very important. For exam-<br>
ple, the first excited state of Figure 7(A) describes the overall Mössbauer effect<br>phenomena in which the mean lifetime of the excited<br>state of the parent nuclei is very important. For exam-<br>ple, the first excited state of  ${}^{57}$ Co has the mean life-<br>t phenomena in which the mean lifetime of the excited<br>state of the parent nuclei is very important. For exam-<br>ple, the first excited state of  ${}^5C$  has the mean life-<br>time of around 100 ns which is normally being used<br>for te of the parent nuclei is very important. For exam-<br>e, the first excited state of  $^{57}Co$  has the mean life-<br>ne of around 100 ns which is normally being used<br> $^{57}Fe$  Mössbauer experiment. As a result of the ra-<br>active de ple, the first excited state of <sup>57</sup>Co has the mean life-<br>time of around 100 ns which is normally being used<br>for <sup>57</sup>Fe Mössbauer experiment. As a result of the ra-<br>dioactive decays, the excited state of <sup>57</sup>Co decays to<br> time of around 100 ns which is normally being used<br>for <sup>57</sup>Fe Mössbauer experiment. As a result of the ra-<br>dioactive decays, the excited state of <sup>57</sup>Co decays to<br><sup>57</sup>Fe excited state by electron capture process. The ex-<br> for <sup>57</sup>Fe Mössbauer experiment. As a result of the radioactive decays, the excited state of <sup>57</sup>Co decays to <sup>57</sup>Fe excited state by electron capture process. The excited state of Mössbauer nucleus decays and comes to gr dioactive decays, the excited state of <sup>57</sup>Co decays to<br><sup>57</sup>Fe excited state by electron capture process. The ex-<br>cited state of Mössbauer nucleus decays and comes to<br>ground state. The decay of <sup>57</sup>Co generates two excite <sup>57</sup>Fe excited state by electron capture process. The ex-<br>cited state of Mössbauer nucleus decays and comes to<br>ground state. The decay of <sup>57</sup>Co generates two excited<br>states of <sup>57</sup>Fe where almost 99% decay occurs for the

onance absorption can only be seen if the emission<br>and as the first excited state from ground state of  ${}^{\circ}$ Fe.<br>and absorption lines overlap conough. When  $\gamma$ -rays The ground state of  ${}^{\circ}$ Fe does not undergo any kin and absorption lines overlap enough. When  $\gamma$ -rays The ground state of "Fe does not undergo any kind of with energy  $E_n$  are enrited or absorbed in freely mov-<br>
decay or sing as on (deal of a state to a state of a state with energy  $E_r$  are emitted or absorbed in freely mov-<br>
ing atom (molecule) of mass m, it undergoes a recoil<br>
are effect with energy keyl. The subspaces are coil<br>
are first with energy keyl. The lattice has the method of ing atom (molecule) of mass m, it undergoes a recoil<br>energy level. Each nuclear level possesses specifie<br>effect with energy  $E_N$  which is many orders of magni-<br>energy  $E_N$  many orders of magni-<br>resonance is not possible in effect with energy  $E_8$  which is many orders of magni-<br>
energy, spin, parity, and docay constant  $k$  mean prob-<br>
tude groater than the natural line-width (1), hence ability of decay per unit time.<br>
resonance is not possi nude greater than the natural line-width (*I*), hence<br>
resonance is not possible in a freely moving atom or As shown in Figure 7(B), due to the electrical mo-<br>
resonance is not possible in a freely moving atom or  $\sim$  As s resonance is not possible in a freely moving atom or<br>
moscille (gas, liquid). As a result, the Mossbaure reff-<br>
monic of the calculation are decarted quadrupole intreac-<br>
ect cannot be seen in the presence of freely moving molecule (gas, liquid). As a result, the Mössbauer eff-<br>conde interaction, the electrical quadrupole interaction<br>atoms or molecules, such as those in a graseous or fuci-<br>energy levels of <sup>3</sup>Fe (ground and first excited sta ect cannot be seen in the presence of freely moving<br>
ion, and the nuclear Zeeman interaction, the nuclear<br>
atoms or molecules, such as those in a gaseous or liq-<br>
emergy levels of <sup>5</sup>Fe (ground and its excited states<br>
uid atoms or molecules, such as those in a gaseous or liq-<br>
urergy levels of <sup>7</sup>Fe (ground and first excited states<br>
urd form. Recoolling the solid state, and basically<br>
be split by electric or magnetic fields acting at the nu uid form. Recoilless emission and absorption of with nuclear spins of 1/2 and 3/2, respectively) can  $\gamma$ -rays are conceivable in the solid state, and basically be split by elective or magnetic fields acing at the nul-<br>uncited state of Mössbauer nucleus decays and comes to<br>ground state. The decay of <sup>57</sup>Co generates two excited<br>states of <sup>57</sup>Fe where almost 99% decay occurs for the<br>lowest energy level which is 14.4 keV and is known<br>as the ground state. The decay of <sup>57</sup>Co generates two excited<br>states of <sup>57</sup>Fe where almost 99% decay occurs for the<br>lowest energy level which is 14.4 keV and is known<br>as the first excited state from ground state of <sup>5</sup>Fe.<br>The states of <sup>37</sup>Fe where almost 99% decay occurs for the lowest energy level which is 14.4 keV and is known as the first excited state from ground state of  $^{5}$ Fe. The ground state of  $^{5}$ Fe does not undergo any kind of d lowest energy level which is 14.4 keV and is known<br>as the first excited state from ground state of  $^{57}Fe$ .<br>The ground state of  $^{57}Fe$  does not undergo any kind of<br>decay and the decay constant is zero for it due to zero<br> as the first excited state from ground state of <sup>5</sup>Fe.<br>The ground state of <sup>5</sup>Fe does not undergo any kind of<br>decay and the decay constant is zero for it due to zero<br>energy level. Each nuclear level possesses specific<br>ene The ground state of <sup>*s*T</sup>Fe does not undergo any kind of decay and the decay constant is zero for it due to zero energy level. Each nuclear level possesses specific energy, spin, parity, and decay constant *k* mean proba decay and the decay constant is zero for it due to zero<br>energy level. Each nuclear level possesses specific<br>energy, spin, parity, and decay constant  $k$  mean prob-<br>ability of decay per unit time.<br>As shown in Figure 7(B), energy level. Each nuclear level possesses specific<br>energy, spin, parity, and decay constant  $k$  mean prob-<br>ability of decay per unit time.<br>As shown in Figure 7(B), due to the electrical mo-<br>nopole interaction, the electr energy, spin, parity, and decay constant  $k$  mean probability of decay per unit time.<br>
As shown in Figure 7(B), due to the electrical mo-<br>
nopole interaction, the electrical quadrupole interac-<br>
tion, and the nuclear Zeem ability of decay per unit time.<br>As shown in Figure 7(B), due to the electrical mo-<br>nopole interaction, the electrical quadrupole interac-<br>tion, and the nuclear Zeeman interaction, the nuclear<br>energy levels of  $^{57}Fe$  (gro As shown in Figure 7(B), due to the electrical mo-<br>nopole interaction, the electrical quadrupole interac-<br>tion, and the nuclear Zeeman interaction, the nuclear<br>energy levels of  $^{57}Fe$  (ground and first excited states<br>wit nopole interaction, the electrical quadrupole interaction, and the nuclear Zeeman interaction, the nuclear energy levels of <sup>57</sup>Fe (ground and first excited states with nuclear spins of 1/2 and 3/2, respectively) can be sp tion, and the nuclear Zeeman interaction, the nuclear energy levels of  ${}^{57}Fe$  (ground and first excited states with nuclear spins of  $1/2$  and  $3/2$ , respectively) can be split by electric or magnetic fields acting at t energy levels of <sup>57</sup>Fe (ground and first excited states<br>with nuclear spins of 1/2 and 3/2, respectively) can<br>be split by electric or magnetic fields acting at the nu-<br>cleus. The hyperfine interactions are able to cause a with nuclear spins of 1/2 and 3/2, respectively) can<br>be split by electric or magnetic fields acting at the nu-<br>cleus. The hyperfine interactions are able to cause a<br>positive or negative shift of the peak position from<br>zer

 $#E \neq (I. Electrochem.)$  2022, 28(3), 2108541 (12 of 31)<br>Usually there are three main parameters obtained spectrum, analyzing it by considering the various hy-<br>from Mössbauer spectroscopy in data form by curve perfine interactions **fitting**<br>**fitting measured Musically there are three main parameters obtained spectrum, analyzing it by considering the various hy-<br>from Mössbauer spectroscopy in data form by curve perfine interactions existed in the nu**  $\frac{f(k\#(J. Electron, 2022, 28(3), 2108541 (12 of 31))}{(12.6 \text{ rad})^2}$ <br>Usually there are three main parameters obtained spectrum, analyzing it by considering the various hy-<br>from Mössbauer spectroscopy in data form by curve perfine int **Figure 1.1**<br> **Example 1.1**<br> **Example 1.1**<br> **Example 1.2**<br> **Example 1.2**  $\pm \frac{\text{rk}\mathcal{L}_F^2(J. Electrochem.) 2022, 28(3), 2108541 (12 of 31)}{\text{Usually there are three main parameters obtained spectrum, analyzing it by considering the various hy-  
from Missbauer spectroscopy in data form by curve-  
formalism measured Missbauer spectra, which gives in-  
function about electrotnic, structural and magnetic  
proporties of investigated material include: 1) Isomer  
properties of investigated material include: 1) Isomer  
material, structural properties and chemical bonding  
shift (δ) arises from Coulombic interaction between  
trung (Δ) arises from the interaction of electric field  
and standard Missbauer **Instrumentation**  
and  
and the electric quadrilateral in the electric field  
of electric field  
and Miss$  $\pm$  *Rectrochem.*) 2022, 28(3), 2108541 (12 of 31)<br>Usually there are three main parameters obtained spectrum, analyzing it by considering the various hy-<br>from Mössbauer spectroscopy in data form by curve perfine interact the  $\mathcal{H}_2$  arises from the interaction of electric material defined the surfactor of electric material and the interactions considering the various hy-<br>from Mössbauer spectroscopy in data form by eurve perfine interact  $\Phi(E^{\omega}(L \cdot Electrockem, 2022, 28(3), 2108541 (12 of 31))$ <br>Usually there are three main parameters obtained spectrum, analyzing it by considering the various hy-<br>from Mössbauer spectroscopy in data form by eurve perfine interactions  $\mathcal{L}(2, \mathcal{L})$ <br>
Usually there are three main parameters obtained spectrum, analyzing it by considering the various hy-<br>
from Mössbauer spectroscopy in data form by curve perfine interactions existed in the nuclear locat **Example 12**<br> **Examply there are three main parameters obtained** spectrum, analyzing it by considering the various hy-<br>
from Missshauer spectroscopy in data form by curve perfine interactions existed in the nuclear locati **HEFT**(*L Electrochem*, 2022, 28(3), 2108541 (12 of 31)<br>
Usually there are three main parameters obtained spectrum, analyzing it by considering the various hy-<br>
Usually there are three main parameters obtained spectrum, a **1988**<br> **ugged the observation**  $\Phi(E \neq t)$ . *Electrochem.*) 2022, 28(3), 2108541 (12 of 31)<br> **Usually there are three main parameters obtained** spectrum, analyzing it by considering the various hy-<br>
from Müssbauer spectro **Fig. 16** E4 (*Electrochem.*) 2022, 28(3), 2108541 (12 of 31)<br>
Usually there are three main parameters obtained spectrum, analyzing it by considering the various hy-<br>
from Mössbauer spectroscopy in data form by curve perf the  $\frac{16}{2}\pi(LEctracken, 2022, 28(3), 2108541 (12 of 31))$ <br>Usually there are three main parameters obtained spectrum, analyzing it by considering the various hy-<br>from Mössbauer spectroscopy in data form by curve perfine interact

 $\text{LCE}(L\text{ }Electrochem.)$  2022, 28(3), 2108541 (12 of 31)<br>Usually there are three main parameters obtained spectrum, analyzing it by considering the various hy-<br>m Mössbauer spectroscopy in data form by curve perfine interactions 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pro  $28(3)$ ,  $2108541$  (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physica 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr (3), 2108541 (12 of 31)<br>
actrum, analyzing it by considering the various hy-<br>
fine interactions existed in the nuclear location,<br>
can study the configuration and distribution of or-<br>
al electrons, the physical properties 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (12 of 31)<br>
a parameters obtained spectrum, analyzing it by considering the various hy-<br>
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ectra, whic

28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr 28(3), 2108541 (12 of 31)<br>spectrum, analyzing it by considering the various hy-<br>perfine interactions existed in the nuclear location,<br>we can study the configuration and distribution of or-<br>bital electrons, the physical pr



 $\#E\#(J. \text{Electrochem.})$  2022, 28(3), 2108541 (13 of 31)<br>temperature during measurement, which make this sweep method in which a moving driver or velocity<br>instrument suitable to operate at a wide range of tem-<br>perature conditio  $\exists \ell \neq (I. Electron, 2022, 28(3), 2108541 (13 of 31)$ <br>temperature during measurement, which make this sweep method in which a moving driver or velocity<br>instrument suitable to operate at a wide range of tem-<br>perature conditions. Sche  $\exists x \in \mathbb{R}$   $\exists x \in \mathbb{R}$  temperature during measurement, which make this sweep method in which a moving driver or velocity instrument suitable to operate at a wid **figure 10**<br>  $\frac{d}{dt}$   $\pm (k\mathcal{F}(J. Electron) 2022, 28(3), 2108541 (13 of 31)$ <br>
temperature during measurement, which make this sweep method in which a moving driver or velocity<br>
instrument suitable to operate at a wide range of tem-<br>
transducer is used t **EXAMPLE 11**<br> **Example 19**<br> **Example 19**<br> **Example absorber (sample under the study)** instrument suitable to operate at a wide range of tem-<br> **Example 19**<br> **Example instrument suitable to operate at a wide range of tem-**<br>  $\pm \frac{1}{2}$  (*L Electrochem.*) 2022, 28(3), 2108541 (13 of 31)<br>
temperature during measurement, which make this sweep method in which a moving driver or velocity<br>
instrument suitable to operate at a wide range of tem-<br>  $\pm \frac{1}{2}$  (*k*<sup>22</sup>(*k k betwochem*) 2022, 28(3), 2108541 (13 of 31)<br>temperature during measurement, which make this sweep method in which a moving driver or velocity<br>instrument suitable to operate at a wide range of **Example 19** and the source and detector of  $14.4 \text{ keV} \gamma$ -rays, and is used to detect and some pind absorption of the source and is instrument suitable to operate at a wide range of tem-<br>transfluence is used to move the  $\pm$ ile  $\frac{1}{2}$  (*L Electrochem*.) 2022, 28(3), 2108541 (13 of 31)<br>
temperature during measurement, which make this sweep method in which a moving driver or velocity<br>
instrument suitable to operate at a wide range of  $\pm \frac{1}{2}$  (*L Electrochera*, ) 2022, 28(3), 2108541 (13 of 31)<br> **comperature** during measurement, which make this sweep method in which a moving driver or velocity<br>
instrument suitable to operate at a wide range of te  $4 \cdot 4 \cdot 2 \cdot 4$ <br>
temperature during measurement, which make this sweep method in which a moving driver or velocity<br>
instrument suidable to operate at a wide range of term larandour f is used to move the source or sample r  $\frac{1}{2}E^{\#}(L\ Etermechem.) 2022, 28(3), 2108541 (13 of 31)$ <br> **instrument suitable to operate at a wide range of tem-**<br> **instrument suitable to operate at a wide range of tem-**<br> **instrument suitable to operate at a wide range of tem EVALUATION EXACT ENECTORET THE SET USING SUBSAT (13 of 31)**<br> **Emperature during measurement,** which make this sweep method in which a moving driver or velocity<br>
instrument suitable to operate at a wide range of tem-<br>
tra mperature during measurer are spectral in the spectra is the section of the solution of the solution of (A) continues the simulate this may be interesting that this may be the solution of (A) continues are determined a mo  $28(3)$ ,  $2108541(13 of 31)$ <br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmitte 28(3), 2108541 (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmitted  $28(3)$ ,  $2108541$  (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmi 28(3), 2108541 (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmitted  $28(3)$ ,  $2108541$  (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmi  $28(3)$ ,  $2108541$  (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmi 28(3), 2108541 (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmitted 28(3), 2108541 (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmitted  $28(3)$ ,  $2108541(13 of 31)$ <br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmitte 28(3), 2108541 (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmitted 28(3), 2108541 (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmitted  $28(3)$ ,  $2108541$  (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmi 28(3), 2108541 (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmitted 28(3), 2108541 (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmitted 28(3), 2108541 (13 of 31)<br>sweep method in which a moving driver or velocity<br>transducer is used to move the source or sample re-<br>peatedly (so-called Doppler moving) with a specific<br>velocity, while  $\gamma$ -rays are transmitted Example 1920 and the memory of the memory and the memory and the memory of the memory of the memory of the source of sample repeatedly (so-called Doppler moving) with a specific velocity, while  $\gamma$ -rays are transmitted o 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (13 of 31)<br>
ent, which make this sweep method in which a moving driver or velocity<br>
the a wide range of tem-<br>
illustration of (A) con-<br>
peatedly (so-called Doppler moving) with



 $\frac{d}{dx}$  =  $\frac{d$  $\#E\#$  (*J. Electrochem.*) 2022, 28(3), 2108541 (14 of 31)<br>instantaneous velocity of the source by advancing ent physical models, but only a few of these models<br>memory address one by one. The triangular wave-<br>form and 10  $\text{E} \{E \neq L\}$ . *Electrochem.*) 2022, 28(3), 2108541 (14 of 31)<br>
instantaneous velocity of the source by advancing ent physical models, but only a few of these models<br>
memory address one by one. The triangular wave-<br>
fo  $\frac{45}{2}$  **Exerible 10.** Electrochem.) 2022, 28(3), 2108541 (14 of 31)<br>
instantaneous velocity of the source by advancing contriphysical models, but only a few of these models<br>
memory address one by one. The triangular w  $4.3$  instantaneous velocity of the source by advanced velocity.  $222, 28(3), 2108541 (14 of 31)$ <br>
instantaneous velocity of the source by advancing ont physical models, but only a few of these models<br>
memory address one by o  $\frac{4!}{2}$  (*k Rectrochem.*) 2022, 28(3), 2108541 (14 of 31)<br>
instantaneous velocity of the source by advancing contently produces the summer and published.<br>
form and 1024 memory channels produce two mir-<br>  $\frac{1}{2}$  chanspectrum.

form and 1024 memory channels produce two mi-<br>
ror-imaged Mössbaurs spectra each with 512 chan-<br>
ror-imaged Mössbaurs spectra each with which the Six former to produce a model spectram with which<br>
ror-imaged Mössbaurs spe ror-imaged Missbauer spectra each with 512 chan-<br>
is software to produce a model spectrum with which<br>
nels. With sufficiently good velocity linearity, the two measured spectrum could be compared, fitted, and<br>
spectra can nels. With sufficiently good velocity linearity, the two measured spectrum could be compared, fitted, and<br>spectra can be easily combined (to increase the signal and product. The fitting of the Mossbauer spectrum<br>for noise spectra can be casily combined (to increase the signal analyzed. The fitting of the Mössbauer spectrum,<br>
to noise ratio) to give one 512 channel Mössbauer should not only be carried out on the obtained data,<br>
spectrum.<br> to noise ratio) to give one 512 channel Missbauer should not only be carried out on the obtained data,<br>
spectrum.<br>
4.3 Data Analysis and Fitting to the first also consider a theoretical model. This is be-<br>
4.3 Data Analys but first also consider a theoretical model. This is be-<br>4.3 Data Analysis and Fitting the acts fitting could get superficially unreasonable<br>4.2 Data Data form experiment, the next<br>chi-squared value ( $\chi$ ) if based on un **4.3 Data Analysis and Fitting** cause fitting could get superficially unreasonable<br>After obtaining data from experiment, the next chi-sequend value ( $\chi$ ) if based on unphysical model.<br>
After obtaining data from experimen After obtaining data from experiment, the next<br>
schies and collect ( $\chi$ ) if based on umphysical model.<br>
step is analysis and fitting of the spectra to determine<br>
Commonly, three are three different line shapes em-<br>
the a step is analysis and fitting of the spectra to determine<br>the active phase sem-<br>the active phase structure and valence states of Fe in phoyed for modeling of a Müssbauer spectrum as<br>the catalyst. The fitting of data is usu the active phase structure and valence states of Fe in ployed for modeling of a Mossbauer spectrum as<br>the catalyst. The fitting of data is usually approximate shown in Figure 9. The combination of Lorentzian<br>delafter comp the catalyst. The fitting of data is usually approximat-<br>shown in Figure 9. The combination of<br>ed after comparison with previously available data in<br>and Gaussian line resultant is called as the<br>help in the iterature or da after comparison with previously available data in and Gaussian line resultant is called as the Voigt line<br>
literature or database system. The past data can shape which is referred to as a pseudo-Voigt function<br>
lip in th the literature or database system. The past data can<br>
shape which is referred to as a pseudo-Voigt function<br>
helap in the attribution and screening of the object<br>
since shape, valence state, spin state and coordination<br>
E help in the attribution and screening of the object<br>
since it is a linear mixture of the two shapes, valence state, spin state and coordination<br>
structure based on the analytical and filted results. Tratory to laboratory. phase, valence state, spin state and coordination Frror in analysis is obvious and it varies from labor-<br>structure based on the analytical and fitted results. Tatoy to laboratory. Double traens are typically state<br>of Ther structure based on the analytical and fitted results. ratory to laboratory. Doublet areas are typically stated<br>Therefore, the database system was developed which to no better than one significant place alter the deci-<br>for Therefore, the database system was developed which<br>
is a globally connocted network of different ecnters<br>
mall somer shift ( $\delta$ ) and quadrupole splitting ( $\Delta$ ) values<br>
is a globally connocted network of different ecnter

ters collect data from the Müssbauer analysis of the field (*B*) values are exceedingly crratic. In general, posibly absolut reduction to the upublished literature where it can be used for fitting Mössbauer technique can published literature where it can be used for fitting Mössbauer technique can detect down to 1% of the encrits of future materials. One of the biggest to fall normal Fis in the sample, but challenging for the secure and sa and analysis of future materials. One of the biggest total normal Fc in the sample, but challenging for the centres of Müssbaucr effect, where the reference and samples containing less than 0.1% weight Fe, the data collec centers of Misschauer effect, where the reference and samples containing less than 0.1% weight Fc, the data collected from the whole world for different Misschauer signal is available to be largely enhanced Misschauer sim data collected from the whole world for different Missbauer signal is available to be largely enhanced<br>Missbauer samples, is Missbauer Effect Data Centre if the emrichd isotope <sup>7</sup>Fe is used for preparing the<br>Missbauer sa Messbauer samples, is Missbauer Fiffect Data Center if the enriched isotope <sup>9</sup>Fe is used for preparing the andarm Institute of Chemisel Sciencess, Chinese somples. Analemy of Sciences (https://med. dicp.ac.en/)<sup>641</sup>. Tak

电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (14 of 31)<br>source by advancing ent physical models, but only a few of these models<br>The triangular wave-<br>have been compared and published.<br>els produce two mir-<br>A variety of physi

 $\frac{4}{3}$  for the source by advancing and  $\frac{4}{3}$  channel  $\frac{4}{3}$  controllar to source by advancing and physical models, but only a few of these models<br>memory address one by one. The triangular wave-<br>form and 1024 mem  $\pm$  *H*/ $\pm$ <sup>2</sup>/*L Electrochem.*) 2022, 28(3), 2108541 (14 of 31)<br>
instantaneous velocity of the source by advancing ent physical models, but only a few of these m<br>
memory address one by one. The triangular wave-<br>
form a **Example 19**<br> **Example 19**  $\frac{1}{2}$  **Example 18**<br> the  $\mathcal{C}_t(L\ Eermohen, 12022, 28(3), 2108541 (14 of 31)$ <br>
instantaneous velocity of the source by advancing ent physical models, but only a few of these models<br>
memory address one by one. The triangular wave-<br>
have been compared  $\frac{4}{5}$  ( $\frac{4}{5}$ ))) instantaneous velocity of the source by advancing can physical models, but only a few of these models<br>memory address one  $4.8 \div 2(1.5 \times 10^{13} \text{m})$ <br>
instantaneous velocity of the source by advancing ent physical models, but only a few of these models<br>
memory address one by one. The triangular wave-<br>
form and 1024 memory charges produce two the  $2^{\pm}(L \cdot R/eromshem.)$  2022, 28(3), 2108541 (14 of 31)<br>
instantaneous velocity of the source by advancing ent physical models, but only a few of these models<br>
memory address one by one. The triangular wave-<br>
have been comp instantaneous velocity of the source by advancing<br>
ent physical models, but only a few of these models<br>
memory address one by one. The triangular wave-<br>
have been compared and published.<br>
form and 1024 memory channels pro instantaneous velocity of the source by advancing ent physical models, but only a few of these models<br>nemmory address one by one. The triangular vavo-<br>hear form and 1024 memory channels produce two mi-<br> $\sim$  A variety of p memory address one by one. The triangular wave-<br>
form and 1024 memory channels produce two mir-<br>
A variety of physical models are used by the analy-<br>
form and 1024 memory channels produce two mir-<br>
a such varyety of physic 28(3), 2108541 (14 of 31)<br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br>
m 28(3), 2108541 (14 of 31)<br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br>
m  $(3)$ ,  $2108541$  ( $14$  of  $31$ )<br>t physical models, but only a few of these models<br>we been compared and published.<br>A variety of physical models are used by the analy-<br>software to produce a model spectrum with which<br>easured  $28(3)$ ,  $2108541$  (14 of 31)<br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with whi  $28(3)$ ,  $2108541 (14 of 31)$ <br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br> 28(3), 2108541 (14 of 31)<br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br> 28(3), 2108541 (14 of 31)<br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br> 28(3), 2108541 (14 of 31)<br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br> 28(3), 2108541 (14 of 31)<br>ent physical models, but only a few of these models<br>have been compared and published.<br>A variety of physical models are used by the analy-<br>sis software to produce a model spectrum with which<br>measu 28(3), 2108541 (14 of 31)<br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br> )<br>
but only a few of these models<br>
and published.<br>
al models are used by the analy-<br>
e a model spectrum with which<br>
could be compared, fitted, and<br>
g of the Mössbauer spectrum<br>
arried out on the obtained data,<br>
a theoretic 28(3), 2108541 (14 of 31)<br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br> 28(3), 2108541 (14 of 31)<br>ent physical models, but only a few of these models<br>have been compared and published.<br>A variety of physical models are used by the analy-<br>sis software to produce a model spectrum with which<br>measu 28(3), 2108541 (14 of 31)<br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br> 28(3), 2108541 (14 of 31)<br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br> 28(3), 2108541 (14 of 31)<br>
ent physical models, but only a few of these models<br>
have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br> **Example 18 and Solution** and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br>
measured spectrum could be compared, fitted, and<br>
analyzed. The fitting o t physical models, but only a few of these models<br>we been compared and published.<br>A variety of physical models are used by the analy-<br>software to produce a model spectrum with which<br>assured spectrum could be compared, fit have been compared and published.<br>
A variety of physical models are used by the analy-<br>
sis software to produce a model spectrum with which<br>
measured spectrum could be compared, fitted, and<br>
analyzed. The fitting of the M A variety of physical models are used by the analy-<br>sis software to produce a model spectrum with which<br>measured spectrum could be compared, fitted, and<br>analyzed. The fitting of the Mössbauer spectrum<br>should not only be c sis software to produce a model spectrum with which<br>measured spectrum could be compared, fitted, and<br>analyzed. The fitting of the Mössbauer spectrum<br>should not only be carried out on the obtained data,<br>but first also cons measured spectrum could be compared, fitted, and<br>analyzed. The fitting of the Mössbauer spectrum<br>should not only be carried out on the obtained data,<br>but first also consider a theoretical model. This is be-<br>cause fitting be compared, fitted, and<br>the Mössbauer spectrum<br>out on the obtained data,<br>oretical model. This is be-<br>uperficially unreasonable<br>ased on unphysical model.<br>different line shapes em-<br> $\frac{1}{10}$  Mössbauer spectrum as<br>ombinati analyzed. The fitting of the Mössbauer spectrum<br>should not only be carried out on the obtained data,<br>but first also consider a theoretical model. This is be-<br>cause fitting could get superficially unreasonable<br>chi-squared

should not only be carried out on the obtained data,<br>but first also consider a theoretical model. This is be-<br>cause fitting could get superficially unreasonable<br>chi-squared value ( $\chi^2$ ) if based on unphysical model.<br>Com but first also consider a theoretical model. This is be-<br>cause fitting could get superficially unreasonable<br>chi-squared value ( $\chi^2$ ) if based on unphysical model.<br>Commonly, there are three different line shapes em-<br>ploy cause fitting could get superficially unreasonable<br>chi-squared value ( $\chi^2$ ) if based on unphysical model.<br>Commonly, there are three different line shapes em-<br>ployed for modeling of a Mössbauer spectrum as<br>shown in Figur chi-squared value  $(\chi^2)$  if based on unphysical model.<br>Commonly, there are three different line shapes em-<br>ployed for modeling of a Mössbauer spectrum as<br>shown in Figure 9. The combination of Lorentzian<br>and Gaussian line Commonly, there are three different line shapes em-<br>ployed for modeling of a Mössbauer spectrum as<br>shown in Figure 9. The combination of Lorentzian<br>and Gaussian line resultant is called as the Voigt line<br>shape which is re samples. own in Figure 9. The combination of Lorentzian<br>d Gaussian line resultant is called as the Voigt line<br>ape which is referred to as a pseudo-Voigt function<br>ce it is a linear mixture of the two shapes.<br>Error in analysis is ob and Gaussian line resultant is called as the Voigt line<br>shape which is referred to as a pseudo-Voigt function<br>since it is a linear mixture of the two shapes.<br>Error in analysis is obvious and it varies from labo-<br>ratory to shape which is referred to as a pseudo-Voigt function<br>since it is a linear mixture of the two shapes.<br>Error in analysis is obvious and it varies from labo-<br>ratory to laboratory. Doublet areas are typically stated<br>to no be since it is a linear mixture of the two shapes.<br>
Error in analysis is obvious and it varies from laboratory to laboratory. Doublet areas are typically stated<br>
to no better than one significant place after the deci-<br>
mal. Error in analysis is obvious and it varies from laboratory to laboratory. Doublet areas are typically stated to no better than one significant place after the decimal. Isomer shift ( $\delta$ ) and quadrupole splitting ( $\Delta$ ) v ratory to laboratory. Doublet areas are typically stated<br>to no better than one significant place after the deci-<br>mal. Isomer shift ( $\delta$ ) and quadrupole splitting ( $\Delta$ ) val-<br>ues are often  $\pm$  0.02 mm·s<sup>-1</sup>, and magnetic

is a globally connected network of different centers mal. Isomer shift ( $\delta$ ) and quadrupole splitting ( $\Delta$ ) val-<br>for Moissbauer cifet reference and data. These cen-<br>der as are often a flo.02.0 mm s<sup>-</sup>, and magnetic hype for Mösshauer effect reference and data. These cen-<br>
cers are often  $\pm$  0.02 mm -s'<sub>1</sub>, and magnetic hyperfine<br>
ters collect data from the Mossbauer analysis of the field (*B*) values are exceedingly reraic. In general,<br> to no better than one significant place after the deci-<br>mal. Isomer shift ( $\delta$ ) and quadrupole splitting ( $\Delta$ ) values<br>are often  $\pm$  0.02 mm·s<sup>-1</sup>, and magnetic hyperfine<br>field ( $B$ ) values are exceedingly erratic. In g mal. Isomer shift ( $\delta$ ) and quadrupole splitting ( $\Delta$ ) values are often  $\pm$  0.02 mm · s<sup>-1</sup>, and magnetic hyperfine field ( $B$ ) values are exceedingly erratic. In general, Mössbauer technique can detect down to 1% of t ues are often  $\pm$  0.02 mm ·s<sup>-1</sup>, and magnetic hyperfine<br>field (*B*) values are exceedingly erratic. In general,<br>Mössbauer technique can detect down to 1% of the<br>total normal Fe in the sample, but challenging for the<br>sam field (*B*) values are exceedingly erratic. In general,<br>Mössbauer technique can detect down to 1% of the<br>total normal Fe in the sample, but challenging for the<br>samples containing less than 0.1% weight Fe, the<br>Mössbauer si Mössbauer technique can detect down to 1% of the<br>total normal Fe in the sample, but challenging for the<br>samples containing less than 0.1% weight Fe, the<br>Mössbauer signal is available to be largely enhanced<br>if the enriched % of the<br>g for the<br>Fe, the<br>Fe, the<br>enhanced<br>aring the<br>de OECs,<br>sst equal<br>sed cata-<br>d of Fe<sup>3+</sup><br>mply ap-<br>nes, only<br>pecies in<br>ll spectra<br>D Loren-<br>, and the<br>pecies in<br>Loren-<br>.<br>Loren-<br>.<br>Loren-<br>.<br>Loren-<br>.<br>Loren-<br>.<br><br><br><br>d fine pe total normal Fe in the sample, but challenging for the samples containing less than 0.1% weight Fe, the Mössbauer signal is available to be largely enhanced if the enriched isotope  ${}^{57}Fe$  is used for preparing the sampl mples containing less than 0.1% weight Fe, the sissbauer signal is available to be largely enhanced the enriched isotope <sup>57</sup>Fe is used for preparing the mples. Taking an example of NiFe-(oxy)hydroxide OECs, nerally two s Mössbauer signal is available to be largely enhanced<br>if the enriched isotope <sup>57</sup>Fe is used for preparing the<br>samples.<br>Taking an example of NiFe-(oxy)hydroxide OECs,<br>generally two symmetrical peaks of almost equal<br>height if the enriched isotope <sup>57</sup>Fe is used for preparing the samples.<br>
Taking an example of NiFe-(oxy)hydroxide OECs, generally two symmetrical peaks of almost equal height are expected for pre-OER powder-based cata-lyst, ind



**Example 1.1** and the fitted with the spectral method of the *spectral method in* and the spectral method of the spectral method of the method of the spectral method of the spectral method of the spectral method of the sp **Example the solution of Example CER**-emhancing impact was<br> **Example OER-emhancing impact** was<br> **Example the solution of**  $\frac{1}{2}$ <br> **Example the solution of**  $\frac{1}{2}$ <br> **Example the solution of**  $\frac{1}{2}$ <br> **Example the Velocity (mm/s)**<br> **Shows** the different conduction of the shapes enployed for approximation of which showed similar improvement in OER activity<br>
practically measured "Fe Morssbeure spectra. (color on line) of NO. Nonchel **Eignre 9** Different line shapes employed for approximation of<br>which showed similar improvement in OER activity<br>practically measured <sup>5</sup>re Missionary eperta (color on line) of NiO. Nonethelets, the degree of Ce<sup>r</sup> -induce reparely. The spectra and the super-<br>
interaction of NO. Nonetheles, the degree of Ce<sup>1</sup>-induced enhancement  $(T_{\rm vir})$ , and the relative resonance area<br>
hancement was near to that of Fe<sup>1+</sup>-induced enhancement<br>
All-franxim Franchinal contents in the state of the state of the state of the matter of the cataloge of the different components of the absorption parameter (A) of the different components of the absorption parameter (A) of the diffe half-maximum  $(I^*_{\infty})$ , and the relative resonance ament. However, until 2014-2015, no significant effort<br>
(A) of the different components of the shorpton pat-<br>
(A) of the different components of the shorpton pat-<br>
tens. half-maximum  $(T_{\text{eq}})$ , and the relative resonance area<br>
was made to give a logical and acceptable explana-<br>
(A) of the different components of the absorption pat-<br>
terms. In the case of two different kinds of Fe<sup>3+</sup> dec (A) of the different components of the absorption pat-<br>
tion for this enhancement. And for more the species in the system which could arise due to different kinds of Fe<sup>3</sup><br>
decades, these findings of Fe impurities-ba<br>
spe terns. In the case of two different kinds of  $Fe<sup>+</sup>$  decades, these lindings of Fe imputties-based engences in the system which could arise due to differ-<br>ence in coordination at the surface and inside to life-<br>time of ence in coordination at the surface and inside the lat-<br>
completely hidden.<br>
tice, the fitting of the spectrum may not be simple<br>
Since Mössbauer study of NiFe-C<br>
and could require fitting with two doublets or one reasona

**Half-Half-Maximum (***Y***<sub>m</sub>**), and the relative resonance area the relation of the relation of the angular contention of the angular contention of the relation of the relation of the relation of the relation of the relatio **Example the different components of the absorption** of the different components of the patent of the patent of the absorption of the different components of the absorption pat-<br> **Example of the strength components of the Example the control in the cases of two different color of the significant**<br> **Examplementarial**<br> **Examplementarism Constructed and the significant<br>
<b>Examplementarism Constructed** and  $\mathbf{r}$  the significant<br> **Examplemen Solution**<br>
Solution and the system of the significant research in the system of the significant<br>
species in which the discovered that when Fe impurities are injected into NiO andots, the OER activity<br>
increases<sup>191</sup>. Aft **Example 19** materials. Corrigan conducted one of the significant<br>
researches in which he discovered that when Fe impurities<br>  $\frac{1}{4}$  of  $\frac{$ **The species of the spectrum may not be simpled to the spectrum may not be summarized for their impact once and internal in the spectrum may not be the spectrum may of the spectrum may other and outder of the spectrum may** The maximal could require the species of the spectrum may not be sumpted to the could require the could require the special of the spectral of the special of the spectral of the spectral of the spectral of the spectral of **Example 1.1** The meanse of two different kinds of Fe<sup>1</sup><br>
Figure 9 Different line shapes employed for approximation of the preformance by impregrating into NiO electrodys<sup>191</sup>.<br> **Primere 9** Different line shapes employed **Figure 9 Different limes of the spectra as Feederal Confirmed intervention and the spectra as Federal Confirmed intervention of Figure 9 Different line shapes employed for approximation of the spectra confirmed be confir** 28(3), 2108541 (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one of the 28(3), 2108541 (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one of the  $28(3)$ , 2108541 (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one of  $28(3)$ ,  $2108541$  (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one o 28(3), 2108541 (15 of 31)<br>
search on electrochemical water splitting processes<br>
utilizing non-precious metal oxides and hydroxides to<br>
replace the precious metal ones and costly anodic<br>
materials. Corrigan conducted one o  $28(3)$ ,  $2108541$  (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one o  $28(3)$ ,  $2108541$  (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one o  $28(3)$ ,  $2108541$  (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one o  $28(3)$ ,  $2108541$  (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one o 28(3), 2108541 (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one of th 28(3), 2108541 (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one of th 28(3), 2108541 (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one of th 28(3), 2108541 (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one of th 28(3), 2108541 (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one of th  $28(3)$ ,  $2108541$  (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one o search on electrochemical water splitting processes<br>search on electrochemical water splitting processes<br>utilizing non-precious metal ones and costly anodic<br>materials. Corrigan conducted one of the significant<br>researches i search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one of the significant<br>researches in utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one of the significant<br>researches in which he discovered that when Fe impu-<br>rities are i replace the precious metal ones and costly anodic<br>materials. Corrigan conducted one of the significant<br>researches in which he discovered that when Fe impu-<br>rities are injected into NiO anodes, the OER activity<br>increases<sup>[</sup> materials. Corrigan conducted one of the significant<br>researches in which he discovered that when Fe impu-<br>rities are injected into NiO anodes, the OER activity<br>increases<sup>[32]</sup>. Afterward, several other transition metal<br>ra earches in which he discovered that when Fe impu-<br>es are injected into NiO anodes, the OER activity<br>reases<sup>[32]</sup>. Afterward, several other transition metal<br>ions were investigated for their impact on OER<br>formance by impregn rities are injected into NiO anodes, the OER activity<br>increases<sup>[32]</sup>. Afterward, several other transition metal<br>cations were investigated for their impact on OER<br>performance by impregnating into NiO electrodes<sup>[35]</sup>.<br>But increases<sup>[32]</sup>. Afterward, several other transition metal<br>cations were investigated for their impact on OER<br>performance by impregnating into NiO electrodes<sup>[35]</sup>.<br>But not a comparable OER-enhancing impact was<br>observed fr cations were investigated for their impact on OER<br>performance by impregnating into NiO electrodes<sup>[35]</sup>.<br>But not a comparable OER-enhancing impact was<br>observed from any other *d*-block element except Ce<sup>4+</sup>,<br>which showed performance by impregnating into NiO electrodes<sup>135</sup>.<br>But not a comparable OER-enhancing impact was<br>observed from any other *d*-block element except Ce<sup>++</sup>,<br>which showed similar improvement in OER activity<br>of NiO. Nonethe But not a comparable OER-enhancing impact was<br>observed from any other  $d$ -block element except Ce<sup>+t</sup>,<br>which showed similar improvement in OER activity<br>of NiO. Nonetheless, the degree of Ce<sup>+1</sup>-induced en-<br>hancement was n **Example 19**<br> **Example 19**<br> **Example 19**<br> **Example 2014**<br> **Example 2014**<br> **Example 2014**<br> **Example 2014**<br> **Example 2014**<br> **Example 30**<br> **Example 19**<br> **Example 19** 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (15 of 31)<br>search on electrochemical water splitting processes<br>utilizing non-precious metal oxides and hydroxides to<br>replace the precious metal ones and costly anodic<br>materials.

species in the system which could arise due to differ-<br>
lanced OER performance of Ni-based materials were<br>
einc, the filting of the spectrum may not be simple<br>
circ, the filting of the spectrum may not be simple<br>
complete tice, the fitting of the spectrum may not be simple<br>
since Mossbauer study of NiFe-OECs provided<br>
and could require fitting with two doublets are one simple. The presence of high valence<br>
doublet and one singlet. The pres could require fitting with two doublets or one<br>
some reasonable explanations to this unprecedented<br>
but and one signica. The presence of high valence<br>
such as Fe<sup>t</sup> could be confirmed if the spectral<br>
and signical doublet Source and one singular conservation of the presence of napar values of the *singular conservation* and the sycket Fr such as Fr could are constrained it to spectral<br>
respect to the simulate parameters for the constrained of Niesbauer electrochemical<br>
could not be fitted with only one doublet and an iso-<br>
we discuss the key challengin could not be intered with any one odubits and an iso-<br>
end the metally among the since a very small among the since the since the since the since of the since the me smit is onserved around are on ever negative<br>
we discuss the key challenging issues and parameters<br>
be confirmed by comparing pre-OER and post-OER<br>
for OER reaction to observe the evolution of the<br>
spectra by fitting c sinc. Moreover, stansity or catalystrative can also the dovelop an *in-situloperando* Mössbauer instrument<br>the confirmed by comparing pre-OFR and post-OFR to OER reaction to observe the colution of the<br>spectra by fitting observed from any other *d*-block element except Ce<sup>++</sup>, which showed similar improvement in OER activity of NiO. Nonetheless, the degree of Ce<sup>4+</sup>-induced enhancement. However, until 2014-2015, no significant effort was which showed similar improvement in OER activity<br>of NiO. Nonetheless, the degree of Ce<sup>4+</sup>-induced en-<br>hancement was near to that of Fe<sup>3+</sup>-induced en-<br>hancement. However, until 2014-2015, no significant effort<br>was made t of NiO. Nonetheless, the degree of Ce<sup>\*1</sup>-induced en-<br>hancement was near to that of Fe<sup>3+</sup>-induced enhance-<br>ment. However, until 2014-2015, no significant effort<br>was made to give a logical and acceptable explana-<br>tion for hancement was near to that of Fe<sup>3+</sup>-induced enhance-<br>ment. However, until 2014-2015, no significant effort<br>was made to give a logical and acceptable explana-<br>tion for this enhancement. And for more than two<br>decades, thes ment. However, until 2014-2015, no significant effort<br>was made to give a logical and acceptable explana-<br>tion for this enhancement. And for more than two<br>decades, these findings of Fe impurities-based en-<br>hanced OER perfo was made to give a logical and acceptable explana-<br>tion for this enhancement. And for more than two<br>decades, these findings of Fe impurities-based en-<br>hanced OER performance of Ni-based materials were<br>completely hidden.<br>S tion for this enhancement. And for more than two<br>decades, these findings of Fe impurities-based en-<br>hanced OER performance of Ni-based materials were<br>completely hidden.<br>Since Mössbauer study of NiFe-OECs provided<br>some rea decades, these findings of Fe impurities-based en-<br>hanced OER performance of Ni-based materials were<br>completely hidden.<br>Since Mössbauer study of NiFe-OECs provided<br>some reasonable explanations to this unprecedented<br>OER ac hanced OER performance of Ni-based materials were<br>completely hidden.<br>Since Mössbauer study of NiFe-OECs provided<br>some reasonable explanations to this unprecedented<br>OER activity, it became crucial to develop and im-<br>prove completely hidden.<br>
Since Mössbauer study of NiFe-OECs provided<br>
some reasonable explanations to this unprecedented<br>
OER activity, it became erucial to develop and im-<br>
prove the *in-situloperando* Mössbauer electrochemi-Since Mössbauer study of NiFe-OECs provided<br>some reasonable explanations to this unprecedented<br>OER activity, it became crucial to develop and im-<br>prove the *in-situ/operando* Mössbauer electrochemi-<br>cal setup and experime some reasonable explanations to this unprecedented<br>OER activity, it became crucial to develop and im-<br>prove the *in-situ/operando* Mössbauer electrochemi-<br>cal setup and experimental understandings. Firstly,<br>we discuss the OER activity, it became crucial to develop and im-<br>prove the *in-situ/operando* Mössbauer electrochemi-<br>cal setup and experimental understandings. Firstly,<br>we discuss the key challenging issues and parameters<br>to develop a prove the *in-situ/operando* Mössbauer electrochemical setup and experimental understandings. Firstly, we discuss the key challenging issues and parameters to develop an *in-situ/operando* Mössbauer instrument for OER rea cal setup and experimental understandings. Firstly,<br>we discuss the key challenging issues and parameters<br>to develop an *in-situ/operando* Mössbauer instrument<br>for OER reaction to observe the evolution of the<br>chemical stat we discuss the key challenging issues and parameters<br>to develop an *in-situ/operando* Mössbauer instrument<br>for OER reaction to observe the evolution of the<br>chemical state of the iron dynamically in the catalyst<br>and struct to develop an *in-situ/operando* Mössbauer instrument<br>for OER reaction to observe the evolution of the<br>chemical state of the iron dynamically in the catalyst<br>and structure of the catalyst during electrochemical<br>reactions.

<sup>i</sup> if  $k\neq (L \text{ *Electrochem.*})$  2022, 28(3), 2108541 (16 of 31)<br>
attached with CHI660E electrochemical station. The and easily machined material<br>
Doppler velocity of the spectrometer is calibrated sorption property. An elect

**EMALE 1989**<br> **EMALE 1989**  $#E\# (J. \nElectrochem.) 2022, 28(3), 2108541 (16 of 31)$ <br>
attached with CHI660E electrochemical station. The and easily machined material with very low  $\gamma$ -ray ab-<br>
Doppler velocity of the spectrometer is calibrated sorption prope  $# \langle \mathcal{K}^{\#}(J. \text{Electrochem.}) 2022, 28(3), 2108541 \text{ (16 of 31)}$ <br>
attached with CHI660E electrochemical station. The and easily machined material with very low γ-ray ab-<br>
Doppler velocity of the spectrometer is calibrated sorpt national recommended standardization. Generally, **Example 1988**<br> **Example 1988**<br> **Example 1988**<br> **Example 1999**<br> **Example 1999 EVALUATION ENTITLE (FOCUS ACCONDMENTATION CONTROLLATION CONTROLLATION CONTROLLATION CONTROLLATION CONTROLLATION**<br> **EXECUTE:** THE SCONDIST CONTROLLATION CONTROLLATION EXECUTION THE SCONDIST CONTROLLATION EXPLAINING THE SC **EVALUATION**<br> **EXECUTE:** Extended with CHI660E electrochemical station. The and easily machined material with very low y-ray ab-<br>
Doppler velocity of the spectrometer is calibrated sorption property. An electrochemical re **Example 19**<br> **Example 19 Example 10**<br>
We diff-difference that the self-developed several the self-state of intervals and the self-state of the self-developed several serveral several several several the respect to  $\alpha$ -Fe at 298 K according to t **EVALUATION EXAMORE (EXAMORE 1992)**, 2008541 (16 of 31)<br> **Example 1998** whether when the spectrometer is calibrated sorption property. An electrochemical reaction cell<br>
Doppler velocity of the spectrometer is calibrated s **HE-F(L Electrochem.) 2022. 28(3), 2108541 (16 of 31)**<br> **1112chocharm ME-MEGOOE** electrochemical station. The and easily machined material with very low y-ray ab-<br> **1200** Doppler velocity of the spectrometer is calibrated **EVALUATION THE CONSECT (EXECT AND ART (16 of 31)**<br> **EUROPER VIOLET AND CONSECT AND CONSECT AND AND CONSECT AND CONSECT AND ART (16 of 21)**<br> **EUROPER VIOLET (2008) EVALUATION CONSECT AND ART (2009) EVALUATION MATEL WI EXALURE 1999**<br> **EXALURE 1999** 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situ/operando* Mössbauer<br>spectral measurements is shown i 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for  $in-situ/operando$  Mössbauer<br>spectral measurements is shown in Figure 1 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situ/operando* M össbauer<br>spectral measurements is shown 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situloperando* Mössbauer<br>spectral measurements is shown i 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situ/operando* Mössbauer<br>spectral measurements is shown i 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situloperando* Mössbauer<br>spectral measurements is shown i 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situloperando* Mössbauer<br>spectral measurements is shown i 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situloperando* Mössbauer<br>spectral measurements is shown i 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situloperando* Mössbauer<br>spectral measurements is shown i 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situloperando* Mössbauer<br>spectral measurements is shown i 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situloperando* Mössbauer<br>spectral measurements is shown i 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situloperando* Mössbauer<br>spectral measurements is shown i 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situloperando* Mössbauer<br>spectral measurements is shown i 28(3), 2108541 (16 of 31)<br>and easily machined material with very low  $\gamma$ -ray ab-<br>sorption property. An electrochemical reaction cell<br>with high versatility for *in-situloperando* Mössbauer<br>spectral measurements is shown i 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (16 of 31)<br>
chemical station. The and easily machined material with very low γ-ray ab-<br>
rometer is calibrated sorption property. An electrochemical reaction cell<br>
according to



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working electrode area leaves from upper side of **EVALUATION**<br> **EXALUAT CONDEGENDER (Example 1922, 28(3), 2108541 (17 of 31)**<br> **Exaction coll where the electrolyte enters from lower** window of the same size as the *in-situloperando* cell,<br>
part of reference electrode an **EVALUATION**<br> **EXALUAT CONDEVALUAT CONDEND**<br> **EXALUAT CONDEND**<br> **EXAL**  $\pm \frac{(\cancel{E} + \cancel{E}) \cdot E \cdot (E \cdot E \cdot \text{F})}{(\cancel{E} + \cancel{E}) \cdot E \cdot \text{F}}$ <br>
Traction cell where the electrolyte enters from lower window of the same size as the *in-situloperando* cell,<br>
part of reference electrode and after passing throu **Example 19**<br> **Reaction cell where the electrolyte enters from lower** window of the same size as the *in-situloperando* cell,<br>
part of reference electrode and after passing through is pressed to the cell's front, ensuring  $4E\div(L \nEleurochem) 2022, 28(3, 2108541 (17 of 31)$ <br>
reaction cell where the electrolyte enters from lower window of the same size as the *in-situloperando* cell,<br>
part of reference electrode and after passing through is pressed t **EVALUATION**<br> **EXECUTE:** Electrochem.) 2022, 28(3), 2108541 (17 of 31)<br> **Exaction coll where the electrolyte enters from lower** window of the same size as the *i*<br>
part of reference electrode and after passing through is **Example 19**<br> **EXAMPLE 19** 

The fabrication of electrode for *in-situloperando* tional <sup>3</sup>Fe Mässbauer spectra measurement, in which<br>Mössbauer test is very sensitive task and necds high the <sup>8</sup><sup>Fe</sup> nucleus, one kind of the Fe isotopes with on-<br>Micro Mossbauer test is very sensitive task and needs high the <sup>5</sup>Fe nucleus, one kind of the Fe isotopes with onti-<br>nite of the catalyst. In a typical OFR experiment in electrochemical <sup>5F</sup>e Missbauer testing, the catalyst is<br> attention. For this purpose, the key step is to prepare  $1y \sim 2.2\%$  natural abundance. For the *in-situdoperando*<br>ink of the caths/st. In a typical ORR cypreiment in electrochemical <sup>3</sup>Fe Müssbauer testing, the catalyst ink of the catalyst. In a typical OER experiment in electrochemical <sup>7</sup>Fe Mössbauer testing, the catalyst is<br>our hab, 10 mg of catalyst powder is added into solu-<br>recommended to be prepared using enriched <sup>5</sup>Fe iso-<br>orion our lab, 10 mg of catalyst powder is added into solu-<br>recommended to be prepared using emriched <sup>5</sup>Fe iso-<br>into continuing 40 µL Nation, 480 µL isopropanol, topos for calmancing the resonant absorption signals.<br>and 480 µL

 $\# \# \# (J. Electrochem.)$  2022, 28(3), 2108541 (17 of 31)<br>reaction cell where the electrolyte enters from lower window of the same size as the *in-situloperando* cell,<br>part of reference electrode and after passing through is presse part of reference electrode and after passing through 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situ/operando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situloperando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situloperando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situ/operando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays 28(3), 2108541 (17 of 31)<br>
window of the same size as the *in-situ/operando* cell,<br>
is pressed to the cell's front, ensuring that the Möss-<br>
bauer  $\gamma$ -rays only can enter from the cell's window,<br>
and the Mössbauer  $\gamma$ -r 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situloperando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays (3), 2108541 (17 of 31)<br>
mdow of the same size as the *in-situ/operando* cell,<br>
pressed to the cell's front, ensuring that the Möss-<br>
uer  $\gamma$ -rays only can enter from the cell's window,<br>
d the Mössbauer  $\gamma$ -rays in othe 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situloperando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (17 of 31)<br>
lyte enters from lower window of the same size as the *in-situ/operando* cell,<br>
after passing through is pressed to the cell's front, ensuring that the Möss-<br>
s fr

 $\pm k \neq 0$ . *Electrochem.*) 2022, 28(3), 2108541 (17 of 31)<br>
the electrolyte enters from lower<br>
window of the same size as the *in-situ/operand*<br>
ctrode and after passing through is pressed to the cell's front, ensuring t **EVALUATION**<br> **EXALUATION THE CONTEXANT (17 (T31)**<br> **EXALUATE:**<br> **EVALUATION THE THE CONSERV (FOR THE THE CONSERVANT (17 of 31)**<br> **Fraction cell where the electrolyte enters from lower** window of the same size as the *in-situ/operundo* cell,<br>
part of reference clectrode and after passi **ig**  $f(\frac{2\pi}{4})$ . *Electrochem*, 2022, 28(3), 2108541 (17 of 31)<br>
practicion cell where the clectrolyte caters from lower window of the same size as the *in-situtogecuado* cell,<br>
part of reference electrode and after pas **EVALUATION**<br> **Example 19 mg**<br> **Ex** ig the  $\frac{4}{3}$  the  $\frac{4}{3}$  Electrochem.) 2022, 28(3), 2108541 (17 of 31)<br>
reaction coll where the electrolyte enters from lower window of the same size as the *in-situ/operando* cell,<br>
part of reference clectrode and reaction cell where the electrolyte cursor and since several in the variable cells, the simulatoperando cell,<br>part of reference electrode and after passing through in pressed to the same size as the in-situatioperando cel reaction cell where the electrolyte enters from lower<br>window of the same size as the *in-stituloperando* cell,<br>part of reference electrode and after passing through<br>the pressed to the ed!'s front, ensuing that Miss-<br>parti part of reference electrode and after passing through<br>
is pressed to the cell's front, ensuring that the Möss-<br>
vorking electrode are lacent of the base from upper side of baser y-rays any can enter from the cell's window working electrode area leaves from upper side of bauer y-rays only ean enter from the cell's window, counter electrole. This kind of flow style can help to and the Mossbauer y-rays in other directions are contrelected. Th counter electrode. This kind of flow style can help to<br>
and the Müssbauer  $\gamma$ -rays in other directions are<br>
maintain the pH during the electrochemical reaction. Shielded by the lead plate. We call this specific lead<br>
The maintain the pH during the electrochemical reaction.<br>
Sincelleded by the lead plate. We call this specific lead<br>
The flow rate of both pumps kccps same during the plate a Missbaurer spectrometre collimator.<br>
The flow rate The flow rate of both pumps keeps same during the<br>
reaction and the amount of electrolyte in a beaker<br>
centeration and the moment of electrolyte in a beaker<br>
centeration is recommended in case of normal iron containing<br>
t reaction and the amount of electrolyte in a beaker<br>
fenerally,  $5 \sim 10$  mg Fe cm<sup>3</sup> of absorber thickness<br>
should be cnosed such as 100 ~ 200 mL to maintain is recommended in case of normal iron containing<br>
the pH of 0.1 should be enough such as 100 ~ 200 mL to maintain is recommended in case of normal iron containing<br>the pH of 0.1 mol - L<sup>+</sup> KOH solution. Substance used in eatalyst preparation for a conven-<br>The fabrication of electrode f the pH of 0.1 mol-L" KOH solution.<br>
The fabrication of clectroche for *in-situal-pertando*<br>
The fabrication of clectrochemic of correct and incell and incell and the periodiscurs pectra measureman, in which<br>
Missishauer t 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situ/operando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situloperando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situ/operando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situloperando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situloperando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situ/operando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays 28(3), 2108541 (17 of 31)<br>window of the same size as the *in-situloperando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays **Excess transformation** sizes as the *in-situloperando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer γ-rays only can enter from the cell's window,<br>and the Mössbauer γ-rays in other directions are<br>s window of the same size as the *in-situloperando* cell,<br>is pressed to the cell's front, ensuring that the Möss-<br>bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays in other directions are<br>sh is pressed to the cell's front, ensuring that the Mössbauer  $\gamma$ -rays only can enter from the cell's window, and the Mössbauer  $\gamma$ -rays in other directions are shielded by the lead plate. We call this specific lead plate bauer  $\gamma$ -rays only can enter from the cell's window,<br>and the Mössbauer  $\gamma$ -rays in other directions are<br>shielded by the lead plate. We call this specific lead<br>plate a Mössbauer spectrometer collimator.<br>Generally,  $5 \sim$ and the Mössbauer  $\gamma$ -rays in other directions are<br>shielded by the lead plate. We call this specific lead<br>plate a Mössbauer spectrometer collimator.<br>Generally,  $5 \sim 10$  mg Fe cm<sup>-2</sup> of absorber thickness<br>is recommended i shielded by the lead plate. We call this specific lead<br>plate a Mössbauer spectrometer collimator.<br>Generally,  $5 \sim 10$  mg Fe cm<sup>2</sup> of absorber thickness<br>is recommended in case of normal iron containing<br>substance used in ca plate a Mössbauer spectrometer collimator.<br>
Generally,  $5 \sim 10$  mg Fe cm<sup>2</sup> of absorber thickness<br>
is recommended in case of normal iron containing<br>
substance used in catalyst preparation for a conven-<br>
tional <sup>57</sup>Fe Möss Generally,  $5 \sim 10$  mg Fe cm<sup>2</sup> of absorber thickness<br>is recommended in case of normal iron containing<br>substance used in catalyst preparation for a conven-<br>tional <sup>57</sup>Fe Mössbauer spectra measurement, in which<br>the <sup>57</sup>Fe is recommended in case of normal iron containing<br>substance used in catalyst preparation for a conven-<br>tional <sup>5</sup>Fe Mössbauer spectra measurement, in which<br>the <sup>5</sup>Fe nucleus, one kind of the Fe isotopes with on-<br>ly ~ 2.2% substance used in catalyst preparation for a conventional <sup>5</sup>Fe Mössbauer spectra measurement, in which<br>the <sup>5</sup>Fe nucleus, one kind of the Fe isotopes with on-<br>ly ~ 2.2% natural abundance. For the *in-situ/operando*<br>elect tional <sup>57</sup>Fe Mössbauer spectra measurement, in which<br>the <sup>57</sup>Fe nucleus, one kind of the Fe isotopes with on-<br>ly ~ 2.2% natural abundance. For the *in-situ/operando*<br>electrochemical <sup>57</sup>Fe Mössbauer testing, the catalyst the <sup>57</sup>Fe nucleus, one kind of the Fe isotopes with on-<br> $ly \sim 2.2\%$  natural abundance. For the *in-situloperando*<br>electrochemical <sup>57</sup>Fe Mössbauer testing, the catalyst is<br>recommended to be prepared using enriched <sup>57</sup>Fe  $\frac{1}{2}N \sim 2.2\%$  natural abundance. For the *in-situloperando*<br>electrochemical <sup>37</sup>Fe Mössbauer testing, the catalyst is<br>recommended to be prepared using enriched <sup>37</sup>Fe iso-<br>topes for enhancing the resonant absorption electrochemical <sup>37</sup>Fe Mössbauer testing, the catalyst is<br>recommended to be prepared using enriched <sup>37</sup>Fe iso-<br>topes for enhancing the resonant absorption signals.<br>Compared with the window material and the carbon<br>paper s commended to be prepared using enriched <sup>57</sup>Fe iso-<br>
exercises for enhancing the resonant absorption signals.<br>
Impared with the window material and the carbon<br>
per substrate, the electrolyte solution is the greatest<br>
ectin



part in the OER reaction and enough amount of Fe<br>
content to detect good quality spignals.<br>
On the other good quality spignals.<br>
On the other good quality signals.<br>
On the other hand, we prefer a continuous flow of hours content to detect good quality signals.<br>
and removal of applied potentials, and then alter 4%<br>
on the other hand, we prefer a continuous flow of thous to  $22 \text{ hours}$  when the clectrode is fully drive<br>dectrodyte rather than On the other hand, we prefer a continuous flow of<br>bours to 72 hours when the electrode is fully dried.<br>Some time as using a small amount of electrolyte and<br>the structure capability at during electrode is they are therefor electrolyte rather than changing electrolyte after<br>
miss will give a clear understanding about the struc-<br>
some time as using a small amount of electrolyte and<br>
ture stability adit in erequired for recovering the o-<br>
high some time as using a small amount of electrolyte and<br>
highly are furge slobility and time required for recovering the<br>
highly are timited between celectroscondalizer and states are riginal structure capability of the cata highly active electrocatalyst may result in change of<br>
priginal structure capability of the elatalyst. If the<br>
electrolyte which could alter the reaction<br>
pre-OFR and post-OFB and post-OFB are solicitated as a stable stru pH of the electrolyte which could alter the reaction<br>
dynamics. Hence, a continuous flow of electrolyte<br>
dynamics. Hence, a continuous flow of electrolyte<br>
that the catalyst has a disposition will prevent this kind of pH namics. Hence, a continuous flow of electrolyte lar and show symmetric, then it could be considered<br>ough reactior will prevent this kind of pH from that the catalyst has a stable stattere and otes so to<br>maging during the through reactor will prevent this kind of pH from<br>
charging during the reaction even at longer rm. And undergo systemetral transformation, and the role of<br>
elate sume time, also enough amount of clectrolyce<br>
at the sume t changing during the reaction even at longer run. And undergo any structural transformation, and the role of<br>the same time, also enongh amount of electrolyte the chemical state change of Fe species could also tenus the pre at the same time, also enough amount of electrolyte<br>
the chemical state change of Fe species could also be<br>
must be presented inside the reaction cell during the<br>
must be presented inside the catalyst working conditions.<br>

 $\frac{d}{dx}$   $\text{tL}(k\#(J. \text{Electrochem.}) 2022, 28(3), 2108541 (18 of 31)$ <br>
statalyst is needed for one OER test, but for the *in*-<br>
situ/operando <sup>57</sup>Fe Mössbauer studies enough Fe con-<br>
is any effect we should consider during analysis and<br>
ten  $\#E \neq (L \n{Electrochem.})$  2022, 28(3), 2108541 (18 of 31)<br>
catalyst is needed for one OER test, but for the *in*-<br>
situdoperando <sup>5</sup>Fe Mössbauer studies enough Fe con-<br>
is any effect we should consider during analysis and<br>
tent  $\frac{dE}{dt}E^{\text{in}}_{\text{int}}(J. \text{ *Electrochem.*}) 2022, 28(3), 2108541 (18 of 31)$ <br>
catalyst is needed for one OER test, but for the *in*——strate to study the effect of substrate because if there<br> *situloperando* <sup>5</sup>Fe Mössbauer studies  $\frac{4}{5}$  and  $\frac{4}{5}$  (*Electrochem.*) 2022, 28(3), 2108541 (18 of 31)<br>
catalyst is needed for one OER test, but for the *in*-strate to study the effect of substrate because if there<br> *situloperando* <sup>*s*</sup>Fe Mössbauer s  $\pm \frac{\text{4}E}{\text{4}C}$  Electrochem.) 2022, 28(3), 2108541 (18 of 31)<br>
ceatalyst is needed for one OER tests, but for the in-<br>
situdoperando <sup>57</sup>Fe Mössbauer studies enough Fe con-<br>
is any effect of substrate because if ther **EVALUATION**<br> **EXECUTE:** Electrochem.) 2022, 28(3), 2108541 (18 of 31)<br> **CELLA** and the istudioperando <sup>5</sup>Fe Missbauer studies enough Fe con-<br>
is any effect we should consider during analysis and<br>
tent in the catalyst is  $\pm \frac{R \pm \epsilon}{L}$  *Leternochem.*) 2022, 28(3), 2108541 (18 of 31)<br>
catalyst is needed for one OER test, but for the *in*-strate to study the effect of substrate because if there<br> *situdoperando* <sup>2</sup>Fe Mössbauer studies enou  $\pm$  the  $\frac{1}{2}$  and the mathemole of the content of the content of the content of the sympable particle of the simulated of the simulated of the simulated of the simulated of the content of the content of the content o **Example 11**<br> **Example 12**<br> **Example 12** the  $\frac{4}{5}$  corrected to the method space of the signal controller in the space of the simulated to detect signal controller in the catalyst is predict in the catalyst is reded for one OER test, but for the in-<br>strate t **EVALUATION**<br> **ENET ALTERT (ACTES AND ASSAMPTED ASSAMPTED** (18 of 31)<br> **ENET ASSAMPTED CONTROL CONTRO** the  $\frac{d_1}{d_2}$  the *Le Hermohem*,) 2022, 28(3), 2108541 (18 of 31)<br>
catalyst is needed for one OFR test, but for the *in*-<br>
situdoperando <sup>9</sup>Fe Mössbauer studies enough  $\bf{F}$  con-<br>
is any effect we should consider du  $\frac{1}{2}E\frac{2\pi}{4}L E$   $Elecmcheen.)$  2022, 28(3), 2108541 (18 of 31)<br>
catalyst is necded for one OER test, but for the *in*-strate to study the effect of substrate because if there<br> *stitutioperundo*<sup>5</sup> Fe Mössbauer studies eno **i EVACUAL EXECTION TENDEV**<br> **EXECUTE: EXECTION ACTES THE CONDUM** THE CONDIDENTIFY THE CONDIDENTIFY THE CONDIDENTIFY THE CONDIDENTIFY THE CONDIDENTIFY THE CONDIDENTIFY THE CONDUCT IS USED THE USED USED THE USED USED T callalyst is needed for one OER test, but for the in-<br>statebog callalyst is needed for one OER test, but for the in-<br>state to state to state of substrate because if there<br>statebog electors due and the catalyst is required catalyst is needed for one OFR test, but for the *in*-<br>strate to study the effect of substrate because if there<br>the study operator of  $\frac{1}{2}$  for the study operator studies enough  $F \in$  con-<br>is any effect or entired we  $\sin\mu\phi_P$  rando <sup>5</sup>Te Müssbauer studies enough Fe con-<br>
is any effect we should consider during analysis<br>
signals. Depositing in the catalyst is required to detect good quality<br>
fitting process to normalize. After pre-OER it in the catalyst is required to detect good quality<br>
mathematics. After pre-OER tests, first<br>
mathematics length in a to thrick catalyst layer on one<br>
in-sidiogrearial (OCP) to study the cifect of clear<br>
exhige dectrode signals. Depositing a too thick catalyst layer on one in-sint/operando test should be conducted at open circulation is not recommended as only sur-<br>contring electroles is not recommended as only sur-<br>caline particles will working electrode is not recommended as only sur-<br>
involucing (OCP) to study the effect of electrolyte.<br>
frace particles will the part in OER while the bottom<br>
invally, the absorption of y-range or strength of the<br>
frace fiace particles will take part in OER while the bottom Usually, the absorption of y-rays or strength of the principles may not take particles of signals could be decreased in the presence of electrofore, to make it sure th particles may not take part, hence signals could be signals could be decreased in the presence of electrolytical mixture of unreducted and reacted Fe in OER catalyst in the profile could consider a could alter that  $\epsilon$  i mixture of unreacted and reacted Fe in OER catalyst.<br>
They the this problem could be lessened if experi-<br>
Therefore, to reake it sure that every particle of the ment continues for longer time. Next, one test should<br>
cataly Therefore, to make it sure that every particle of the ment continues for longer time. Next, one test should<br>catalyst is pixcinated to detect signals, we need sover-<br>endatored at an applied potential lower than the<br>catalys catalyst is taking part in OFR, and enough amount of<br>early the conducted at an applied potential lower than the<br>catalyst is presented to detect signals, we need sever-<br>potential required for OER, i.e., lower than the onse enalyst is presented to detect signals, we need sever-<br>
potential required for OFR, i.e., lower than the onset<br>
al plectes of well-prepared working electrodes placed and potential. This will show whether there is any chan al pieces of well-prepared working electrodes placed<br>potential. This will show whether there is any change<br>pieces of electrodes deposited with the same catalyst tais or not. Alter these initial experiments, now we<br>pieces o parallel to each other as shown in Figure 11(B)  $(3-5$  of electrochemical state of Fe species at lower potenties<br>picces of electrochas deposited with he same catalyst tails or not. After tense initial experiments, now we<br> pieces of electrodes deposited with the same eatalyst<br>
ink placed at the distance of less than 1 mm). This can go for onset potentials and higher potentials<br>
ink placed at the distance of less than 1 mm). This can go for o ink placed at the distance of less than 1 mm). This can go for onset potentials and higher potentials. Af-<br>will fielilitate cocyparied of the cathlyst to take the retudying at different potentials, one should test the<br>par will firelihate every particle of the eatalyst to take<br>part in the OFR reaction and enousdy anomat of Fe post-OER inmediately after taking out the electrolyte<br>point in the OER reaction and enousd for a point of reaction s 28(3), 2108541 (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situ/operando test should 28(3), 2108541 (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situ/operando test should 28(3), 2108541 (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br> $in-situ/operando$  test should be cond 28(3), 2108541 (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situ/operando test should 28(3), 2108541 (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situ/operando test should 28(3), 2108541 (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situloperando test should 28(3), 2108541 (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>*in-situloperando* test shou  $28(3)$ ,  $2108541$  (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situ/operando test sh  $28(3)$ ,  $2108541$  (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>*in-situloperando* test 28(3), 2108541 (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situ/operando test should 28(3), 2108541 (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situ/operando test should 28(3), 2108541 (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>*in-situloperando* test shou  $28(3)$ ,  $2108541 (18 of 31)$ <br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situ/operando test shoul 28(3), 2108541 (18 of 31)<br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>*in-situloperando* test shou  $28(3)$ ,  $2108541 (18 of 31)$ <br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situ/operando test shoul  $26(3), 2106341 (18.0131)$ <br>strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>*in-situloperando* test shoul strate to study the effect of substrate because if there<br>is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situ/operando test should be conducted at open cir-<br> is any effect we should consider during analysis and<br>fitting process to normalize. After pre-OER tests, first<br>in-situ/operando test should be conducted at open cir-<br>cuit potential (OCP) to study the effect of electrolyte. fitting process to normalize. After pre-OER tests, first in-situ/operando test should be conducted at open circuit potential (OCP) to study the effect of electrolyte. Usually, the absorption of  $\gamma$ -rays or strength of th in-situloperando test should be conducted at open circuit potential (OCP) to study the effect of electrolyte.<br>Usually, the absorption of  $\gamma$ -rays or strength of the signals could be decreased in the presence of electroly cuit potential (OCP) to study the effect of electrolyte.<br>Usually, the absorption of  $\gamma$ -rays or strength of the signals could be decreased in the presence of electrolyte, but this problem could be lessened if experiment c Usually, the absorption of  $\gamma$ -rays or strength of the signals could be decreased in the presence of electrolyte, but this problem could be lessened if experiment continues for longer time. Next, one test should be conduc signals could be decreased in the presence of electrolyte, but this problem could be lessened if experiment continues for longer time. Next, one test should be conducted at an applied potential lower than the potential req trolyte, but this problem could be lessened if experi-<br>ment continues for longer time. Next, one test should<br>be conducted at an applied potential lower than the<br>potential required for OER, i.e., lower than the onset<br>potent ment continues for longer time. Next, one test should<br>be conducted at an applied potential lower than the<br>potential required for OER, i.e., lower than the onset<br>potential. This will show whether there is any change<br>of elec be conducted at an applied potential lower than the<br>potential required for OER, i.e., lower than the onset<br>potential. This will show whether there is any change<br>of electrochemical state of Fe species at lower poten-<br>tials potential required for OER, i.e., lower than the onset<br>potential. This will show whether there is any change<br>of electrochemical state of Fe species at lower poten-<br>tials or not. After these initial experiments, now we<br>can potential. This will show whether there is any change<br>of electrochemical state of Fe species at lower poten-<br>tials or not. After these initial experiments, now we<br>can go for onset potentials and higher potentials. Af-<br>ter of electrochemical state of Fe species at lower potentials or not. After these initial experiments, now we can go for onset potentials and higher potentials. After studying at different potentials, one should test the post tials or not. After these initial experiments, now we<br>can go for onset potentials and higher potentials. Af-<br>ter studying at different potentials, one should test the<br>post-OER immediately after taking out the electrolyte<br> can go for onset potentials and higher potentials. Af-<br>ter studying at different potentials, one should test the<br>post-OER immediately after taking out the electrolyte<br>and removal of applied potentials, and then after 48<br>h ter studying at different potentials, one should test the post-OER immediately after taking out the electrolyte and removal of applied potentials, and then after 48 hours to 72 hours when the electrode is fully dried. Thi post-OER immediately after taking out the electrolyte<br>and removal of applied potentials, and then after 48<br>hours to 72 hours when the electrode is fully dried.<br>This will give a clear understanding about the struc-<br>ture st rs to 72 hours when the electrode is fully dried.<br>
so will give a clear understanding about the structions and three reading about the structions and structure capability of the catalyst. If the OER and post-OER spectra o This will give a clear understanding about the structure stability and time required for recovering the original structure capability of the catalyst. If the pre-OER spectra of a catalyst are similar and show symmetric, t riginal structure capability of the catalyst. If the<br>pre-OER and post-OER spectra of a catalyst are simi-<br>lar and show symmetric, then it could be considered<br>that the catalyst has a stable structure and does not<br>undergo a pre-OER and post-OER spectra of a catalyst are similar and show symmetric, then it could be considered that the catalyst has a stable structure and does not undergo any structural transformation, and the role of the chemi lar and show symmetric, then it could be considered<br>that the catalyst has a stable structure and does not<br>undergo any structural transformation, and the role of<br>the chemical state change of Fe species could also be<br>confir that the catalyst has a stable structure and does not<br>undergo any structural transformation, and the role of<br>the chemical state change of Fe species could also be<br>confirmed under OER catalyst working conditions.<br>One most undergo any structural transformation, and the role of<br>the chemical state change of Fe species could also be<br>confirmed under OER catalyst working conditions.<br>One most important point here is that the current<br>density must 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (18 of 31)<br>
R test, but for the *in*-<br>
strate to study the effect of substrate because if there<br>
udies enough Fe con-<br>
is any effect we should consider during analysis and<br>
to

# 6 In-Situ/Operando  $57Fe$  Mössbauer

the chemical state change of Fe species could also be confirmed under OER catalyst working conditions.<br>One most important point here is that the current density must be stable during whole reaction at every applied potent

 $\#E\# (J. \text{Electrochem.}) 2022, 28(3), 2108541 (19 of 31)$ <br>formed to NiOOH<sub>2-x</sub> during OER, which generate Ni<sup>4+</sup> stability issue of NiFe-OEC was investigated and the<br>hence the OER activity was improved. These contra-<br>dictory and inc hence the OER activity was improved. These contra- $\text{d}t/\mathcal{C}^*$  (*J. Electrochem.*) 2022, 28(3), 2108541 (19 of 31)<br>
formed to NiOOH<sub>2x</sub> during OER, which generate Ni<sup>4+</sup> stability issue of NiFe-OEC was investigated and the<br>
hence the OER activity was improved. These  $\frac{dE}{dt}$   $\frac{f(k\cdot\frac{2\pi}{3})}{\pi}$ <br>
formed to NiOOH<sub>2</sub><sub>2</sub> during OER, which generate Ni<sup>+</sup><br>
stability issue of NiFe-OEC was investigated and the<br>
hence the OER activity was improved. These contra-<br>
real phase of the catalyst was conf  $\frac{4E}{2}$  (*J. Electrochem..*) 2022, 28(3), 2108541 (19 of 31)<br>formed to NiOOH<sub>2</sub>, during OER, which generate Ni<sup>4+</sup> stability issue of NiFe-OEC was investigated and the<br>hence the OER activity was imposed. These contra-<br>

of NiFe-OECs is responsible for higher OER activity. pared with that of NiFe@C obtained from highter<br>of Therefore, we tried to solve both these challenging perature pyrolization of NiFe-PBAs. The NiFe-issues and address w Therefore, we tried to solve both these challenging perature pyrolization of NiFe-PBAs. The NiFe-PBAs'<br>
issues and address well in our following suchces.<br>
CNTS OBR activity with the optimum annount of<br>
We have so far disc issues and address well in our following studies.<br>
CNTs OFR activity with the optimum amount of<br>
We have so far dissuesed the effects of doped<sup>7</sup>in-<br>
socyts of CNTs was found to be significantly superi-<br>
socyts of CNTs wa We have so far discussed the effects of doped/in-<br>
Sur,<sup>9</sup>% of CNTs was found to be significantly superi-<br>
corporated Fe in Ni-based OECs, their activity. But it is Tafel slope showed the smallest value of 83 mV dec<sup>-</sup><br>
i corporated Fe in Ni-based OECs, their activities, pos-<br>
sub-factors responsible for higher activities, pos-<br>
sub-factors responsible for higher activity. But it is Tarisl slope showed the smallest value of 88 mV  $\text{deg}\cdot\text$ sible factors responsible for higher activity. But it is<br>
imperiate showed the smallest value of 83 mV-dec<sup>3</sup><br>
imperiate to highlight the suitable meltod for their among other samples as shown in Figure [210). The<br>
synthe important to highlight the suitable method for their<br>sympless as shown in Figure 12(B). The<br>symphosis and *in-sinius/operando* studies. We here rece-<br>Symphoffraction (XRD) results before and after de-<br>ommend a suitable an synthesis and *in*-situ/operando studies. We here rec-<br>
Dommend a surishe and fine in enchancement as with the synthesis position and entired in The electrophycPlance and fine in the synthesis position and entired in the ommend a suitable and facile method for the synthesis position and activation by CV of NiFe-PBA/CNTs of NiFe-(wyphydroxica) CBCs by using PBAs as pre-<br>
environg met materials and the crystal process are standed that the c of NiFe-(oxy)hydroxide OECs by using PBAs as pre-<br>
ucusors, which was newly developed in our lab<sup>ton</sup>. We structure was changed after eatition is abown in<br>
know that PBAs have the typical chemical formula<br>
Eignic 12(C). T cursors, which was newly developed in our lab<sup>ton</sup>. We<br>structure was changed after activation as shown in<br>Know that PBAs have the typical chemical formula<br> $F$ Eigure 12(C). The XRD peaks interstities vore signif-<br>KnoW[M/m( know that PBAs have the typical chemical formula<br>
Figure 12(C). The XRD peaks intensities were signif-<br>
A<sub>D</sub>M<sub>M</sub>(M<sub>M</sub>C(N)<sub>b</sub>)<sup>-1</sup>x14O, where A indicates the alkili cannot betweed atter attivation, which suggests that<br>
met  $A/M_n[Mm(CN)_0] \cdot xH_2O$ , where A indicates the alkali icantly reduced after activation, which suggests that metal ions such as Na' and K' etc., while M dm of the crystal structures of Nire-BIAs sures horked during represent t metal ions such as Na<sup>2</sup> and K<sup>2</sup> etc., while M and M<br>
the crystal structure of NiFe-PBAs was broken during<br>
represent the transition metal cations<sup>8,690</sup>. In the PBAs the acirtation process. The characteristic peaks repr represent the transition metal cations<sup>(66,69</sup>). In the PBAs the activation process. The characteristic peaks represent after the transition metal ions ( $M^2$ -C=N-M<sup>o</sup>). Due to experige Spectra and the difference in elect erystal structures, eyanide groups serve as bridges be-<br>
serving NiFe-PBAs completely disappeared after 125<br>
tween transition media long ( $W^2 - C = N - N^1$ ). Due to eyeles, indecting that the NiFe-PBA structure had<br>
the diffe application.  $A_1M_n[\dot{M}_m(CN)_6] \cdot xH_2O$ , where A indicates the alkali corresponding M and M metals show high and low

# Electrocatalysts

 $4R^2(L \text{ Electrowehem.}) 2022, 28(3), 2108541 (19 of 31)$ <br>formed to NiOOH<sub>2</sub><sub>2</sub> during OER, which generate Ni<sup>\*</sup> stability issue of NiFe-OEC was investigated and the<br>hence the OER activity was improved. These contra-<br>real phase of the  $\pm$  *Wetter (J. Electrochem.*) 2022, 28(3), 2108541 (19 of 31)<br>formed to NiOOH<sub>2</sub>, during OER, which generate Ni<sup>+1</sup> stability issue of NiFe-OEC was investigated and the<br>hence the OER activity was improved. These contra- $\pm \frac{1}{2}$  ( $\pm \frac{1}{2}$  and NiOOHL<sub>3</sub>, during OER, which generate Ni<sup>\*</sup> stability issue of NiFe-OEC was investigated and the hence the OER activity was improved. These contra-<br>
real phase of the catalyst was confirmed du the Sigman model of NiOOH<sub>2</sub>, during OER, which generate Ni<sup>44</sup> stability issue of NiFe-OEC was investigated and the hence the OFR activity was improved. These contra-<br>nearly these of NiFe-OEC was investigated and the hen  $\frac{1}{2}$   $\frac{1}{2}$  **FE in Ni-Based Acts** ( $E/K$  ( $E/K$  acts ( $E/K$  acts ( $E/K$ )) and  $E/K$  ( $E/K$ ) and  $E/K$ ) and  $E/K$  ( $E/K$  ( $E/K$ ) and  $E/K$  ( $E/K$ ) and **EVALUATION THE FACT CONTIC CONTIC SUPPORT (19 (19 THE SUPPORT) (19 THE SUPPORT ACT (19 THE SUPPORT ACT THE SUPPORT ACT THE SUPPORT ACT THE SUPPORT ACT THE SUPPORT (19 THE SUPPORT ACT THE SUPPORT ACT THE SUPPORT ACT THE S**  $\frac{16}{22}(L\text{E}/$ . Electrosheem.) 2022, 28(3), 2108541 (19 of 31)<br>
formed to NiOOH<sub>2</sub>, during OER, which generate Ni<sup>+</sup> stability issue of NiFe-OEC was investigated and the<br>
between the OER activity was improved. These c the  $2^x (L. R/2e$  and the  
three the OKIOOH<sub>2</sub>, during OFR, which generate Ni<sup>+</sup>  
subility (size of NFE-GDFC was investigated and the  
hence the OER activity was improved. These contra-  
real phase of the catalyst was confirmed during OER  
diatory and incomplete claims indicated two major  
before going for *in-siuuperanab* Massbauer study to  
the clearly understood and explained and 2) the  
this purpose, composites of NiFe-PBAs/carbon nano-  
reversible structural transformation of NiFe-OBCs  
in the OER, making it unclear that which phase were synthesized and the  
inotropic  
of NiFe-OBCs is responsible for higher OFR activity.   
one  
of NiFe-OBCs is responsible for higher OFR activity**From the NiOOHL<sub>3</sub>, during OER, which generative surespects to the state of SF-OEC was investigated and the hence the OFR activity was improved. These contra-<br>heliots and facile method for the synthesis of NiFe-OEC was i** formed to NiOOH<sub>2</sub>, during OER, which generate Ni<sup>41</sup> stability issue of NiFe-OEC was investigated and the hoffs activity was improved. These contra-<br>
real phase of the castalyst was confirmed during OER<br>
herects be distr hence the OER activity was improved. These contra-<br>electrons and incomplete calimits indicated two major before going for *in-sinulogremalo* Missbaucar study to<br>draws in these studies: 1) the real role of Fe<sup>4</sup> could furt dictory and incomplete claims indicated two major<br>
brakes was in the serval is: 1) the real root of Fe<sup>-</sup> could further elucidat and understand the mechanism. For<br>
flaxes in thes studies: 1) the real root of the chemical flaws in these studies: 1) the real role of Fe<sup>t</sup>' could<br>
further clucidate and understand the mechanism. For<br>
not be clearly understood and explained and 2) the this purpose, composites of NiFe-OBA<br>
orders are therefore not be clearly understood and explained and 2) the<br>
this purpose, composites of NiFe-PBAs<br>
reversible structural transformation of NiFe-OECs<br>
tubes (CNTs) with the optimized am<br>
during the OER, making it urelare that whic of and explained and 2) the this purpose, composites of NiFe-PBAs/carbon rnsformation of NiFe-OECs tubes (CNTs) with the optimized amount of  $\zeta$  it unclear that which phase were synthesized and their OER activities were explained and 2) the this purpose, composites of NiFe-PBAs/carbon nano-<br>tion of NiFe-OECs tubes (CNTs) with the optimized amount of CNTs<br>respectively. where synthesized and their OER activities were com-<br>higher OER activi reversible structural transformation of NiFe-OECs<br>
urbese (CNTs) with the optimized amount of CNTs<br>
during the OFR, making it unclear that which phase<br>
or NiFe-OECs is responsible for higher OER activity. Pared with that during the OER, making it unclear that which phase<br>
or Syruence synthesized and their OFR activities were com-<br>
or NiFe-OECs is responsible for higher OFR activity . pared with that of NiFe-@C obtained from high-tem-<br>
The higher OER activity. pared with that of NiFe@C obtained from high-tem-<br>
th these challenging perature pyrolization of NiFe-PBAs. The NiFe-PBAs/<br>
CNITs OER activity, with the optimum amount of<br>
effects of doped/in8wt.% o 28(3), 2108541 (19 of 31)<br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situ/operando* Mössbauer study to<br>further elucidate and understand the 28(3), 2108541 (19 of 31)<br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand the  $28(3)$ ,  $2108541$  (19 of 31)<br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situ/operando* Mössbauer study to<br>further elucidate and understan  $28(3)$ ,  $2108541 (19 of 31)$ <br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for  $in-situ/operando$  Mössbauer study to<br>further elucidate and understand the mechani 28(3), 2108541 (19 of 31)<br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand the  $28(3)$ ,  $2108541$  (19 of 31)<br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understan  $28(3)$ ,  $2108541 (19 of 31)$ <br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand t  $28(3)$ ,  $2108541 (19 of 31)$ <br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situ/operando* Mössbauer study to<br>further elucidate and understand t  $28(3)$ ,  $2108541 (19 of 31)$ <br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situ/operando* Mössbauer study to<br>further elucidate and understand t 28(3), 2108541 (19 of 31)<br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand th 28(3), 2108541 (19 of 31)<br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situ/operando* Mössbauer study to<br>further elucidate and understand th 28(3), 2108541 (19 of 31)<br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand th 28(3), 2108541 (19 of 31)<br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand th 28(3), 2108541 (19 of 31)<br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand th 28(3), 2108541 (19 of 31)<br>stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand th **Example 12** (B) is sure of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand the mechanism. For<br> stability issue of NiFe-OEC was investigated and the<br>real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand the mechanism. For<br>this purp real phase of the catalyst was confirmed during OER<br>before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand the mechanism. For<br>this purpose, composites of NiFe-PBAs/carbon nano-<br>tubes (CNTs) before going for *in-situloperando* Mössbauer study to<br>further elucidate and understand the mechanism. For<br>this purpose, composites of NiFe-PBAs/carbon nano-<br>tubes (CNTs) with the optimized amount of CNTs<br>were synthesized further elucidate and understand the mechanism. For<br>this purpose, composites of NiFe-PBAs/carbon nano-<br>tubes (CNTs) with the optimized amount of CNTs<br>were synthesized and their OER activities were com-<br>pared with that of this purpose, composites of NiFe-PBAs/carbon nanotubes (CNTs) with the optimized amount of CNTs were synthesized and their OER activities were com-<br>pared with that of NiFe@C obtained from high-tem-<br>perature pyrolization of tubes (CNTs) with the optimized amount of CNTs<br>were synthesized and their OER activities were com-<br>pared with that of NiFe@C obtained from high-tem-<br>perature pyrolization of NiFe-PBAs. The NiFe-PBAs/<br>CNTs OER activity with were synthesized and their OER activities were com-<br>pared with that of NiFe@C obtained from high-tem-<br>perature pyrolization of NiFe-PBAs. The NiFe-PBAs/<br>CNTs OER activity with the optimum amount of<br>8wt.% of CNTs was found pared with that of NiFe@C obtained from high-tem-<br>perature pyrolization of NiFe-PBAs. The NiFe-PBAs/<br>CNTs OER activity with the optimum amount of<br>8wt.% of CNTs was found to be significantly superi-<br>or to NiFe@C as shown i perature pyrolization of NiFe-PBAs. The NiFe-PBAs/<br>CNTs OER activity with the optimum amount of<br>8wt.% of CNTs was found to be significantly superi-<br>or to NiFe@C as shown in Figure 12(A), and the<br>Tafel slope showed the sma TTs OER activity with the optimum amount of  $t.*$  of CNTs was found to be significantly superito NiFe@C as shown in Figure 12(A), and the fel slope showed the smallest value of 83 mV·dec<sup>-1</sup> to NiFe@C as shown in Figure 12 8wt.% of CNTs was found to be significantly superior to NiFe@C as shown in Figure 12(A), and the Tafel slope showed the smallest value of 83 mV·dec<sup>-1</sup> among other samples as shown in Figure 12(B). The X-ray diffraction ( or to NiFe@C as shown in Figure 12(A), and the<br>Tafel slope showed the smallest value of 83 mV·dec<sup>-1</sup><br>among other samples as shown in Figure 12(B). The<br>X-ray diffraction (XRD) results before and after de-<br>position and act Tafel slope showed the smallest value of 83 mV  $\cdot$  dec<sup>-1</sup><br>among other samples as shown in Figure 12(B). The<br>X-ray diffraction (XRD) results before and after de-<br>position and activation by CV of NiFe-PBA/CNTs<br>(8wt.%) on among other samples as shown in Figure 12(B). The<br>X-ray diffraction (XRD) results before and after de-<br>position and activation by CV of NiFe-PBA/CNTs<br>(8wt.%) on carbon paper indicated that the crystal<br>structure was change X-ray diffraction (XRD) results before and after de-<br>position and activation by CV of NiFe-PBA/CNTs<br>(8wt.%) on carbon paper indicated that the crystal<br>structure was changed after activation as shown in<br>Figure 12(C). The X metal ions such as Na<sup>+</sup> and K<sup>+</sup> etc., while M and M the crystal structure of NiFe-PBAs was broken during 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (19 of 31)<br>
R, which generate Ni<sup>4+</sup> stability issue of NiFe-OEC was investigated and the<br>
proved. These contra-<br>
indicated two major before going for *in-situ/operando* Mössba

the difference in electronegativities of C and N, the been completely transformed into other structures.<br>
spin states, respectively, These materials are useful to 125 cycles, which and low<br>
for different applications such n states, respectively. These materials are useful<br>
to 125 cycles, which suggests that there is no further<br>
different applications solid as batteries, gas store, change in crystal structure after 125 cycles. Further-<br>
dif position and activation by CV of NiFe-PBA/CNTs (8wt.%) on carbon paper indicated that the crystal structure was changed after activation as shown in Figure 12(C). The XRD peaks intensities were significantly reduced after (8wt.%) on carbon paper indicated that the crystal<br>structure was changed after activation as shown in<br>Figure 12(C). The XRD peaks intensities were signif-<br>icantly reduced after activation, which suggests that<br>the crystal structure was changed after activation as shown in<br>Figure 12(C). The XRD peaks intensities were signif-<br>icantly reduced after activation, which suggests that<br>the crystal structure of NiFe-PBAs was broken during<br>the activa Figure 12(C). The XRD peaks intensities were significantly reduced after activation, which suggests that the crystal structure of NiFe-PBAs was broken during the activation process. The characteristic peaks representing N icantly reduced after activation, which suggests that<br>the crystal structure of NiFe-PBAs was broken during<br>the activation process. The characteristic peaks repre-<br>senting NiFe-PBAs completely disappeared after 125<br>cycles, the crystal structure of NiFe-PBAs was broken during<br>the activation process. The characteristic peaks repre-<br>senting NiFe-PBAs completely disappeared after 125<br>cycles, indicating that the NiFe-PBA structure had<br>been compl the activation process. The characteristic peaks representing NiFe-PBAs completely disappeared after 125 cycles, indicating that the NiFe-PBA structure had been completely transformed into other structures. The XRD result senting NiFe-PBAs completely disappeared after 125<br>eycles, indicating that the NiFe-PBA structure had<br>been completely transformed into other structures.<br>The XRD results after the 500 cycles were similar<br>to 125 cycles, whi eycles, indicating that the NiFe-PBA structure had<br>been completely transformed into other structures.<br>The XRD results after the 500 cycles were similar<br>to 125 cycles, which suggests that there is no further<br>change in crys been completely transformed into other structures.<br>
The XRD results after the 500 cycles were similar<br>
to 125 cycles, which suggests that there is no further<br>
change in crystal structure after 125 cycles. Further-<br>
more, The XRD results after the 500 cycles were similar<br>to 125 cycles, which suggests that there is no further<br>change in crystal structure after 125 cycles. Further-<br>more, *ex-situ* <sup>5</sup>Fe Mössbauer spectral measurement<br>shown in to 125 cycles, which suggests that there is no further<br>change in crystal structure after 125 cycles. Further-<br>more, *ex-situ* <sup>5</sup>Fe Mössbauer spectral measurement<br>shown in Figure 12(D) before and after activation of<br>NiFe-



**Examples and Source COX**<br>
From S<sup>1</sup>)<br>  $\frac{6.80 \text{ V}}{1000}$ <br>  $\frac{1500}{1000}$ <br>  $\frac{1500}{2000}$ <br>  $\frac{2500}{2000}$ <br>  $\frac{2500}{400}$ <br>  $\frac{450}{400}$ <br>  $\frac{550}{500}$ <br>  $\frac{1500}{1000}$ <br>  $\frac{1500}{2000}$ <br>  $\frac{2500}{400}$ <br>  $\frac{450}{400}$ In the first part of our case study above, we con-Figure 12 (mm s<sup>1</sup>)<br>
Figure 12 (A) Linear sweep voltammetric results of NiFe-PBAs/CNTs with varying amount of CNTs. (B) Tafel slopes for NiFe-PBAS/CNTs (C) C NEO among the sinuture of NiFe-PBAS/CNTs (C),  $D_{\text{ex}}$  can be **Figure 12** (A) Linear sweep voltammetric results of NiFe-PBAs/CNTs with varying amount of CNTs. (B) Tafel slopes for NiFe-PBAs/CNTs, (C) XRD analysis showing the structures of NiFe-PBAs/CNTs, carbon paper, NiFe-PBAs/CNTs

trochemical activation in the alkaline condition gen-<br>
to the atomic ratio of Fc/Ni) catalysts<br>
rerating Ni(OH)<sub>2</sub>/NiOOH. In this stage, this Ni(OH)<sub>2</sub>/ sized by the topotactic transformation<br>
NiOOH can be considered to t

strategy<sup>[71]</sup>. **Example 18.1**<br> **Example 18.0**<br> **Concerned by**<br> **Concerned by**<br> **Concerned by**<br> **Concerned by**<br> **Concerned by**<br> **Concerned and the novelties in our second part case study. (D)** *Ex-situ* **<sup>2</sup>Fe Mössbauer spectroscopic analy** 1500 2000 2500 460 4450 560 550 600<br>
hift (cm<sup>-1</sup>) Raman shift (cm<sup>-1</sup>)<br>
Raman shift (cm<sup></sup>

**FIGURE 12 CONSTRANTS (C) XRIS analysis showing the structures of NiFe-PIAACCNTs, cumben paper, Niic-PIAACCNTs vacuum control. And the report of the CHAME (NIFe-PIAACCNTs vacuum of the CHAME (NIFe-PIAACCNTs before and aft** Where  $\text{PAW}$  and the properties and the state of the production of the production of the production of the production (F) Remains pectometer for Nire-PBAseCNTs before and after CV activation. (E) Raman spectra for Nire-Nite-PHAeVCNTs before and after CV activution. (E) Raman spectra for Nite-PHAeVCNTs before and after CV activution. (F) *In-situal*<br>operando Raman spectroscopic analysis for NiFe-PHAs/CNTs at different applied potentials. sperson Raman spectroscopic analysis for NiFe-PBAsCNTs at different applied potentials. (color on line)<br>trochemical activation in the alkaline condition gen-<br>to the atomic ratio of Fe/Ni) catalysts were synthe-<br>erreductin trochemical activation in the alkaline condition gen-<br>
contemption From extraing Ni(OH)/NiOOH. In this stage, this Ni(OH)/sized by the topotactic transformation of NiFe<sub>G-</sub>Fe<br>
NiOOH can be considered to the real active ph trochemical activation in the alkaline condition gen-<br>
to the atomic ratio of Fe/Ni) catalysts were synthe-<br>
erating Ni(OH), Ni on this stage, this Ni(OH) $y$  sized by the topotactic transformation of NiFe<sub>x</sub>-Fe<br>
NiOOH can erating Ni(OH)<sub>N</sub>NiOOH. In this stage, this Ni(OH)y sized by the topotactic transformation of NiFe<sub>5</sub>-Fe NOMOH can be considered to the real active phase **properties** which is more considered to the real active phase whic NiOOH can be considered to the real active phase, PBAs in an alkaline solution. The NiFe<sub>m</sub>-Fe PBAs which is more reasonable to claim that why NiFe-PBAs materials with different iron contents doped in the has high activit series of NiFe<sub>n</sub>-(oxy)hydroxides (NiFe<sub>n</sub>-O<sub>x</sub>H<sub>1</sub>, m refers spectroscopy is so high that it could even differentiate Raman shift (cm<sup>-1</sup>)<br>
NTs with varying amount of CNTs. (B) Tafel slopes for NiFe-<br>
As/CNTs, carbon paper, NiFe-PBAs/CNTs/carbon paper, and<br>
mmetry. (D) *Ex-situ* <sup>37</sup>Fe Mössbauer spectroscopic analysis of<br>
for NiFe-PBAs/C Raman shift (cm<sup>-1</sup>)<br>
CNTs. (B) Tafel slopes for NiFe-<br>
IFe-PBAs/CNTs/carbon paper, and<br>
dissbauer spectroscopic analysis of<br>
and after CV activation. (F)  $In-situ/$ <br>
or on line)<br>
Fe/Ni) catalysts were synthe-<br>
transformatio NTs with varying amount of CNTs. (B) Tafel slopes for NiFe-<br>As/CNTs, carbon paper, NiFe-PBAs/CNTs/carbon paper, and<br>mmetry. (D) *Ex-situ* <sup>37</sup>Fe Mössbauer spectroscopic analysis of<br>for NiFe-PBAs/CNTs before and after CV a VIS with varying amount of CNTs. (B) Tatel slopes for NIFe-<br>As/CNTs, earbon paper, NiFe-PBAs/CNTs/cardon paper, and<br>mmetry. (D) *Ex*-situ <sup>3</sup>Fe Mossbauer spectroscopic analysis of<br>for NiFe-PBAs/CNTs before and after CV ac states in the system of the system of the system and the system of the system of the Mic-FBAs'CNTs before and after CV activation. (F)  $In-situl$ <br>pramety. (D) Ex-situ <sup>5</sup>Fe Missbauer spectroscopic analysis of<br>prent applied po Example 2.1 Section and after CV activation. (F) *In-situl*<br>for NiFe-PBAs/CNTs before and after CV activation. (F) *In-situl*<br>rent applied potentials. (color on line)<br>to the atomic ratio of Fe/Ni) catalysts were synthe-<br>s stream applied potentials. (color on line)<br>
to the atomic ratio of Fe/Ni) catalysts were synthe-<br>
sized by the topotactic transformation of NiFe<sub>ar</sub>Fe<br>
PBAs in an alkaline solution. The NiFe<sub>a</sub>Fe PBAs<br>
materials with diff to the atomic ratio of Fe/Ni) catalysts were synthe-<br>sized by the topotactic transformation of NiFe<sub>*m*</sub>-Fe<br>PBAs in an alkaline solution. The NiFe<sub>*m*</sub>-Fe</sub> PBAs<br>materials with different iron contents doped in the Ni<br>sites to the atomic ratio of Fe/Ni) catalysts were synthe-<br>sized by the topotactic transformation of NiFe<sub>ar</sub>-Fe<br>PBAs in an alkaline solution. The NiFe<sub>ar</sub>-Fe<br>PBAs<br>materials with different iron contents doped in the Ni<br>sites we to the atomic ratio of Fe/Ni) catalysts were synthe-<br>sized by the topotactic transformation of NiFe<sub>*m*</sub>-Fe<br>PBAs in an alkaline solution. The NiFe<sub>m</sub>-Fe<br>PBAs materials with different iron contents doped in the Ni<br>sites we sized by the topotactic transformation of NiFe<sub>*m*</sub>-Fe</sub><br>PBAs in an alkaline solution. The NiFe<sub>m</sub>Fe PBAs<br>materials with different iron contents doped in the Ni<br>sites were prepared by our previously developed<br>strategy<sup>*rm*</sup> PBAs in an alkaline solution. The NiFe<sub>m</sub>Fe PBAs<br>materials with different iron contents doped in the Ni<br>sites were prepared by our previously developed<br>strategy<sup>*Fu*1</sup>.<br>One of the novelties in our second part case study<br>h



 $\frac{1}{2}$   $\frac{1}{2}$ 

The spin Fe3+ was observed in the Nisibality of High spin Fe3+ was observed in ex-situ and in-stitute per spin spin Fe3+ was observed in the Spin Fe3+ was observed in the Spin Fe3+ was observed in the Spin Fe3+ was obse The sum of the set of the control of the control of the control of the set of **Example 10**<br> **Example 10** s a continue of Nice and Parameteria shape and parameterial and parameterial shape and parameters as shown in Figure 13 (A-D) *Fession* "Fe Missbauer analysis of Nire<sub>c</sub>-Fe PBAs by topolarities trains of Nirec. (E-H) *Ex-*Figure 13 (A-D)  $E_{\text{ex}}$ /mans y<br>
Figure 13 (A-D)  $E_{\text{ex}}$ -inn <sup>2</sup>Fe Mossbauer analysis of NiFe<sub>a</sub>-Fe PBAs doped with different ratios of NiFe. (E-H)  $E_{\text{ex}}$ -stin<sup>2</sup>Fe Nossbauer analysis of NiFe<sub>a</sub>-O<sub>L</sub>H, derived from Examples and  ${}^{2}$ Figure 13(A-D) *Ex*-sita.<sup>3</sup>Figure 14(A) shows that the NiFeq<sub>x</sub>-O<sub>t</sub>H, exhibited the lowest operators in the precursor NiFe, Fe PBAs doped with different ratios of NiFe, (E-H) *Ex*-sita.<sup>33</sup>Fe Missonar Figure 14(A) shows that the NiFe<sub>02</sub>-O<sub>x</sub>-O<sub>x</sub>-O<sub>x</sub>-C<sub>1</sub>H<sub>v</sub> exhibited lysts shown in Figure 14(F-G) indicate that NiFe<sub>02</sub>-O<sub>x</sub>buser analysis of Nite<sub>x</sub>-O,14, derived from the precursors Nite<sub>x</sub><sup>1</sup>e PHAs by topolactic transformation, (color on line)<br>between two different crystallographic positions of gen coordinated octahedral in both crystal pha between two different crystallographic positions of<br>
Fe<sup>11</sup> which is usually not possible with other charac-<br>
Fe<sup>21</sup> which is usually not possible with other charac-<br>
terization techniques. This makes <sup>27</sup>Fe Mössbauer val between two different erystallographic positions of<br>
gen coordinated ottahedral in both crystal phases of<br>
Fei<sup>2</sup> which is usually not possible with other charec-<br>
erization its mailers. This makes <sup>sp</sup>ie Mössbauer value Fe<sup>11</sup> which is usually not possible with other charac-<br>
Fe<sup>13</sup> which smaller  $\Delta$ <br>
etrization techniques. This makes <sup>3</sup>Fe Missbauer value indicating the occhical symetry should be<br>
espectroscopy more sophisticated and s terization techniques. This makes <sup>37</sup>Fe Mössbauer value indicating the octahedral symmetry should be<br>spectroscopy more sophisticated an sensitive to higher in later one than the former one. This also fur-<br>study Fe contai spectroscopy more sophisticated and sensitive to higher in later one than the former one. This also fur-<br>study Fe containing materials. Moreover, increasing the illustrates the high energy resolution of the "Fe<br>the Fe rat study Fe containing materials. Moreover, increasing ther illustrates the high energy resolution of the <sup>97</sup>Fe<br>which, later on, will be transformed into Nife<sub>ia</sub>, O, M, the changes in the electron chinique where even sub-<br> the Fe ratio increases the Fe<sup>\*</sup> amount in the Ni sites,<br>
whisehauer spectroscopic technique where even sub-<br>
which, later on, will be transformed into NiFeg., O<sub>1</sub>H, the changes in the electron density and the electric<br>  $\frac{1}{\sqrt{2}}$ <br>  $\frac{1}{\sqrt{2}}$ G 100<br>  $\sum_{\substack{99 \text{ s}}^{90} \text{ s}}$ <br>  $\sum_{\substack{99 \text{ s}}^{90} \text{ s}}$ <br>  $\sum_{\substack{99 \text{ s}}^{90} \text{ s}}$ <br>  $\sum_{\substack{10 \text{ m/s} \cdot 10}}^{90}$ <br>  $\sum_{\substack{10 \text{ m/s} \$ Figure 2011 and  $\frac{1}{2}$  and  $\frac{1}{2}$ Examples and the similar similar tries in pectra with the simple example and in-situ ioperando Raman spectra with the similar trend was also observed at the similar trend was also observed with different ratios of Ni:Fe. Example 1<br>
by a subset of the situal specific and the situation of Ni-Fe. (E-H) Ex-situ <sup>5</sup>Fe Möss-<br>
BAs by topotactic transformation. (color on line)<br>
gen coordinated octahedral in both crystal phases of<br>  $\alpha$ -phase Ni(O **PERTUBE SHEAD SH**  $\frac{1}{2}$   $V_{\text{t}}$  results for different ratios of Ni:Fe. (E-H) *Ex-situ* <sup>5</sup>Fe Möss-<br>BAs by topotactic transformation. (color on line)<br>gen coordinated octahedral in both crystal phases of<br> $\alpha$ -phase Ni(OH)<sub>2</sub> and  $\gamma$ -phase NiOOH (D-E). The LSV results for different  $NiFe<sub>m</sub>-O<sub>x</sub>H<sub>y</sub>$  catas doped with different ratios of Ni:Fe. (E-H) *Ex-situ* <sup>3</sup>Fe Möss-<br>BAs by topotactic transformation. (color on line)<br>gen coordinated octahedral in both crystal phases of<br> $\alpha$ -phase Ni(OH)<sub>2</sub> and γ-phase NiOOH, with smal en de la componentation de<br>La componentation de la co  $H<sub>v</sub>$  exhibited the lowest overpotential of 263 mV at by topotactic transformation. (color on line)<br>
a coordinated octahedral in both crystal phases of<br>
hase Ni(OH)<sub>2</sub> and  $\gamma$ -phase NiOOH, with smaller  $\Delta$ <br>
ue indicating the octahedral symmetry should be<br>
her in later one gen coordinated octahedral in both crystal phases of  $\alpha$ -phase Ni(OH)<sub>2</sub> and  $\gamma$ -phase NiOOH, with smaller  $\Delta$  value indicating the octahedral symmetry should be higher in later one than the former one. This also furth gen coordinated octahedral in both crystal phases of  $\alpha$ -phase Ni(OH)<sub>2</sub> and  $\gamma$ -phase NiOOH, with smaller  $\Delta$  value indicating the octahedral sym metry should be higher in later one than the former one. This also furt  $\alpha$ -phase Ni(OH)<sub>2</sub> and  $\gamma$ -phase NiOOH, with smaller  $\Delta$ <br>value indicating the octahedral sym metry should be<br>higher in later one than the former one. This also fur-<br>ther illustrates the high energy resolution of the <sup></sup> NiOOH, with smaller  $\Delta$ <br>1 sym metry should be<br>mer one. This also fur-<br>resolution of the <sup>57</sup>Fe<br>nique where even sub-<br>nsity and the electric<br>uclide position can be<br>1 was also observed in<br>Raman spectra where<br>rise in peaks value indicating the octahedral sym metry should be<br>higher in later one than the former one. This also fur-<br>ther illustrates the high energy resolution of the <sup>57</sup>Fe<br>Mössbauer spectroscopic technique where even sub-<br>tle c higher in later one than the former one. This also fur-<br>ther illustrates the high energy resolution of the <sup>57</sup>Fe<br>Mössbauer spectroscopic technique where even sub-<br>tle changes in the electron density and the electric<br>fiel is illustrates the high energy resolution of the <sup>57</sup>Fe<br>issbauer spectroscopic technique where even sub-<br>changes in the electron density and the electric<br>dd distribution at the iron nuclide position can be<br>arly identified Mössbauer spectroscopic technique where even sub-<br>the changes in the electron density and the electric<br>field distribution at the iron nuclide position can be<br>clearly identified. Similar trend was also observed in<br>ex-situ

		电化学(J. Electrochem.) 2022, 28(3), 2108541 (22 of 31)				
	Table 1 Ex-situ <sup>57</sup> Fe Mössbauer spectral parameters of NiFe <sub>m</sub> -Fe PBAs at room temperature					
Sample	Valence/spin state	$\delta$ /Fe (mm·s <sup>-1</sup> )	$\Delta$ (mm·s <sup>-1</sup> )	$\Gamma_{\rm exp}(m m \cdot s^{-1})$	A(%)	
$NiFe0.11-Fe PBA$	Fe <sup>III</sup> high spin	0.44	0.50	0.43	14	
	Fe <sup>III</sup> low spin	$-0.17$	0.50	0.38	86	
NiFe <sub>0.2</sub> -Fe PBA	Fe <sup>III</sup> high spin	0.40	0.65	0.38	19	
	$\rm Fe^{III}$ low spin	$-0.16$	0.59	0.46	81	
NiFe <sub>0.25</sub> -Fe PBA	Fe <sup>III</sup> high spin	0.37	0.65	0.34	21	
	Fe <sup>III</sup> low spin	$-0.15$	0.62	0.45	79	
$NiFe0.29 - Fe PBA$	Fe <sup>III</sup> high spin	0.37	0.64	0.36	24	
	$\rm Fe^{III}$ low spin	$-0.15$	0.57	0.44	76	
	<b>Table 2</b> Ex-situ <sup>57</sup> Fe Mössbauer spectral parameters of NiFe <sub>m</sub> -O <sub>x</sub> H <sub>y</sub> at room temperature					
Sample	Valence/spin state	$\delta$ /Fe (mm·s <sup>-1</sup> )	$\Delta$ (mm $\cdot$ s <sup>-1</sup> )	$\Gamma_{\text{exp}}\left(\text{mm}\cdot\text{s}^{\text{-1}}\right)$	A(%)	
$NiFe0.11-OxHy$	$Fe3+$ high spin	0.32	0.44	0.28	100	
$NiFe0.2-OxHy$	$Fe3+$ high spin	0.32	0.58	0.42	100	
$NiFe0.25-OxHy$	$Fe3+$ high spin $Fe3+$ high spin	0.34 0.32	0.35	0.36	100 100	

	Fe <sup>m</sup> low spin	$-0.16$	0.39	<b>0.46</b>	81
	Fe <sup>III</sup> high spin	0.37	0.65	0.34	21
NiFe <sub>0.25</sub> -Fe PBA	Fe <sup>III</sup> low spin	$-0.15$	0.62	0.45	79
	$Fe^{III}$ high spin	0.37	0.64	0.36	24
NiFe <sub>0.29</sub> -Fe PBA	Fe <sup>III</sup> low spin	$-0.15$	0.57	0.44	76
	<b>Table 2</b> Ex-situ <sup>57</sup> Fe Mössbauer spectral parameters of NiFe <sub>m</sub> -O <sub>x</sub> H <sub>v</sub> at room temperature				
Sample	Valence/spin state	$\delta$ /Fe (mm·s <sup>-1</sup> )	$\Delta$ (mm $\cdot$ s <sup>-1</sup> )	$\Gamma_{\text{exp}}\left(\text{mm}\cdot\text{s}^{\text{-1}}\right)$	A(%)
$NiFe0.11-OxHy$	$Fe3+$ high spin	0.32	0.44	0.28	100
$NiFe0.2-OxHy$	$Fe3+$ high spin	0.32	0.58	0.42	100
$NiFe0.25-OxHy$	$Fe3+$ high spin	0.34	0.35	0.36	100
$NiFe0.29-OxHy$	$Fe3+$ high spin	0.32	0.43	0.33	100
	(oxy)hydroxides (NiFe <sub>m</sub> -O <sub>x</sub> H <sub>v</sub> ) was firstly developed through the topotactic transformation of $NiFem$ -Fe		zation instrument as shown in Figure $10(D)$ <sup>[66]</sup> . This in- strument has also been applied in ORR[72] and $CO_2RR$ [73],		
	PBAs in an alkaline solution. Furthermore, the phase		which provides a high level of research tool for the		
	purity and stability were thoroughly confirmed by		preparation of highly efficient catalysts of electrolytic		
	$ex$ -situ and in-situ/operando Raman and $ex$ -situ ${}^{57}Fe$		water, carbon dioxide reduction, fuel cell develop-		
	Mössbauer spectroscopies in combination with other		ment and even for detecting the structural and elec-		
	several kinds of conventional techniques. It is indi- cated that phase structure of NiFe <sub>0.2</sub> -O <sub>x</sub> H <sub>y</sub> was irre-		tronic properties of single atom catalysts $[72,73]$ . The id- entifications of durable and non-durable $FeNx$ sites in		
	versibly transformed from $\alpha$ -Ni(OH) <sub>2</sub> to $\gamma$ -NiOOH by		Fe-N-C materials for PEMFCs towards practical ap-		
	applying an anodic potential which is different from		plications have also been successfully investigated,		

**THURE 2** EX-8481 TE MOSSOURT SPOCIES OF THE-C-O.F, at room experience<br>
Simple Valence/operation can alternation of the canon of the canon of the canon of the caponal of the caponal of the caponal of the caponal of the ca Sample Valence/spin state  $\delta$ Fe (mm·s<sup>c</sup>)  $\Delta$  (mm·s<sup>c</sup>)  $I_{\text{vs}}(\text{mm}\cdot\text{s}^-)$   $I_{\text{vs}}(\text{mm}\cdot\text{s}^-)$ <br>
NiFe<sub>50</sub>-O,H, Fe<sup>31</sup>high spin 0.32 0.44 0.28 100<br>
NiFe<sub>50</sub>-O,H, Fe<sup>31</sup>high spin 0.32 0.43 0.35 0.36 100<br>
NiFe<sub>50</sub>-O,H, NiFe<sub>ar</sub>-O,II, Fe<sup>3</sup> high-spin 0.32 0.44 0.28 100<br>
NiFe<sub>ar</sub>-O,II, Fe<sup>3</sup> high-spin 0.32 0.58 0.42 100<br>
NiFe<sub>ar</sub>-O,II, Fe<sup>3</sup> high-spin 0.32 0.33 100<br>
NiFe<sub>ar</sub>-O,II, Fe<sup>3</sup> high-spin 0.32 0.43 0.33 100<br>
NiFe<sub>ar-</sub>O,II, Vas fir NiFe<sub>0c</sub>-O<sub>J</sub>H, Fe<sup>1</sup> high spin 0.32 0.58 0.42 100<br>
NiFe<sub>0c</sub>-O<sub>JH</sub>, Fe<sup>1</sup> high spin 0.32 0.35 0.36 100<br>
NiFe<sub>0c</sub>-O<sub>JH</sub>, Fe<sup>1</sup> high spin 0.32 0.43 0.33 100<br>
(oxy)hydroxides (NiFe<sub>2</sub>-O<sub>J</sub>H), was firstly developed zation ins NiFe<sub>0</sub>-O,H, Fe<sup>3</sup> high-spin 0.34 0.35 0.36 100<br>
NiFe<sub>0</sub>-O,H, Fe<sup>3</sup> high-spin 0.32 0.43 0.33 100<br>
(oxy)hydroxides (NiFe<sub>0</sub>-O,H,) was firstly developed zation instrument as shown in Figure 10(D)<sup>98</sup>. This in-<br>
drown) the t NiFe<sub>xr</sub>-O.Hy Fe<sup>+</sup> high spin 0.32 0.43 0.33 100<br>
(oxy)hydroxides (NiFe<sub>xr</sub>-O.H) was firstly developed zation instrument as shown in Figure 10(D)<sup>i64</sup>. This in-<br>
through the topotactic transformation of NiFe<sub>x</sub>-Fe strumen Example 1.1 That is to say, the matter of NiFe<sub>a</sub>,-Dependent and shown in Figure 10(D)<sup>88</sup>. This in-<br>through the topotactic transformation of NiFe<sub>a</sub>,-Fe strument has also been applied in ORR<sup>pc</sup> and CO,RR<sup>pci</sup>,<br>PBAs in a (oxy)hydroxides (NiFe<sub>x</sub>-O,II,) was firstly developed zation instrument as shown in Figure 10(D)<sup>os</sup>. This in-<br>through the topotactic transformation of NiFe<sub>x</sub>-Fe strument has also been applied in ORR<sup>721</sup> and CO<sub>-</sub>RR<sup>73</sup> (oxy)hydroxides (NiFe<sub>xr</sub>-O,H.) was firstly developed zation instrument as shown in Figure 10(D)<sup>i60</sup>. The through the topotactic transformation of NiFe<sub>xr</sub>-Fe strument has also been applied in ORR<sup>179</sup> and CO<sub>2</sub>I<br>PBAs in through the topotactic transformation of NiFe<sub>a</sub>, Fe strument has also been applied in ORR<sup>pa</sup> and CO<sub>S</sub>RR<sup>pa</sup>). The particular colution. Furthermore, the phase which provides a high level of research to for the purity an in an alkaline solution. Furthermore, the phase which provides a high level of research tool for the and stability were thoroughly confirmed by preparation of highly efficient catalysts of electrolytic at and in-situlayer purity and stability were thoroughly confirmed by<br>
perparation of highly efficient catalysts of electrolytic<br>
and in-station mechanism and car-situs <sup>29</sup>Fe water, cathon dioxide reduction, fuel cell-develop-<br>
Self-develop Müssbauer spectroscopies in combination with other<br>neeral and elec-<br>several kinds of conventional techniques. It is indi-<br>tronic properties of single atom catalysts<sup>prom</sup>, The idea<br>seted that phase structure of NiFe<sub>tro</sub>-

**Table 2** Ex-siza <sup>26</sup>C Missbauer spectral parameters of NiFe<sub>G</sub>-O,H, at room temperature<br>
Sample Valence/spin state  $\delta \text{Te}$  (mm·s<sup>c)</sup>  $\Delta$  (mm·s<sup>c</sup>)  $\Delta$  (%)<br>
NiFe<sub>Gr</sub>-O,H, Fe<sup>3+</sup>high spin 0.32 0.58 0.42 100<br>
NiFe<sub>Gr</sub>-**Table 2** *Ex*-sita<sup>2</sup>Fe Mossbauer spectral parameters of NiFe<sub>2</sub>-O.II, at room temperature<br>
NiFe<sub>0ar</sub>-O,H, Fe<sup>3</sup> bigh spin 0.32 0.44 0.28 100<br>
NiFe<sub>0ar</sub>-O,H, Fe<sup>3</sup> bigh spin 0.32 0.44 0.28 100<br>
NiFe<sub>0ar</sub>-O,H, Fe<sup>3</sup> bigh 0.59 0.46 81<br>
0.65 0.34 21<br>
0.62 0.45 79<br>
0.64 0.36 24<br>
0.57 0.44 76<br>
1.125 0.67 0.44 76<br>
1.125 0.7 0.44 76<br>
1.125 0.36 1.10<br>
0.44 0.28 1.00<br>
0.58 0.42 1.00<br>
0.35 0.36 1.00<br>
0.43 0.33 1.00<br>
2.41 0.28 1.00<br>
2.43 0.35 0.36 0.65 0.34 21<br>
0.62 0.45 79<br>
0.64 0.36 24<br>
0.57 0.44 76<br>
1.47 1.65 0.57 0.44 76<br>
1.47 1.65 0.45 1.95 0.44 1.65 0.45 0.47 1.00<br>
0.44 0.28 1.00<br>
0.58 0.42 1.00<br>
0.35 0.36 1.00<br>
0.43 0.33 1.00<br>
2.43 0.33 1.00<br>
2.43 0.33 1.00<br> 0.62 0.45 79<br>
0.64 0.36 24<br>
0.57 0.44 76<br>
1.47 1.69<br>
neters of NiFe<sub>n</sub>-O<sub>s</sub>H, at room temperature<br>  $\Delta$  (mm·s<sup>+)</sup>)  $\Gamma_{\text{ce}}$  (mm·s<sup>+)</sup>  $\Delta$  (%)<br>
0.44 0.28 100<br>
0.58 0.42 100<br>
0.35 0.36 100<br>
0.43 0.33 100<br>
2.43 0.33 100<br> 0.64 0.36 24<br>
0.57 0.44 76<br>
1.44 76<br>
1.65 0.57 0.44 76<br>
1.65 0.44 76<br>
1.66 0.47 1.00 0.58 0.42 100<br>
0.35 0.36 100<br>
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2.45 1.00<br>
2.45 1.00<br>
2.45 1.00<br>
2.45 0.57 0.44 76<br>  $\frac{1}{2}$ <br>  $\frac{1}{2}$ neters of NiFe<sub>n</sub>-O<sub>s</sub>H, at room temperature<br>  $\Delta$  (mm·s<sup>r)</sup>  $I_{\text{exp}}$  (mm·s<sup>r)</sup>  $\Delta$  (%)<br>  $0.44$  0.28 100<br>  $0.58$  0.42 100<br>  $0.35$  0.36 100<br>  $0.43$  0.33 100<br>  $0.43$  0.33 100<br>  $0.45$  100<br>  $0.47$ <br>  $0.48$  2.33 100<br>  $0.49$ <br> neters of NiFe<sub>n</sub>-O<sub>s</sub>H, at room temperature<br>  $\Delta$  (mm·s<sup>1</sup>)  $\Gamma_{\text{exp}}$  (mm·s<sup>1</sup>)  $\Delta$  (%)<br>  $0.44$   $0.28$   $100$ <br>  $0.58$   $0.42$   $100$ <br>  $0.35$   $0.36$   $100$ <br>  $0.43$   $0.33$   $100$ <br>  $0.43$   $0.33$   $100$ <br>  $0.44$   $0.28$   $0.42$   $10$ This in-<br> $D_2RR^{[73]}$ , for the trolytic evelop-<br>delec-<br>The id-sites in cal ap-<br>tigated, that re-Evers of Nire, -0,r, at room temperature<br>  $\Delta$  (mm·s<sup>2</sup>)  $\Gamma_{\text{exp}}$  (mm·s<sup>2</sup>)  $\Delta$  (%)<br>  $0.44$   $0.28$   $100$ <br>  $0.58$   $0.42$   $100$ <br>  $0.35$   $0.36$   $100$ <br>  $0.43$   $0.33$   $100$ <br>  $0.43$   $0.33$   $100$ <br>  $0.45$ <br>  $0.47$ <br>  $0.48$   $0.49$  $\Delta$  (mm·s<sup>-1</sup>)  $I_{\text{exp}}$  (mm·s<sup>-1</sup>)  $\Delta$  (%)<br>  $0.44$   $0.28$   $100$ <br>  $0.58$   $0.42$   $100$ <br>  $0.35$   $0.36$   $100$ <br>  $0.43$   $0.33$   $100$ <br>  $2.43$   $0.33$   $100$ <br>  $2.5$ <br>  $2.5$   $0.36$   $100$ <br>  $2.5$   $0.36$   $100$ <br>  $2.5$   $0.37$   $0.30$ <br> 0.44 0.28 100<br>
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2.79<sup></sup> . The in鄄situ/operando 57Fe M觟ssbauer results indi-0.43 0.33 100<br>
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20.5 ment as shown in Figure 10(D)<sup>(66)</sup>. This in-<br>also been applied in ORR<sup>[73]</sup> and CO<sub>3</sub>RR<sup>[73]</sup>,<br>des a high level of research tool for the<br>of highly efficient catalysts of electrolytic<br>on dioxide reduction, fuel cell devel zation instrument as shown in Figure 10(D)<sup>[66]</sup>. This in-<br>strument has also been applied in ORR<sup>[73]</sup> and CO<sub>2</sub>RR<sup>[73]</sup>,<br>which provides a high level of research tool for the<br>preparation of highly efficient catalysts of e zation instrument as shown in Figure 10(D)<sup>[66]</sup>. This instrument has also been applied in ORR<sup>[73</sup>] and CO<sub>2</sub>RR<sup>[73</sup>], which provides a high level of research tool for the preparation of highly efficient catalysts of ele strument has also been applied in ORR<sup>[73]</sup> and CO<sub>2</sub>RR<sup>[73]</sup>, which provides a high level of research tool for the preparation of highly efficient catalysts of electrolytic water, carbon dioxide reduction, fuel cell deve which provides a high level of research tool for the<br>preparation of highly efficient catalysts of electrolytic<br>water, carbon dioxide reduction, fuel cell develop-<br>ment and even for detecting the structural and elec-<br>troni

 $NiFe<sub>02</sub>-O<sub>x</sub>H<sub>y</sub>$  at lower applied potentials, such as 1.22 V preparation of highly efficient catalysts of electrolytic<br>water, carbon dioxide reduction, fuel cell develop-<br>ment and even for detecting the structural and elec-<br>tronic properties of single atom catalysts<sup>[72,73</sup>]. The i water, carbon dioxide reduction, fuel cell development and even for detecting the structural and electronic properties of single atom catalysts<sup>[72,73]</sup>. The identifications of durable and non-durable FeN, sites in Fe-N-C ment and even for detecting the structural and electronic properties of single atom catalysts<sup>[72,73]</sup>. The identifications of durable and non-durable FeN, sites in Fe-N-C materials for PEMFCs towards practical applicatio



ctro-<br>elec-<br>Ra-<br>tion.<br>.. (I)<br>cm<sup>-2</sup><br>right The main and the set of  $\frac{3}{2}$  and  $\frac{3}{2}$  and Example the most of Nike, O.B, before the state of t Figure 14 (A) Cysic with the R<sub>2</sub> on the observed spectral shape has been changed at the observed spectral shape has been changed at the observed spectral shape has been changed at the shape of the observed spectral shape **Example 12**<br>
Example 11 and 12 and 12 and 1<sup>2</sup> and 1<sup>2</sup> and 1<sup>2</sup> and 1<sup>2</sup> and 12 **Further increases as the applied model increases as the applied model increases (Figure 15(Ci)). The growth of Fe' (increase as the applied potential increase (Figure 15(Ci)). The amount of Fe' increase the applied poten** Example the (A) Cysin and Region Content (Figure 14(A) Cycle (The amount of Figure 14(A) Cycle (The black curves,  $\alpha$ -phase NiOOII streature). (B-C) "Fe Missbeure spectra of NiFe<sub>4</sub>-OH, before and after electro-<br>chemical **Eigure 14** (A) Cyclic vollummetric curves of NiFe<sub>3</sub>-O.II, before (the bluck curves,  $\alpha$ -phase Ni(OII), structure) and after electro-<br>chemical activation, (the declares, s-phase NiOOH structure), (H-2)<sup>-5</sup>/e Mossbauer s ehemical activation (the red curves, y-phase NiOOH structure). (B-C) <sup>2</sup>Fe Mossbauer spectra of NiFe<sub>a</sub>-O,H, before and after electronical activition. (D) Depending activation, (D) Depending activation, (D) Operator And a trochemical activation. (D) Ramam spectra (NFig-0, OII, better (black) and after (red) spepting anodic potential (E) Operato AB-<br>man spectra of Nite<sub>n</sub>-O.H, collected at different applied potentials (V vs. RHE). (i) The O  $\frac{1}{2}$ <br>  $\frac{1}{2}$ <br> spectra were similar, which indicates the excellent<br>spectra were similar, which indicates the excellent<br>spectra were similar, which indicates the excellent structure)<br>that different mole points of NiFe<sub>a</sub>-O,H, before and

to develop with the  $\delta$  value of around -0.25 mm  $\cdot s^{-1}$  OER (Figure 15(F)). The pre-OER and post-OER

Structure stability of the catalyst The resulting the catalystration of the stability which can also confirmed through the catalyst loading and a constant current density of 100 mA c m<sup>-26</sup>.<br>
C.M. The stability of the sta strate the streament of current density of the current density<br>  $\frac{1}{2}$  and  $\frac{1}{12}$  and  $\frac{1}{25}$  and  $\frac{1}{25}$  and  $\frac{1}{25}$  and after electro-<br>  $C$ ) <sup>7</sup>Fe Mössbauer spectra of NiFe<sub>a</sub>-O,H, before and after elect  $\frac{68}{1.0}$   $\frac{1}{12}$   $\frac{1}{14}$   $\frac{12}{12}$   $\frac{1}{13}$   $\frac{1}{25}$   $\frac{69}{35}$   $\frac{75}{75}$   $\frac{100}{100}$ <br>
Fime (h)<br>
he black curves, *c*-phase Ni(OH)<sub>2</sub> structure) and after electro-<br>
ck) and after (red) applying anodic Time (h)<br>
the black curves,  $\alpha$ -phase Ni(OH)<sub>2</sub> structure) and after electro-<br>  $\gamma$ <sup>pr</sup>Fe Mössbauer spectra of NiFe<sub>a2</sub>-O<sub>-</sub>H, before and after elec-<br>
ck) and after (red) applying anodic potential. (E) Operando Ra-<br>
vs. he black curves, *a*-phase Ni(OH)<sub>2</sub> structure) and after electro-<br>c)<sup>*s*T</sup>Fe Mössbauer spectra of NiFe<sub>02</sub>-O<sub>-</sub>H, before and after elec-<br>ck) and after (red) applying anodic potential. (E) Operando Ra-<br>vs. RHE). (F) The O 2) <sup>ST</sup>Fe Mössbauer spectra of NiFe<sub>02</sub>-O<sub>r</sub>H, before and after elec-<br>ck) and after (red) applying anodic potential. (E) Operando Ra-<br>*V* vs. RHE). (F) The OER polarization curves with  $i_n$  correction.<br>totally with differ ck) and after (red) applying anodic potential. (E) Operando Ra-<br>
v vs. RHE). (F) The OER polarization curves with  $i_n$  correction.<br>
tot different molar ratios of Fe/Ni and commercial RuO<sub>2</sub>. (I)<br>
tt etatalyst loadings at

 $\mathbb{E} \{k \neq (J. \text{Electrochem.}) 2022, 28(3), 2108541 (24 of 31) \}$ <br>
RHE of OER (Figure 15(C)). As listed in Table 3, it potential rather than around onset potential<sup>[47]</sup>. There-<br>
is different from the previous *in-situl operando* M 28(3), 2108541 (24 of 31)<br>potential rather than around onset potential<sup>[47]</sup>. There-<br>fore, the *in-situ* produced abundant  $Fe^{4+}$  at onset po-<br>tential and different applied potentials suggested that 28(3), 2108541 (24 of 31)<br>potential rather than around onset potential<sup>[47]</sup>. There-<br>fore, the *in-situ* produced abundant  $Fe^{4+}$  at onset po-<br>tential and different applied potentials suggested that 28(3), 2108541 (24 of 31)<br>potential rather than around onset potential<sup>[47]</sup>. There-<br>fore, the *in-situ* produced abundant  $Fe^{+}$  at onset po-<br>tential and different applied potentials suggested that<br> $\frac{C_{100}\left(\frac{100}{1.37$ 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (24 of 31)<br>
As listed in Table 3, it potential rather than around onset potential<sup>[47]</sup>. There-<br> *n-situ/operando* Möss-<br>
observed at a higher tential and different applied pot



40 <sub>1</sub> Current (mA) $\frac{8}{5}$ $\frac{8}{6}$ $\frac{8}{6}$ $-10.0$ 2.0	1.57V 1.47V $1.42$ V 1.37V 4.0 6.0 Time (h)	Current $(mA)$ $=$ $\frac{8}{3}$ $\frac{8}{3}$ $-10$ $-20 - 0$ 1.2	Current (mA) $^{40}_{30}$ $^{40}_{10}$ 1.42V 1.37V 1.47V 1.4 1.6 Potential (V vs. RHE)	40 $Fe4+$ Pre 1.37 Potential (V vs. RHE)	$40^\circledcirc$ $Fe4+$ 26 Content of 23 10 8 1.42 1.47 1.57 Post
15 The in-situ/operando ${}^{57}Fe$ Mössbauer spectra of NiFe <sub>0.2</sub> -O <sub>s</sub> H <sub>y</sub> collected at (A) the open circuit voltage, (B) 1.37 V, (C)					
(D) 1.47 V, and (E) 1.57 V (vs. RHE). (F) $Ex-situ$ <sup>57</sup> Fe Mössbauer spectrum of NiFe <sub>02</sub> -O <sub>3</sub> H <sub>v</sub> collected after OER. The unit of					
ssbauer parameter of isomer shift $(\delta)$ is mm · s <sup>-1</sup> relative to standard $\alpha$ -Fe foil. (G) The current-time curves at different applied					
als obtained during the <i>in-situ/operando</i> measurements. (H) Cyclic voltammogram without <i>i<sub>R</sub></i> correction of NiFe <sub>02</sub> -O <sub>s</sub> H <sub>y</sub> record-					
			ng the in-situ/operando measurements. (I) The content of Fe <sup>4+</sup> and corresponding electric current determined at different ap-		
otentials <sup>[66]</sup> . Copyright 2021. ELSEVIER B.V. Reproduced with permission. (color on line) 3 The content of high-valent iron in-situ produced in the NiFe <sub>02</sub> -O <sub>i</sub> H <sub>y</sub> electrocatalyst during the oxygen evolution reaction.	This work <sup>[65]</sup> (NiFe <sub>0.2</sub> -O <sub>x</sub> H <sub>y</sub> )	$Fe4+$			Previous report <sup>[47]</sup> (Layered 3:1 NiFe oxyhydroxide) $Fe4+$
Potential (V vs. RHE)	$Fe^{4+}(%)$	$\delta$ /Fe (mm·s <sup>-1</sup> )	Potential (V vs. RHE)	$Fe^{4+}(%)$	$\delta$ /Fe (mm·s <sup>-1</sup> )
1.32	$\mathbf{0}$				
1.37	2	$-0.25$			
1.42 (around onset)	12	$-0.24$	1.49 (around onset)	$\boldsymbol{0}$	
1.47	23	$-0.25$			
1.52	36	$-0.24$			
1.57	40	$-0.25$	1.62	12	$-0.27$

 $#E#(J. Electronen) 2022, 28(3), 2108541 (25 of 31)$ Fe<sup>4+</sup> has critical role in OER as shown in the last<br>
Figure 15(I). Therefore, it can be concluded that the<br>
Figure 15(I). Therefore, it can be concluded that the<br>
amount of high-v  $\frac{d_1}{k}$   $\frac{d_2}{k}$  (*L Electrochem.*) 2022, 28(3), 2108541 (25 of 31)<br>
Fe<sup>4+</sup> has critical role in OER as shown in the last and reaction intermediates interacting with each oth-<br>
Figure 15(I). Therefore, it can be  $\mathbb{E}e^{4t}$  has critical role in OER as shown in the last<br>Fe<sup>4+</sup> has critical role in OER as shown in the last and reaction intermediates interacting with each<br>Figure 15(I). Therefore, it can be concluded that the er. T

# 7 Summary

probe precursors for preparing highly efficient NiFe-<br>is an active site or Ni as some *in-situloperundo* results<br>(oxy)hytoxide (NiFe<sub>x</sub>-O.H) OER electrocatallysts by indicated that Fe sots as an active site for OER, while sphauer spectroscopic technology independently de-<br>
veloped is used to conduct in-depth research on the<br>
OER enter as  $\alpha$ -Ni(OH)<sub>2</sub>,<br>
veloped is used to conduct in-depth research on the<br>
main interaction strength of<br>
the

sis, simulation and study case results discussion. The<br> *in-situloperando* <sup>5</sup>Te Mössbauer technique has also<br>
of several other disturbing facto<br>
been successfully applied in the ORR, CO<sub>S</sub>RR, etc., selective intermediate *in-situloperando* <sup>3</sup>Fe Mössbauer technique has also of several other disturbing factors while focusing on<br>becom successibily applical in the ORR, CORR, ctc., selective interemediates. Another factor is the differ-<br>the a been successfully applied in the ORR, CO<sub>S</sub>RR, etc.,<br>the subtrofice alight bered to from configurations of *in-situl/operando* cells for the freacution both the preparation of highly efficient study of materials and *in-s* the authors hope to be able to provide a high level of the configurations of *in-situ/operando* cells for the present tool for the preparation of highly efficient study of materials under *in-situal operando* conditions.<br> research tool for the preparation of highly efficient<br>study of materials under in-situ/operando conditions.<br>etaulysis of electrolytic water, carbora dioxide reduc-<br>As per the curre stanks of in-studioperando chane-<br>tion, f catalysts of electrolytic water, carbon dioxide reduc-<br>
As per the current status of *in-situloperando* characterizations<br>
iten, ful cell development and even for detecting the<br>
itenzations analysis, on any single techniq

**in the NiFe-2-** (*J. Electrochem.*) 2022, 28(3), 2108541 (25 of 31)<br> **in the Nife-2-** (*J.* Electrochem.) 2022, 28(3), 2108541 (25 of 31)<br> **in the 2-** (16). Therefore, it can be concluded that the<br>
er. The focus of all t  $\frac{f_0(K) + f_1(K) - f_2(K) - f_3(K))}{f_0(K) + f_1(K) - f_2(K) + f_3(K))}$ <br>
Fe<sup>\*</sup> has critical role in OER as shown in the last<br>
and reaction intermediates interacting with each oth-<br>
Figure 15(I). Therefore, it can be concluded that the er.  $Ee^{i\theta}$  has critical role in OER as shown in the last<br>  $E^e$ <sup>th</sup> has critical role in OER as shown in the last<br>
and reaction internediates interacting with each other<br>
striguent IS(1). Therefore, it can be concluded tha **Example 19**<br> **Example 19 EVALUATION THE SECT ALTERT (SECT ALTERT ALTER Example 12**<br> **Example 12**  $\frac{dE}{dt}$ <br>
We  $\frac{dE}{dt}$  the service of the in OER as shown in the last<br>
Fe<sup>4+</sup> has critical role in OER as shown in the last<br>
Figure 15(I). Therefore, it can be concluded that the cr. The focus of all these *in-situlog*  $\pm \frac{\text{Re}\frac{1}{2}x}{L}$  Electrochem.) 2022, 28(3), 2108541 (25 of 31)<br>
ER as shown in the last and reaction intermediates interacting with each other<br>
enable concluded that the error and these *in-situloperando* techniques<br> **EVALUATION THE CONSECT (EXAMPLE 1900)** THE CONSECT (THE TOWER THE REST CONSECT AND THE REST AND THE REST AND NOTE THE SURFACT (OF THE TOWER THE TOWER THE TOWER THE TOWER (CONSECT) THE TOWER (THE TOWER AND THE NEW THE TOW **Home-made in the spectrochemical Fe M<sub>i</sub>-** and intermediates interacting with each of-<br>Figure 15(I). Therefore, it can be concluded that the c r. The focus of all these *in-situtoperando* techniques<br>amount of high-valent  $\mathbb{E}(E\#C, Ebeconben)$ <br>  $\mathbb{E}(E\#C, Ebeconben)$  2022, 28(3), 2108541 (25 of 31)<br>
Figure 15(I). Therefore, it can be concluded that the<br>
err. The focus of all these *in-situipperando* techniques<br>
in the NiFe<sub>xt</sub>-(oxy)hydroxide has Fe<sup>41</sup> has critical role in OER as shown in the last<br>
Figure 15(f). Therefore, it can be concluded that the<br>
or. The focus of all these *in-sinuingeranda* techniques<br>
amount of high-valent iron species *in-sinu* produced<br> Fe<sup>4</sup> has critical role in OER as shown in the last<br>
refuge 15(1). There Core, it eanls concluded that the cr. The focus of all these *th*-aid inperpendict chining<br>
Figure 15(1). The results concluded the create for the c Figure 15(1). Therefore, it can be concluded that the err. The focus of all these in-stitu/operando techniques<br>
im the NiFe<sub>Na</sub>r-(oxy)hydroxide has a positive correla-<br>
NiOOH and the changes in oxidation states of Ni and<br> amount of high-valent iron species *in-situ* produced was to investigate the transformation of Ni(OH), to<br>in the NiFe<sub>ctr</sub> (oxy)hyroxide has a positive correla-<br>bin on the inference involved as a positive correla-<br>to in w in the NiFe<sub>tie</sub>- (oxy)hydroxide has a positive correla-<br>
NiOOH and the changes in oxidation states of Ni and<br>
which is which that water oxidation reaction performance,<br>
Fe. And not of the reaction different particle in N cion with its water oxidation reaction performance,<br>
which further detections in the matrix phase for OER, while both Ni and Fe are<br>
which further detection, data and the matrix phase for OER, while both Ni and Fe are<br>
me which further deepens the understanding in the an active phase for OER, while both Ni and Fe are<br>
mechanism of NiFe-hased electrocatalysts.<br>
The important for high OFR activity in NiFe-hased OFCs.<br>
The this tutorial revie mechanism of NiFe-based electrocatalysts. important for high OER activity in NiFe-based OECs.<br> **To Summarry**<br> **The main of these** in-stinulogeneod chanceterizations could<br> **IN this turbid review, NiFe<sub>x</sub>-Eve PBAs are used EXECT THE SUMMERY FOLUT THE SULLY THE SULLY THE SULLY INTERFORMAT THE ARE THE SULLY INTERFORMAT THE PROPERTIENT AND THE SULLY AND THE SU** In this tutorial review, NiFe<sub>a</sub>-Fe PBAs are used as<br>not bring researchers to reach a consensus whether Fe<br>(oxy)hydroxide (NiFe<sub>in</sub>OH) of ER electrocatallysts by<br>indicated that Fe acts as an active site for OER, while<br>a n (oxy)hydroxide (NiFe<sub>a</sub>-O,H<sub>3</sub>) OFR electrocatalysts by<br>
indicated that Fe acts as an active site for OER, while<br>
an avole lopotactic transformation method and the NiOOII helps in the stabilization of Fe<sup>4+131</sup>. On the<br>
s a novel topotactic transformation method and the NiOOH helps in the stabilization of  $\text{Fe}^{*(78)}$ . On the home-made in-studiongermade electrochemical<sup>2</sup>Fe-Mos- other hand, some others uggested that Ni is the active soluti home-made *in-situloperando* electrochemical <sup>57</sup>Fe M<sub>0</sub>. ofter hand, some others suggested that Ni is the active<br>stehar spectroscopic technology independently de-<br>**OER** center as c-Ni(OH), nanostructure showed better<br>vel oped is used to conduct in-depth research on the<br>
OER activity compared to RuO<sub>c</sub><sup>58</sup>. Moreover, the opti-<br>
Re al active intermediates and working mecha-<br>
m. Here we introduced in detail the application of the Sahatier cr OFR real active intermediates and working mechanismum and interaction strength of Ni with OH<sub>56</sub> also satisfies<br>insulling or three wintroduced in detail the application of the Sahatier criteria for the required design of mism. Here we introduced in detail the application of<br> *in-sintopermale* <sup>3</sup>TE Misschaure rechnique in the catalytes<sup>879</sup>. There coold be several factors that are con-<br> *in-sintopermale* Selectrochemical OER test process, in-situ/operando <sup>3</sup>Fe Mössbauer technique in the catalysts<sup>59</sup>. There could be several factors that are con-<br>electrochemical OEC test process, including its won<sup>t</sup>- sidered as the potential factors for different active<br>i electrochemical OFR test process, including its work-<br>
sidered as the potential fietors for different active<br>
ing principle, instrumentation, the design of reaction slits during *in-situlogerando* chancetterizations. For<br> ing principle, instrumentation, the design of reaction<br>
sites during *in-situ/operando* characterizations. For<br>
cell, NiFe-based OFCs sample detection, data analy-<br>
sample during *in-situ/operando* situles, it becomes<br>
in cell, NiFe-based OECs sample detection, data analy<br>
example, during *in-situ/operando* studies, it becomes<br>
sis, simulation and study case results discussion. The<br>
difficult to find out real active sites in the presences<br> 28(3), 2108541 (25 of 31)<br>and reaction intermediates interacting with each oth-<br>er. The focus of all these *in-situ/operando* techniques<br>was to investigate the transformation of  $Ni(OH)_2$  to<br>NiOOH and the changes in oxidati 28(3), 2108541 (25 of 31)<br>and reaction intermediates interacting with each oth-<br>er. The focus of all these *in-situ/operando* techniques<br>was to investigate the transformation of Ni(OH)<sub>2</sub> to<br>NiOOH and the changes in oxida  $28(3)$ ,  $2108541$  ( $25$  of  $31$ )<br>and reaction intermediates interacting with each oth-<br>er. The focus of all these *in-situ/operando* techniques<br>was to investigate the transformation of Ni(OH)<sub>2</sub> to<br>NiOOH and the changes 28(3), 2108541 (25 of 31)<br>
and reaction intermediates interacting with each oth-<br>
er. The focus of all these *in-situ/operando* techniques<br>
was to investigate the transformation of Ni (OH)<sub>2</sub> to<br>
NiOOH and the changes in 28(3), 2108541 (25 of 31)<br>
and reaction intermediates interacting with each other.<br>
The focus of all these *in-situ/operando* techniques<br>
was to investigate the transformation of Ni(OH)<sub>2</sub> to<br>
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NiOOH helps in the stabilization of Fe<sup>4+(75</sup>!. O is an active site or Ni as some *in-situ/operando* results<br>indicated that Fe acts as an active site for OER, while<br>NiOOH helps in the stabilization of Fe<sup>4+(75</sup>l. On the<br>other hand, some others suggested that Ni is the ac indicated that Fe acts as an active site for OER, while<br>NiOOH helps in the stabilization of Fe<sup>4+[75]</sup>. On the<br>other hand, some others suggested that Ni is the active<br>OER center as  $\alpha$ -Ni(OH)<sub>2</sub> nanostructure showed bett NiOOH helps in the stabilization of  $Fe^{i+[75]}$ . On the<br>other hand, some others suggested that Ni is the active<br>OER center as  $\alpha$ -Ni(OH)<sub>2</sub> nanostructure showed better<br>OER activity compared to RuO<sub>2</sub><sup>76</sup>. Moreover, the opt other hand, some others suggested that Ni is the active<br>OER center as  $\alpha$ -Ni(OH)<sub>2</sub> nanostructure showed better<br>OER activity compared to RuO<sub>2</sub><sup>rog</sup>. Moreover, the opti-<br>mal interaction strength of Ni with OH<sub>ad</sub> also sa OER center as  $\alpha$ -Ni(OH)<sub>2</sub> nanostructure showed better<br>OER activity compared to RuO<sub>2</sub><sup>rog</sup>. Moreover, the opti-<br>mal interaction strength of Ni with OH<sub>ad</sub> also satisfies<br>the Sabatier criteria for the required design of OER activity compared to RuO<sub>2</sub><sup>76</sup>. Moreover, the optimal interaction strength of Ni with OH<sub>ad</sub> also satisfies<br>the Sabatier criteria for the required design of OER<br>catalysts<sup>259</sup>. There could be several factors that are al interaction strength of Ni with OH<sub>ad</sub> also satisfies<br>
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lered as the potential factors for different activ the Sabatier criteria for the required design of OER<br>catalysts<sup>[39]</sup>. There could be several factors that are con-<br>sidered as the potential factors for different active<br>sites during  $in\text{-}situ/operatordo$  characterizations. For<br>exa catalysts<sup>250</sup>. There could be several factors that are considered as the potential factors for different active<br>sites during *in-situloperando* characterizations. For<br>example, during *in-situloperando* studies, it become sidered as the potential factors for different active<br>sites during *in-situloperando* characterizations. For<br>example, during *in-situloperando* studies, it becomes<br>difficult to find out real active sites in the presences<br>o sites during *in-situloperando* characterizations. For<br>example, during *in-situloperando* studies, it becomes<br>difficult to find out real active sites in the presences<br>of several other disturbing factors while focusing on<br>s example, during *in-situ/operando* studies, it becomes<br>difficult to find out real active sites in the presences<br>of several other disturbing factors while focusing on<br>selective intermediates. Another factor is the differ-<br>e 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (25 of 31)<br>as shown in the last and reaction intermediates interacting with each oth-<br>be concluded that the er. The focus of all these *in-situloperando* techniques<br>ecies *in-s* 

difficult to find out real active sites in the presences<br>of several other disturbing factors while focusing on<br>selective intermediates. Another factor is the differ-<br>ent configurations of *in-situloperando* cells for the<br> of several other disturbing factors while focusing on selective intermediates. Another factor is the different configurations of *in-situ/operando* cells for the study of materials under *in-situ/operando* conditions. As p selective intermediates. Another factor is the different configurations of *in-situ/operando* cells for the study of materials under *in-situ/operando* conditions.<br>As per the current status of *in-situ/operando* conditions ent configurations of *in-situ/operando* cells for the<br>study of materials under *in-situ/operando* conditions.<br>As per the current status of *in-situ/operando* charac-<br>terizations analysis, not any single technique has<br>show study of materials under *in-situ/operando* conditions.<br>As per the current status of *in-situ/operando* charac-<br>terizations analysis, not any single technique has<br>shown comprehensively the ability to study the phase<br>transf As per the current status of *in-situloperando* characterizations analysis, not any single technique has shown comprehensively the ability to study the phase transformation, changes in valence state and variation in morpho

of probi ng the local electronic structure, identifying  $\#E\#(J. Electron) \geq 222, 28(3), 2108541 (26 of 31)$ <br>of probing the local electronic structure, identifying<br>the active sites and phases, determining the crystal<br>structure, and tracking the oxidation state and environ-<br>tional Natura the  $\mathbb{E}\{\mathcal{E}^{\#}(J. Electron})$  2022, 28(3), 2108541 (26 of 31)<br>of probing the local electronic structure, identifying **Acknowledgements**<br>the active sites and phases, determining the crystal This work was financially supported  $\frac{dE\#(J. Electronchem)}{dt}$ <br>
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## Acknowledgements

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# 原位 5Fe 穆斯堡尔光谱技术及其在 Ni-Fe 基析氧反应电催化剂中的应用 1963/1 Electrochem.) 2022, 28(3), 2108541 (30 of 31)<br>
Jafar Hussain Shah<sup>1</sup>, 谢起贤<sup>2</sup>, 匡智崇<sup>1</sup>,格日乐<sup>1</sup>, 周雯慧<sup>1</sup>, 刘朵绒<sup>1</sup>,<br>
Jafar Hussain Shah<sup>1</sup>, 谢起贤<sup>2</sup>, 匡智崇<sup>1</sup>, 格日乐<sup>1</sup>, 周雯慧<sup>1</sup>, 刘朵绒<sup>1</sup>,<br>
Alexandre I. Rykov<sup>1</sup>, 李旭宁<sup>1</sup>, 罗景山<sup>2</sup>, 电化学(*L Electrochem.*) 2022, 28(3), 2108541 (30 of 31)<br> **位 <sup>57</sup>Fe 穆斯堡尔光谱技术及其在**<br> **Fe 基析氧反应电催化剂中的应用**<br>
sain Shah<sup>1</sup>, 谢起贤?, 匡智崇!, 格日乐!, 周雯慧!, 刘朵绒!,<br>
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<br>  $\bf{Fe}$  穆斯堡尔光谱技术及其在</sub>

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**有效: 有效: 有效: 502-2502-28(3), 2108541 (30-6731)**<br> **原位『Fe 穆斯堡尔光谱技术及其在**<br>
Ni-Fe 基析氧反应电催化剂中的应用<br>
Jafar Hussain Shah!, 谢起贤?, 匡智棠!, 格日乐!, 周变慧!, 刘朵线!,<br>
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(1. 中国学院大连任物理研究所, 大道 116023; 2. 南开大学, 利用可再生能源存储系统和水分解生产清洁氢能燃料的重大障碍。OER 过程涉及四电子、四质子耦合并形成氧-氧(O-O)键,因此动力学上进程缓慢。为提升其在水分解产氢及二氧化碳还原反应中的应用,需要开发高效催化 剂,降低 OER 过电位,以减轻能量转换过程中固有的能量损失。研究表明,IrO2 和 RuO2 具有较低析氧过电位,但 储量低、价格昂贵,大大限制了其在析氧反应中的大规模应用。而 Ni-Fe 基析氧催化剂在碱性水分解反应中展现 了优异的性能,其在水分解过程中的催化机制仍有待进一步研究。

为了解决 Ni-Fe 基催化剂在析氧反应过程中反应位点及催化反应机制等关键问题。迫切需要更先进的原位 技术来准确表征,原位追踪催化剂形态变化与电解质/电极之间的界面相互作用的影响。光谱与电化学结合的原 位技术可以监测析氧反应过程催化剂自身的变化。目前,已有大量原位光谱技术与电化学进行结合,揭示 Ni-Fe 基催化剂在 OER过程中的反应机理及活性位点, 包括原位表面增强拉曼光谱、原位同步辐射 X 射线吸收光谱、原 位紫外-可见光谱、原位扫描电化学显微镜及原位穆斯堡尔光谱等。其中,原位拉曼技术可以观察 Ni-Fe 催化剂的 振动,可以在电解液中施加测试电压条件下监测电化学反应过程中的中间体,从而提供实时反应信息,有助于追 踪电化学驱动反应是如何发生的。原位同步辐射技术可以研究OER 过程中 Ni-Fe 催化剂材料的电子结构和局部 几何结构的信息,但目前的研究中更多的是探究 Ni 的价态变化,对 Fe 的研究信息较少。原位紫外-可见光谱也主 要是针对 Ni(OH)2 的变化展开研究,逐渐提高施加电位, Ni(OH)2 会向着 NiOOH 逐渐变化, 紫外-可见技术可以追 踪 Ni-Fe 基电催化剂中的金属氧化过程。众多电化学原位光谱技术中,"Fe 穆斯堡尔谱因具有超高的能量分辨率, 是确定催化剂相结构、鉴定活性位点、阐明催化机理以及确定催化活性与催化剂配位结构之间关系的最佳手段。 此外,原位穆斯堡尔光谱技术基于原子核和核外电子的超精细相互作用而给出的同质异能移、四极矩分裂以及有 效磁场等针对催化剂中的 Fe 位点的氧化态、电子自旋构型、对称性和磁性信息进行研究、为 Ni-Fe 基催化剂在析 氧反应中的应用提供强有力的支持。 <sup>1957</sup> <sup>年</sup>袁德国科学家鲁道夫窑路德维希窑穆斯堡尔渊Rudolf Ludwig M觟ssbauer冤在其 <sup>27</sup> 岁时袁发现作为晶格谐

振子的原子在发射或吸收 γ 射线时以一定的概率不会改变它们的量子力学状态, 而这一 γ 射线的核共振吸收现 象于 1961 年获得诺贝尔物理学奖,不久后被命名为穆斯堡尔效应。穆斯堡尔效应是来自于无反冲的 γ 射线吸收 和发射的核共振现象,能量 E。处于激发态的原子核(Z 质子和 N 中子)通过产生能量为 E<sub>y</sub> 的 p 射线跃迁到能量 为  $E_\mathrm{g}$  的基态,  $\gamma$  射线可能会被处于基态的另一个相同类型的原子核(相同的 Z 和 N)吸收 ,从而转变为能量  $E_\mathrm{e}$  的 激发态。只有当发射线和吸收线足够重叠时,才能看到共振吸收。

原位穆斯堡尔谱在 Ni-Fe 催化剂析氧反应中应用,首先需要搭建 SFe 穆斯堡尔谱仪与电化学工作站联用。标 准的穆斯堡尔光谱仪主要由放射源(通常是 SCo 在 Rh 或 Pd 金属基质中用于 SFe 穆斯堡尔光谱)、速度传感器、 速度校准装置、波形发生器和同步器、y射线检测系统、多通道分析仪、计算机,并且可选配低温恒温器或高温烘 箱,以控制测量过程处于适宜温度。实际测试过程中,穆斯堡尔谱可以通过速度扫描方法生成,利用移动驱动器 或速度传感器以特定速度重复移动源或样品(所谓的多普勒运动),同时 γ 射线连续传输或发射穿过样品并计数 在同步通道上。获得穆斯堡尔谱图后,基于穆斯堡尔谱数据库(https://medc.dicp.ac.cn/,由中国科学院大连化学研 究所穆斯堡尔效应数据中心从全世界收集的穆斯堡尔谱样品数据).对 <sup>5</sup>Fe 穆斯堡尔谱进行分析拟合, 对含 Fe 基 材料的物相、价态、自旋态和配位结构进行归因和分析。数据分析拟合主要利用 MossWinn 数据分析和拟合软件 (http://www.mosswinn.com/)。以 Ni-Fe 氢氧化物催化剂为例,对于原始催化剂,其仅存在一种 Fe3+ 物种,当该催化 剂参与 OER 过程后,可能会存在 Fe\*,在双峰基础上, 拟合结果中则会出现肩峰向负侧移动现象, 可以确认高价

Fe 的存在,例如 Fe4+。为充分证明高价 Fe 的存在,对于 Ni-Fe 基催化剂的穆斯堡尔谱测试,还需在工况条件下进 行原位测试。

20 世纪 80 年代后期, 非贵金属氧化物和氢氧化物代替贵金属氧化物阳极催化剂的电解水研究开始受到关 注。Corrigan 等通过将 Fe 杂质引入 NiO 阳极, 测试过程中发现 OER 活性会增加, 但后续的研究中对于 Fe 究竟如 何改变 Ni基催化剂的 OER 性能仍旧不清晰。尔后,原位穆斯堡尔谱的引入逐渐揭开 Fe 在 Ni-Fe 电催化水分解析 氧反应中的作用。为提高测试准确性并保证穆斯堡尔谱信号的稳定,本实验室对原位穆斯堡尔谱装置做了开发和 改进。主要包括三部分:(1) 穆斯堡尔光谱仪, (2) 电化学工作站, 以及(3) 自主设计的原位 OER 电化学反应池。在 我们的实验室中袁使用了具有 14.4 keV <sup>级</sup> <sup>酌</sup> 射线的单线57Fe 穆斯堡尔谱放射源 57Co渊Rh冤袁可以减少电解液中的信 电化学(*J. Electrochem.*) 2022, 28(3), 2108541 (31 of 31)<br>Fe 的存在,例如 Fe\*。为充分证明高价 Fe 的存在,对于 Ni-Fe 基催化剂的德斯堡尔谱测试, 还需在工民条件下进<br>行业处理论。<br>生长Corrigat 等通过有限,非费金属氧化物和氢氧化物代替要金属氧化物和限能化剂的研究中对下 Pe 预靠的 Fe 化工作器 2011年30 年代后期,非费金属氧化物和氢氧化物分类原理和 行测试,测试前首先用 a-Fe 对穆斯堡尔谱仪进行多普勒速度校准, 在进行原位穆斯堡尔谱-OER 实验之前, 电解 液用氮气或氩气饱和以去除溶解的氧气。为了保证测试信号的准确性,实验中所使用的电解池不含任何 Fe 杂质, 因此采用了 Teflon 材料。为避免测试过程中产生的 O2 气泡对信号产生干扰,可以采用蠕动泵循环电解液,并且 保证测试过程中局部的微反应环境的一致性。对于普通 OER 测试,仅需要少量催化剂,但对于原位 SFe 穆斯堡尔 谱测试,只有保证 Ni-Fe 催化剂中 STe 含量充足的条件下,才可以获得高质量信号。但 OER 过程中,不建议催化 剂载量过高,催化过程中主要是表面催化剂在反应,当样品过厚时,深层样品无法参与析氧反应过程,可能会有部 分 Fe 仍旧维持 Fe<sup>3+</sup>状态。通常,对于常规 <sup>5</sup>Fe 穆斯堡尔光谱测量的催化剂,若在制备中使用普通 Fe 源,则需要 Fe 含量在 5 ~ 10 mg窑cm-2 Fe 含量在 5~10 mg·cm<sup>2</sup>.这其中仅有~2.2%的自然丰度 <sup>s</sup>Fe同位素,需要长时间监测才可以采集到信号。为保证 实验的顺利进行,可以在样品制备过程中直接使用 5Fe 源, 方便快捷采集高质量信号。为了保证样品测试的准确 性,在 OER开始前,我们可以在同一电解液中,在开路电位(OCP)下,对其进行测试,这一原始样品的测试可与后 续施加电位的 Ni-Fe 催化剂测试结果进行对比。有外加电压测试时,需要保证催化剂处于稳定状态下进行测试, 整个测试过程中保持电流密度稳定,这不仅可以保证催化剂的稳定性,还有助于确定催化剂的真实结构。 氣反应中的作用、为接高割近准确,用于原证检验整治措置与销检信息、本实验室室的原位数据维生素,正常是监管管理、开关性能、指标、提供性能、有效性。其他是当前一般性能、相关性能够、对应当性能够的。其他是当前,但是当前进行的,其他是当前进行的。<br>我们的实验室中"海川"(1月每14年以前模式的电位学生标准,以及的直接数约GKS (2013)、可以成少事情来了。<br>我们的实验室中"奥斯塔的单位 1.22 V 的类的单线下的、对应当放射器 2.22 V 我们的实金中,使用了具有14年以下,使用我的"按照整备增强系指数的需求需要提高系指数。"我们的实验室,从时间的实验,不是一种需要的。<br>行为就让新活动首先用品+2 被爆炸器分离性后程的方式,或者使用的使用需要量量,但CB 基层,则是,则是,可以使用的变化,需要是可以,进行可以表明的要求,而且,所以,进行可以使用的时间,不同时,其时间的是应对数据的事件。我们可以按照时间,我们可以按照时间,我们可以按照时间,我们可以按照时间,我们可以按照时间, 号資源并承得全大層金的信噪比。兩臂空間和6.45 mm程序,对于常规的 OER 测试,在鉴温 298 K条件下进<br>传统进入测试商品,在多效率增强的需求增强性化学生性化学生性化学性化学性化学生性化学验与所 -0.15 mm -0.25 mm -含量逐渐增加<sup>遥</sup> <sup>在</sup>OER 过程中袁施加电位 1.42 V vs. RHE <sup>时</sup>袁Fe4+ 含量 ~ 12%<sup>遥</sup> 当施加的电势达到 1.57 V <sup>时</sup>袁催化 电化学渊J. Electrochem.<sup>冤</sup> 2022, 28(3), 2108541 (31 of 31)

利用原位 <sup>s</sup>Te 穆斯堡尔谱, 我们对通过 Ni-Fe 普鲁士蓝类似物原位拓扑转换获得的 Ni-Fe 羟基氧化物电催 化剂进行了测试。基于原位拉曼技术,我们发现在阳极电位下,Ni-Fe 催化剂中 α-Ni(OH), 相会不可逆转变为 NiFe<sub>0.7</sub>-O.H. 中仅处于 +3 氧化态, 其光谱结果与开路电位下 NiFe<sub>0.7</sub>-O.H. 谱图相似, 其中只有一个双峰, 两个峰的  $^{\text{-1}}$ ,可以归属为 Fe $^{\text{4+}}$  。随着电压的逐渐增加 ,催化剂中的 Fe $^{\text{4+}}$ 剂中 Fe<sup>4+</sup> 的含量进一步增加到约 40%。这一实例充分展现了原位 Spe 穆斯堡尔谱与 Ni-Fe 催化 OER 过程的应 用,也体现了 NiFe0.2-O,H,催化剂原位产生的 Fe<sup>4+</sup> 物种的量与其水氧化反应性能呈正相关,进一步加深了对 Ni-Fe 水氧化催化机理的理解。

Ni-Fe 基催化剂因其价格低廉, 电催化析氧性能优异, 因此成为碱性水分解析氧过程的理想候选者。虽然 Ni-Fe 基电催化剂表现出优异的 OER 活性,但缺乏长期稳定性阻碍了其在商业中的应用。因此,充分了解 Ni-Fe 催化剂的衰减机理,包括形态、组成、晶体结构和活性位点数量的变化,对于设计稳定和高效 Ni-Fe 催化材料非常 重要,充分了解 Ni-Fe 催化剂在 OER 过程中的电子结构及其与析氧反应中间体的相互作用尤为重要。原位拉曼 及原位紫外-可见光谱可以对 Ni-Fe 催化剂中的 Ni(OH)2 到 NiOOH 的变化进行深入探究,而原位 5Fe 穆斯堡尔 谱测试则可以揭示 Ni-Fe 基催化剂中 Fe 的电子环境及其电子的、结构的和磁性的变化。穆斯堡尔光谱为研究 Ni-Fe 催化剂中 Fe 的局部电子结构、局部配位、键合和氧化态的提供了强大技术支撑。最近,穆斯堡尔光谱在电 催化领域获得了越来越多的关注,它对于检测不同铁基催化材料中的主要活性位点有着重要作用。

关键词: 析氧反应: Ni-Fe 羟基氧化物电催化剂:原位电化学穆斯堡尔光谱技术: Ni-Fe 普鲁士蓝类似物:关键中 间物表征