TEM in In-CAEM - CAT

Prof. Jordi Arbiol

Scientific Coordinator of In-CAEM

GAe-N GROUP OF ADVANCED ELECTRON NANOSCOPY

ICREA

ICN2 Institut Català de Nanociència i Nanotecnologia

BIST Barcelona Institute of Science and Technology

CSIC

e⁻ DREAM EUROPEAN DISTRIBUTED RESEARCH INFRASTRUCTURE FOR ADVANCED ELECTRON MICROSCOPY

ICN2 Institut Català de Nanociència i Nanotecnologia

IFAE Institut de Física d'Altes Energies EXCELENCIA SEVERO OCHOA

ICMAB INSTITUT DE CIÈNCIA DE MATERIALS DE BARCELONA EXCELENCIA SEVERO OCHOA CSIC

ALBA

Finançat per:

Unió Europea Fons Europeu Next Generation

GOBIERNO DE ESPAÑA

Plan de Recuperación, Transformación y Resiliencia

Next Generation Catalunya

Generalitat de Catalunya Departament de Recerca i Universitats

In-situ Correlative facility for Advanced Energy Materials (In-CAEM)

In-CAEM aims to develop and commission a singular infrastructure for research in advanced materials in order to address the scientific challenges of the European Green Deal and contribute to the promotion of a more sustainable economy of the European Union.

In-CAEM will enable correlative in-situ experiments combining (S)TEM (Scanning Transmission Electron Microscopy), AFM / STM (Atomic Force Microscopy / Scanning Tunneling Microscopy) and synchrotron radiation beamlines at ALBA.

- Structure ↔ Function.
- Operando & in-situ.
- Multi-modal & multi-lengthscale analysis.
- Advanced data analysis: HPC, deep learning,…

Mix & Match techniques and methodologies to tackle complex problems

Single entry point: ALBA User office

In-CAEM can be regarded as an expansion of the JEMCA advanced microscopy center in development at ALBA and could be a model for the future ALBA Science, Technology and Innovation Park (ASTIP).

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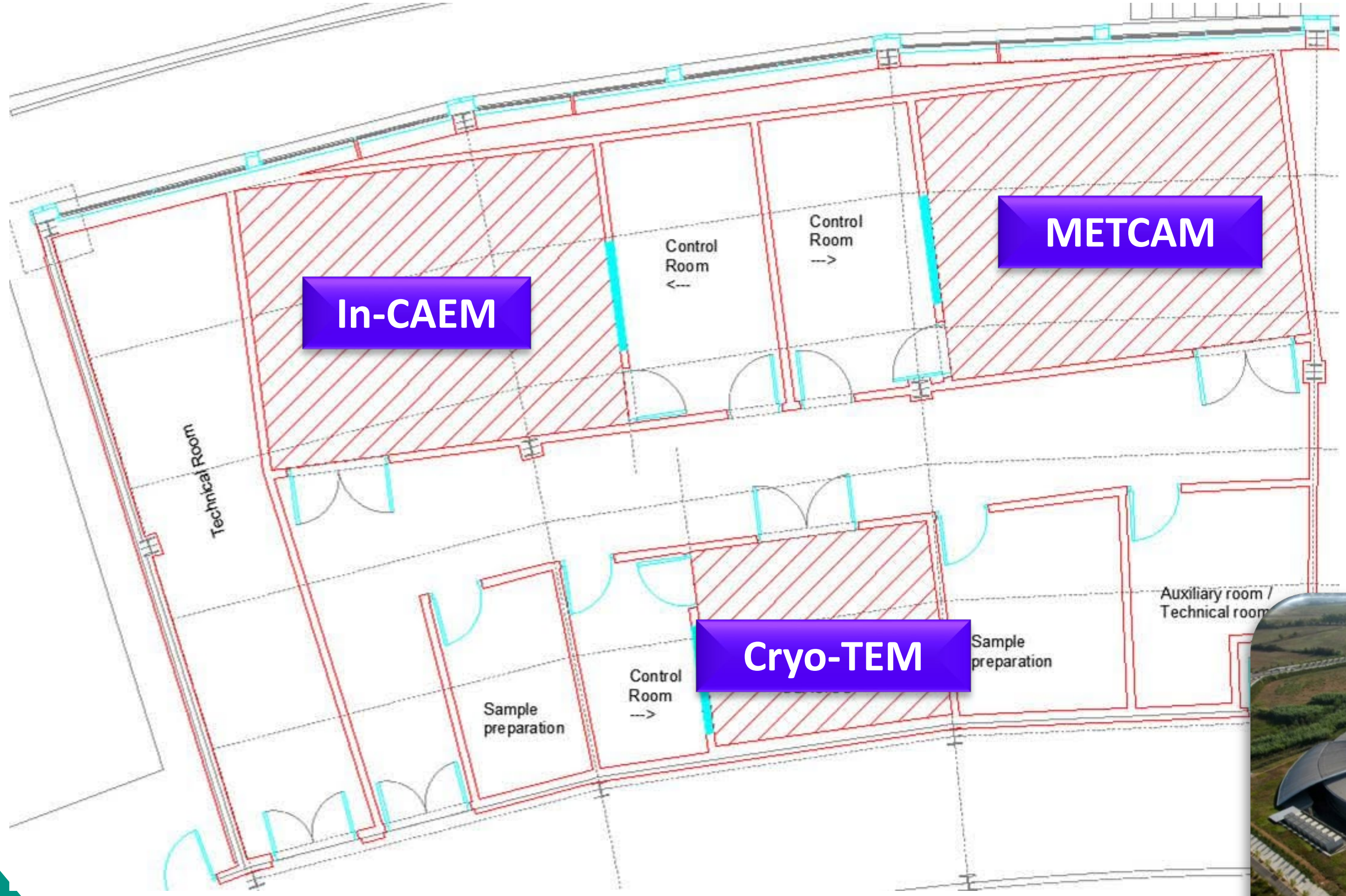
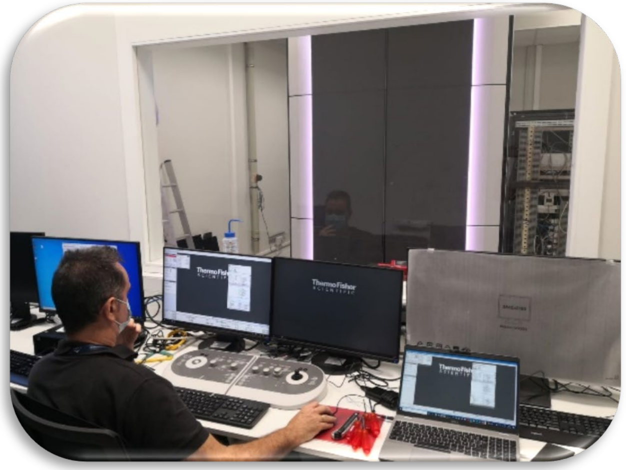
Joint EM Center at ALBA (JEMCA)





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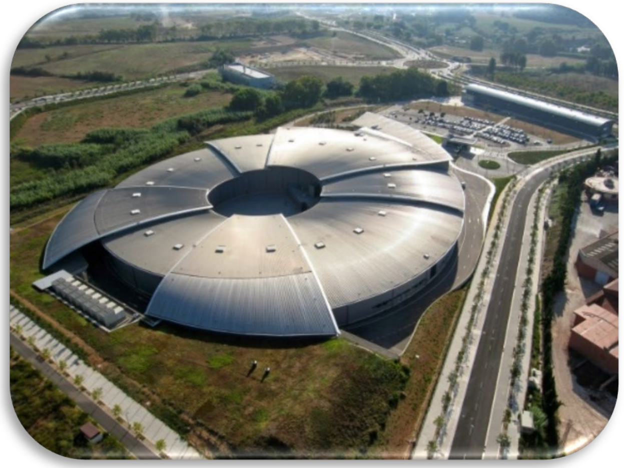
JEMCA METCAM-FIB

Unió Europea
Fons Europeu de Desenvolupament Regional
Generalitat de Catalunya
Departament de Recerca i Universitats

ICN2 Institut Català de Nanociència i Nanotecnologia — Excelencia Severo Ochoa
ALBA
CSIC Consejo Superior de Investigaciones Científicas
ICMAB Institut de Ciència de Materials de Barcelona — Excelencia Severo Ochoa
UAB Universitat Autònoma de Barcelona
Unió Europea Fons Europeu de Desenvolupament Regional
Institució CERCA Centres de Recerca de Catalunya
Generalitat de Catalunya Departament de Recerca i Universitats
QTEP* Quantum Technologies Platform
CSIC Consejo Superior de Investigaciones Científicas
Ministerio de Ciencia e Innovación
Financiado por la Unión Europea NextGenerationEU
Plan de Recuperación, Transformación y Resiliencia
Agencia Estatal de Investigación



Private donors/sponsors:

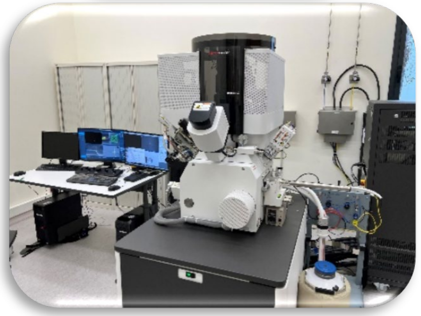
BIST

Microsoft

Budget: 4,098 M€ + 0,850 M€ *

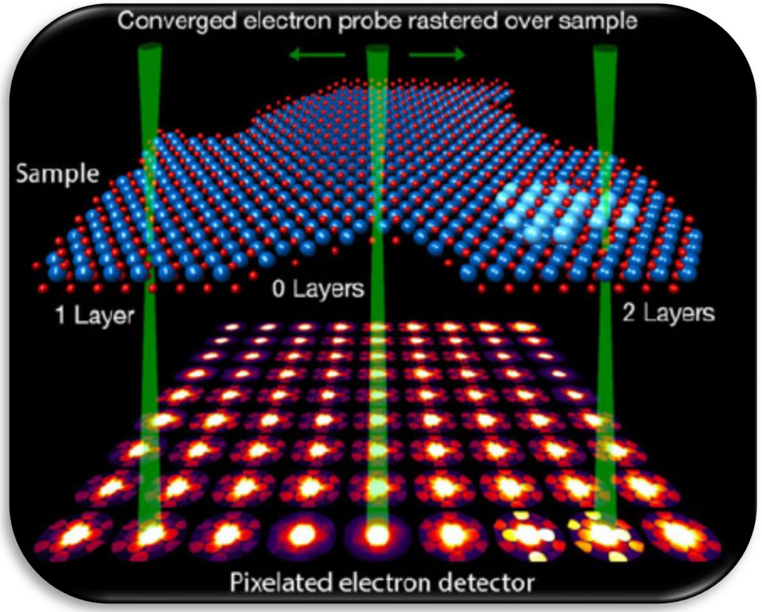
- **METCAM 60-300 keV (S)TEM:**
  - Double corrected and Monochromated
  - HREELS with direct electron detector *
  - Super EDX
  - 4D STEM
  - Spatial res. < 50 pm
  - Energy res. < 30 meV

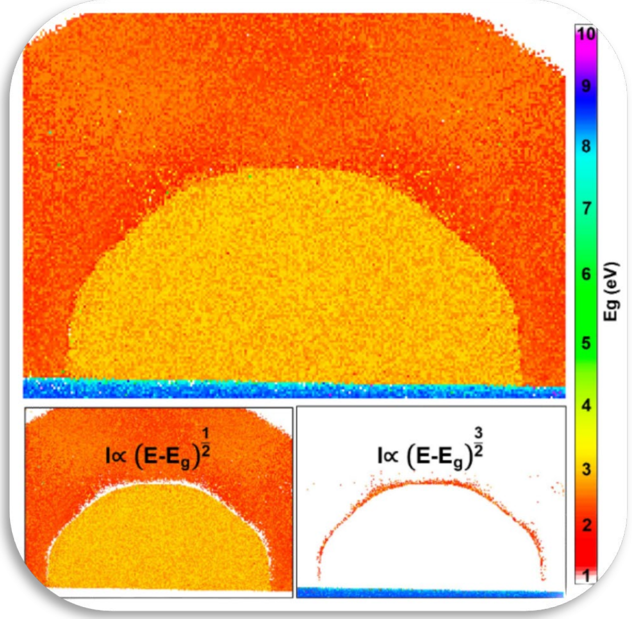
- **FIB for sample prep:**
  - Cryo-FIB holder *
  - Highly precise Ion gun
  - FEG HRSEM column
  - C and W deposition
  - Automated sample prep.

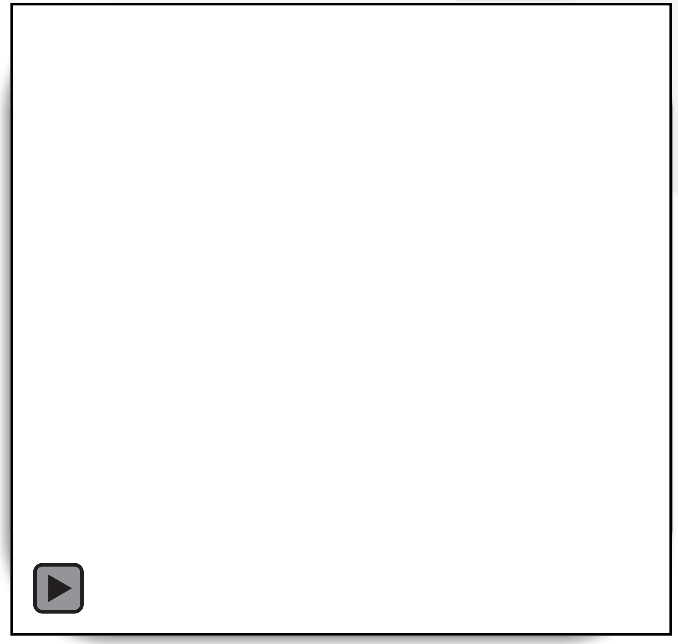


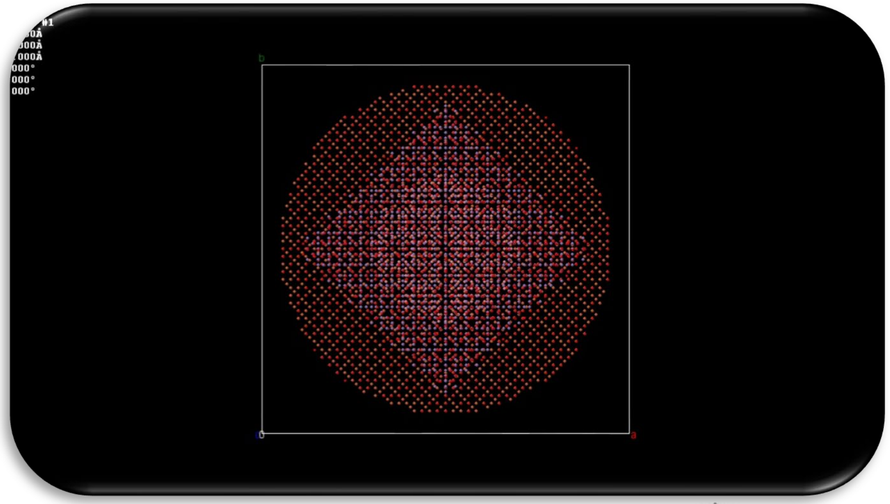
HR imaging, spectroscopy & diffraction

JEMCA









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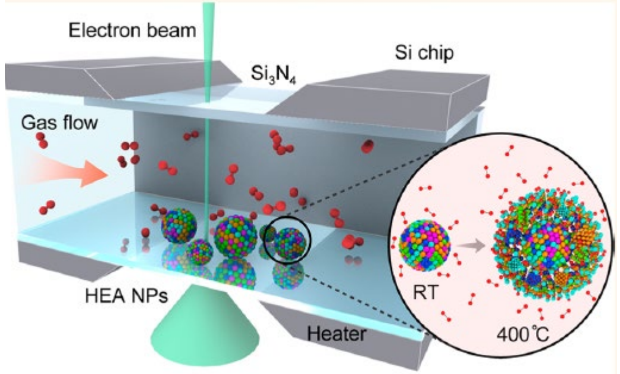
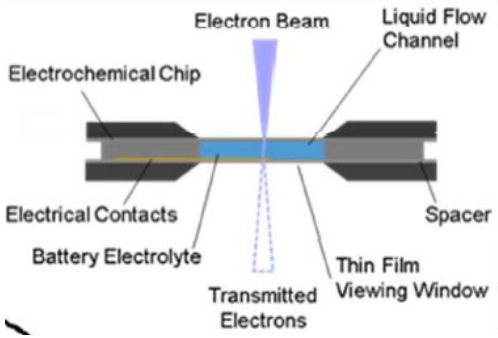
In-situ (S)TEM

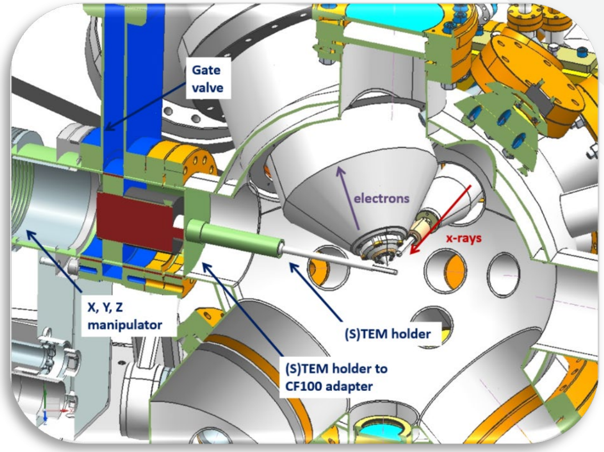
- $(AC)^2$ & Monochromated (S)TEM:
  - 4D STEM
  - EDX
  - EELS
- Catalysis sample holder system
  - Controlled gas mixing and flow
  - RGA
  - Controlled T + bias
- Electrochemistry sample holder system
  - Liquid Flow
  - In-situ electrodes
  - Potentionstat
- Liquid nitrogen cooled sample holder
- $H_2/O_2/Ar$ Plasma cleaner



Song et al, ACS Nano (2020)

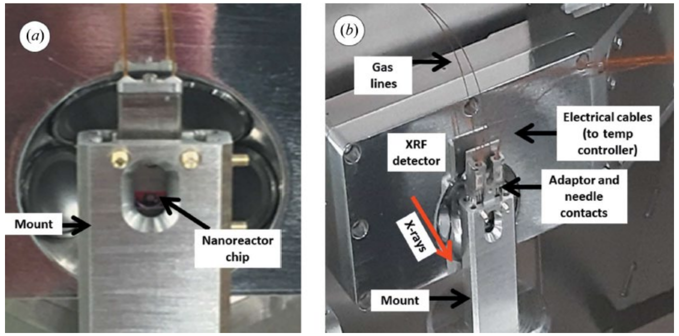


Singh et al, ACS App. Ener. Mat. (2020)



ALBA BLs

XAS, XRD, …



Parker et al, JSR (2021)

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Infrastructure for Correlative Analysis of Energy Materials

In-CAEM will develop a **singular infrastructure** for research in **advanced energy materials** in order to address the scientific challenges of the European Green Deal:

Multi-modal
Multi-lengthscale
Correlative

} **in situ/operando** experiments, combining {

(Scanning) transmission electron microscopy
Scanning probe microscopies
Synchrotron X-rays (spectroscopy, diffraction,…)
Advanced data analytics (HPC, deep learning, AI,…)

Open to all the scientific community.

Mix & Match techniques and methodologies to tackle complex problems

Single entry point: ALBA User office

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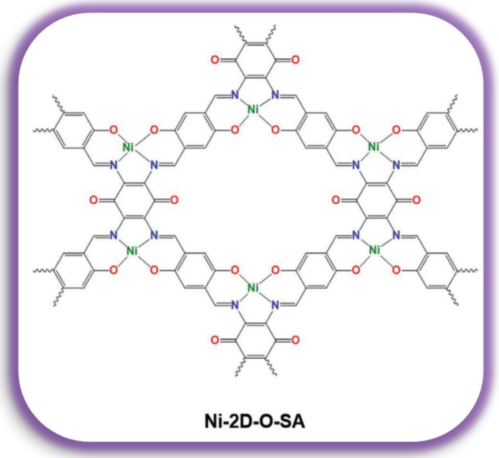
MULTIMODAL NANOCHARACTERIZATION

With: IREC — Institut de Recerca en Energia de Catalunya / Catalonia Institute for Energy Research

1. Ni-Fe SACs for CO_2 RR

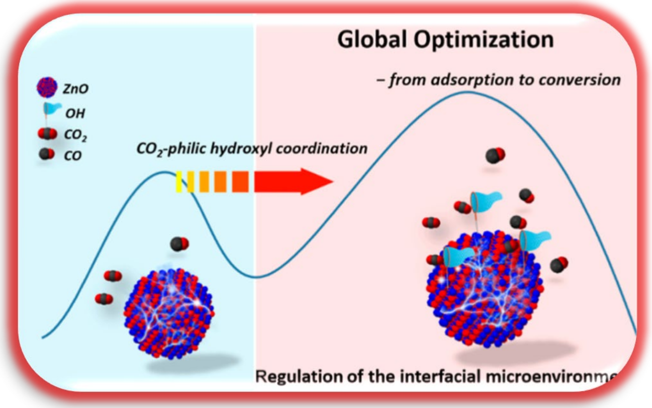
Energy Environ. Science, **14**, 4847 (2021)

2. Ni-SACs for Alcohol Oxidation



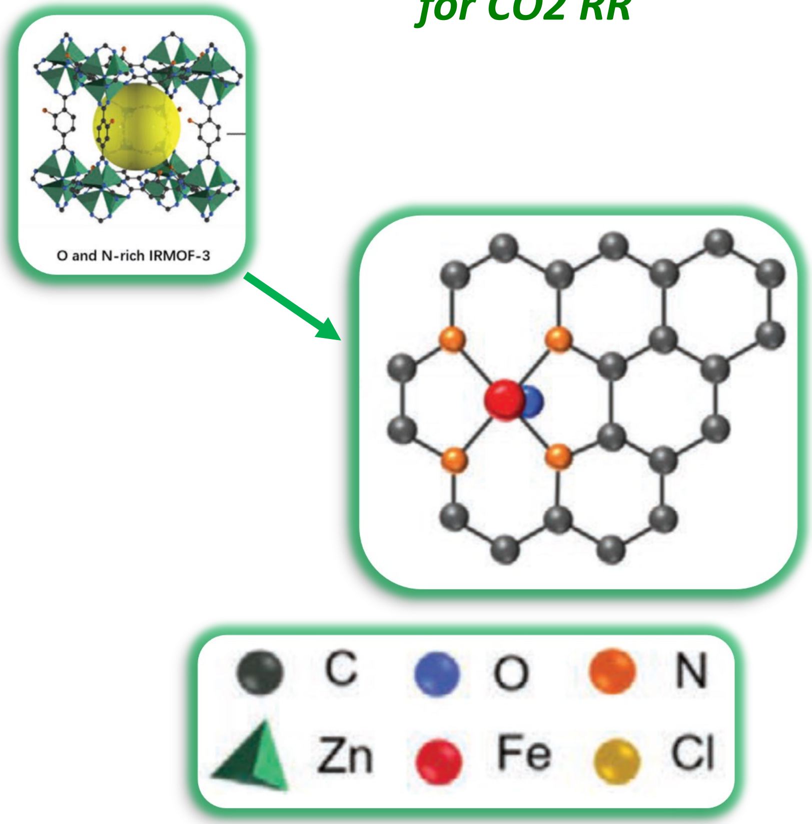
Adv. Funct. Mater., **31**, 2106349 (2021)

3. Surface Hydroxylation for CO2 RR



ACS Appl. Mater. Interfaces, **DOI:** 10.1021/acsami.2c09129 (2022)

4. Axial Oxygen Coordinated FeN4 for CO2 RR

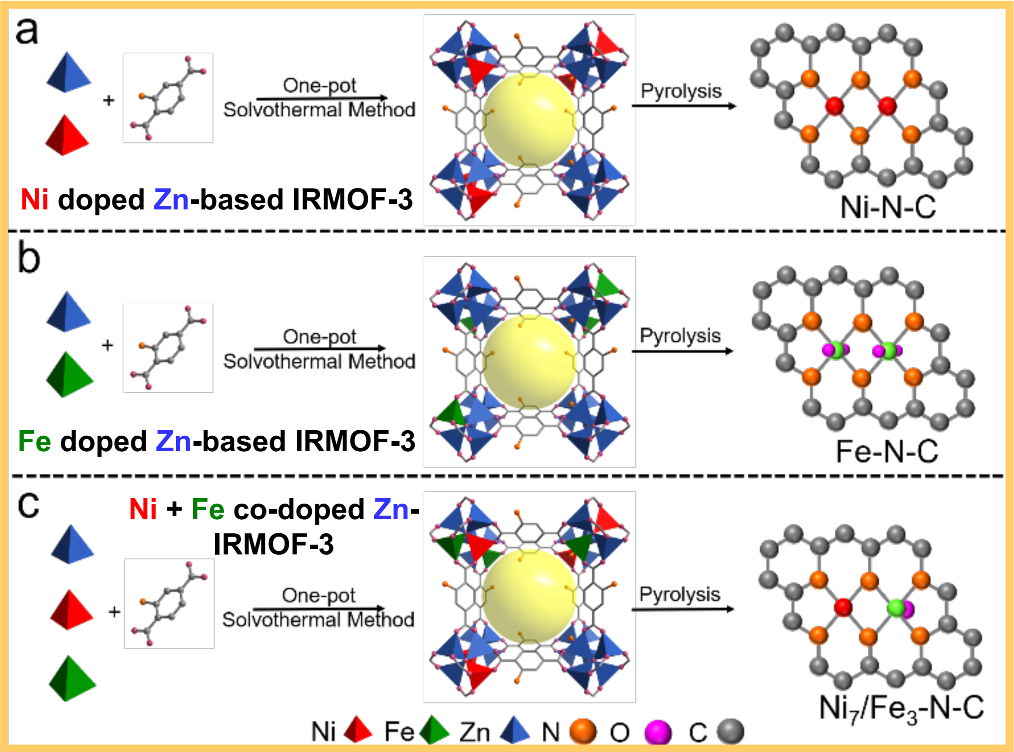


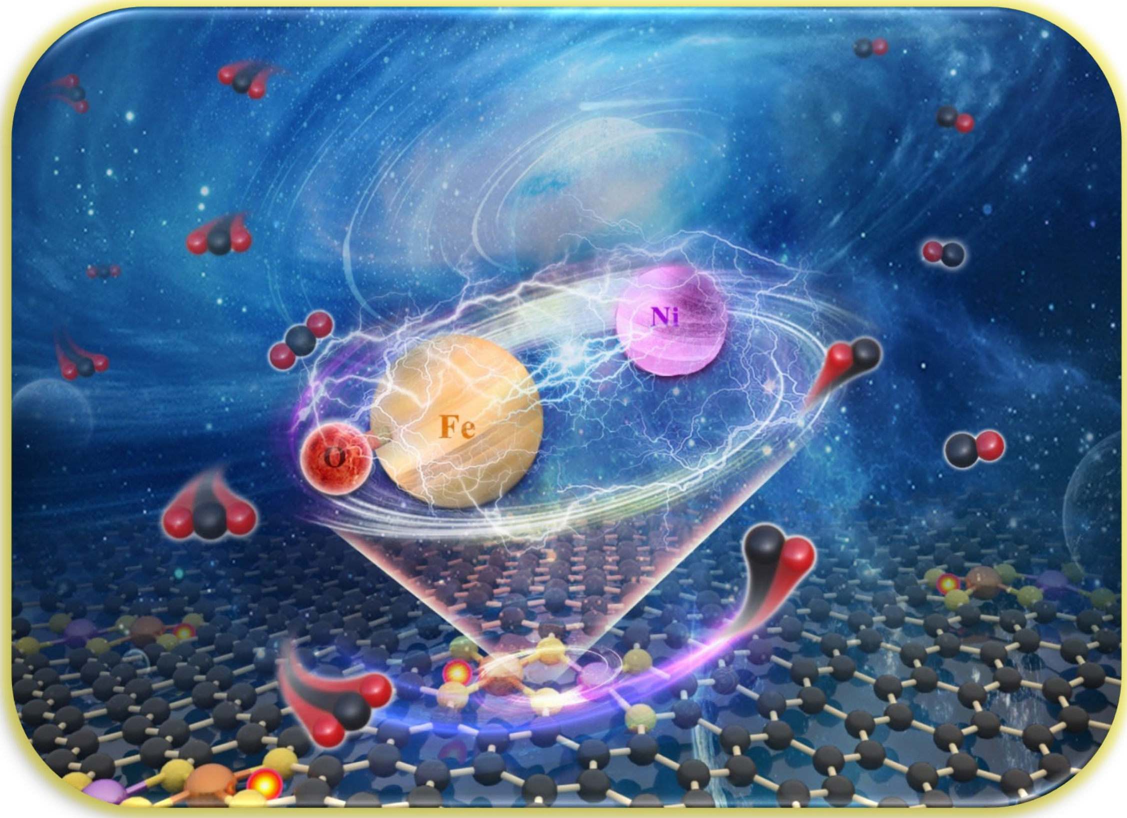
Adv. Funct. Mater., **32**, 2111446 (2022)

1. Ni-Fe SACs for CO2 RR

Quasi-Double-Star Nickel and Iron Active Sites for High-Efficient Carbon Dioxide Electroreduction.

With: IREC
Institut de Recerca en Energia de Catalunya
Catalonia Institute for Energy Research

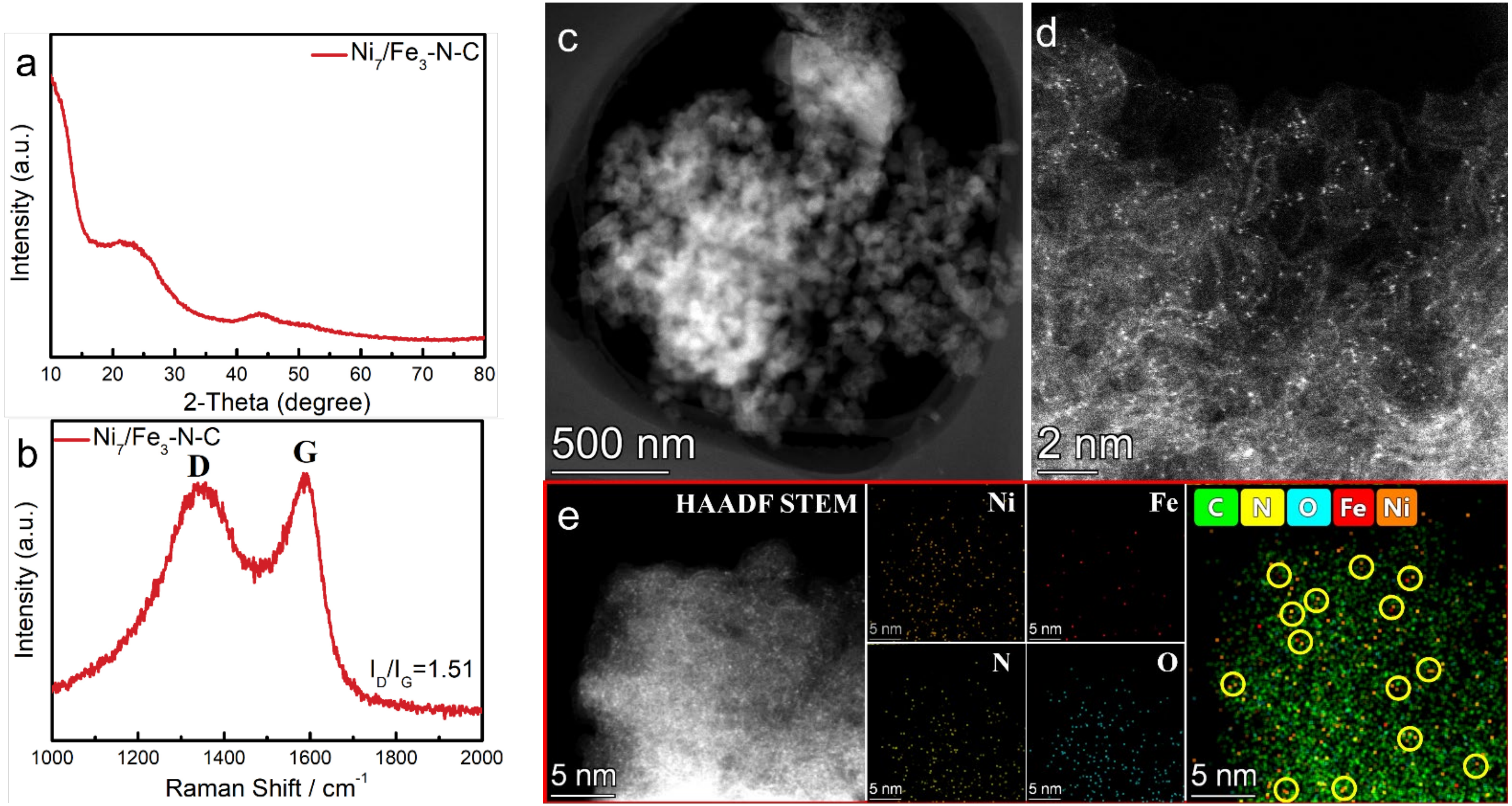




Schematic illustration of the preparation process of (a) Ni-N-C, (b) Fe-N-C and (c) Ni7/Fe3-N-C samples.

Energy Environ. Science, **14**, 4847 (2021)

1. Ni-Fe SACs for CO2 RR

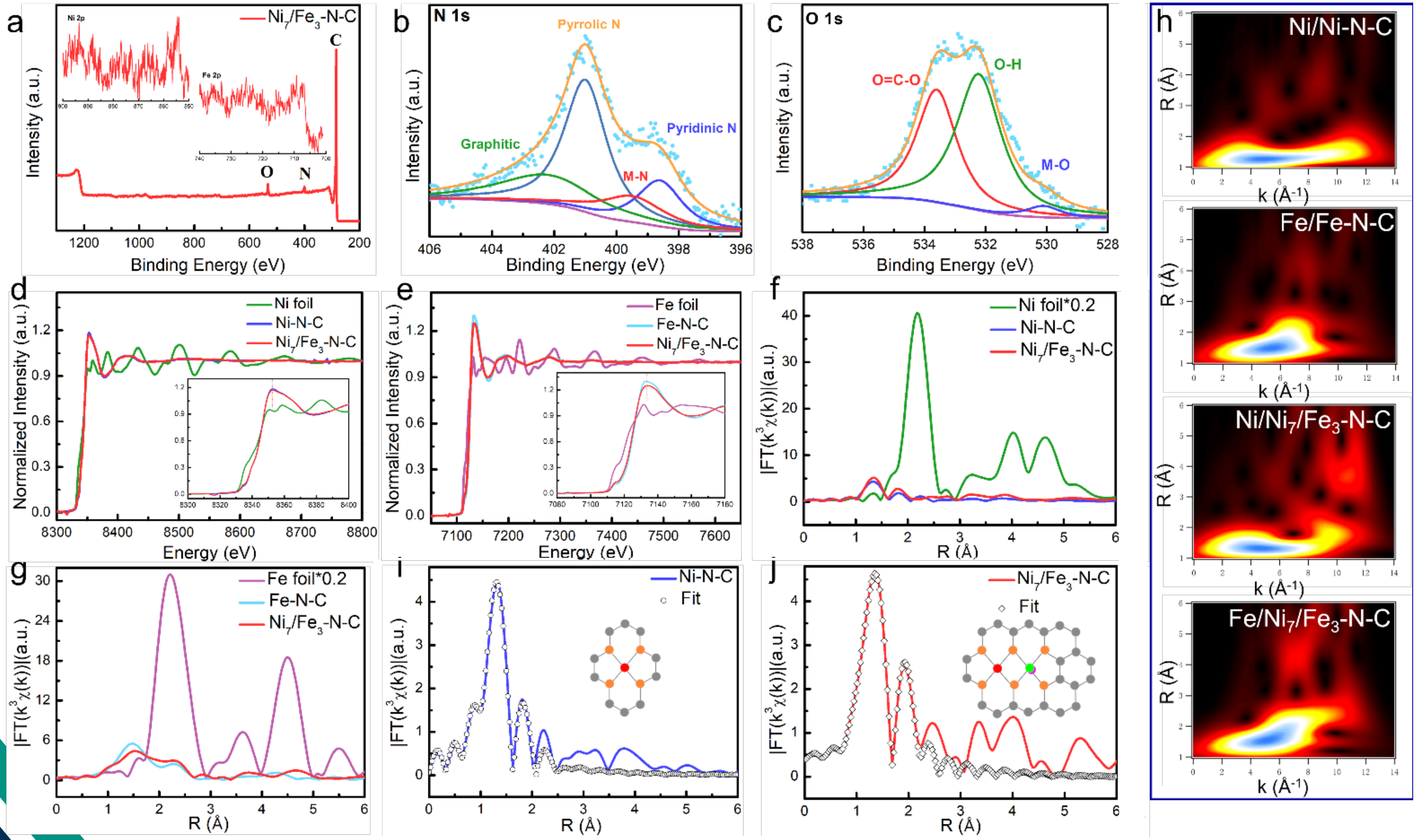


Ni_7/Fe_3-N-C Sample Characterization: (a) XRD patterns; (b) Raman spectra; (c) HAADF STEM (d) Atomic res. HAADF STEM images; (e) HAADF STEM image and EDS

With: IREC — Institut de Recerca en Energia de Catalunya, Catalonia Institute for Energy Research

Energy Environ. Science, **14**, 4847 (2021)

1. Ni-Fe SACs for CO2 RR



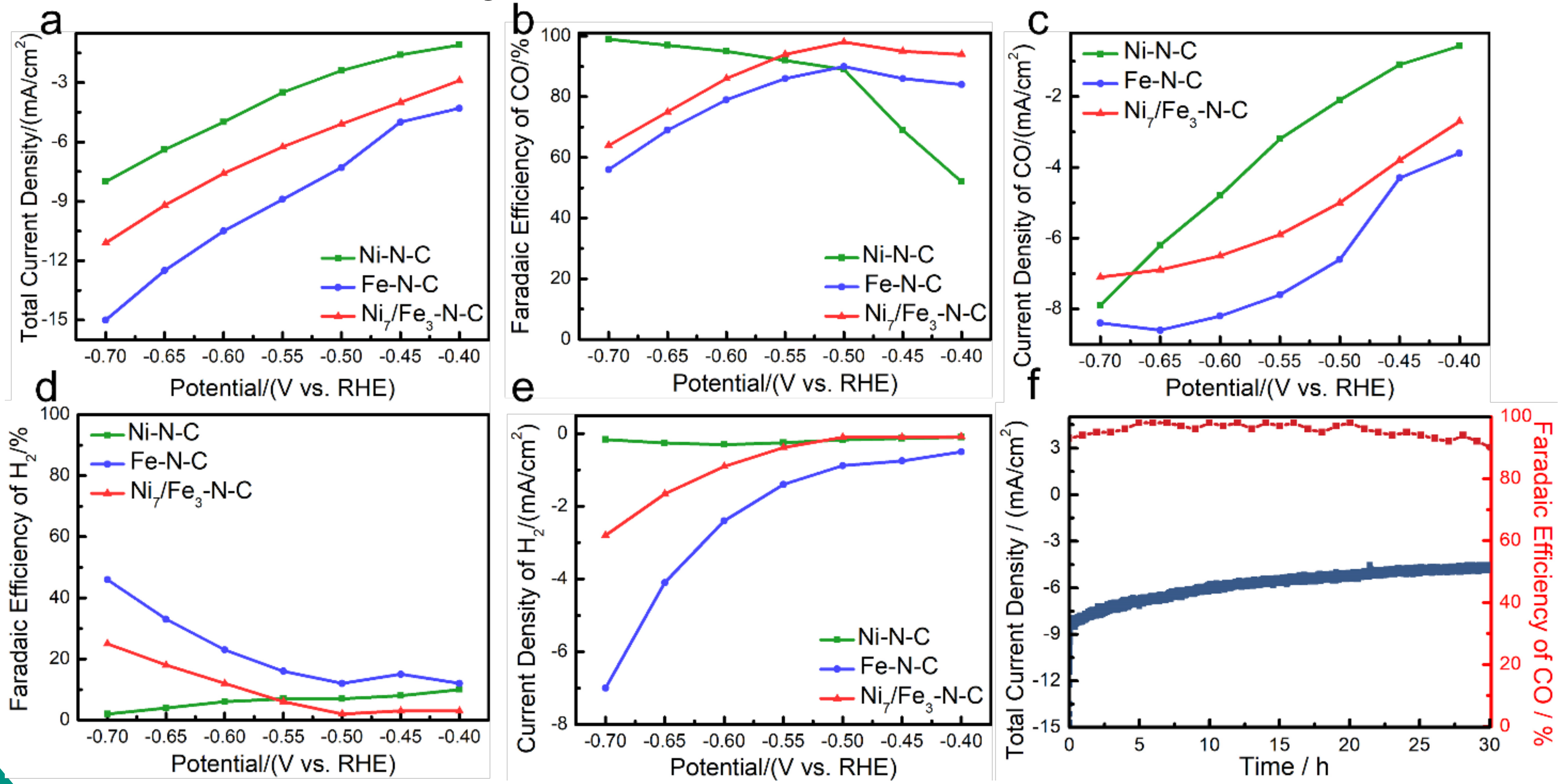
(a) XPS spectra
(b) HR XPS for N 1s
(c) HR XPS for O 1s
(d) XANES Ni K-edge
(e) XANES Fe K-edge
(f) EXAFS Ni K-edge
(g) EXAFS Fe K-edge
(h) WT-EXAFS

Ni K-edge EXAFS fitting:
(i) Ni-N-C
(j) Ni7/Fe3-N-C
(Ni, Fe, O, N, C atoms)

Energy Environ. Science, **14**, 4847 (2021)

1. Ni-Fe SACs for CO2 RR

Electrocatalytic Performance of CO_2 Reduction



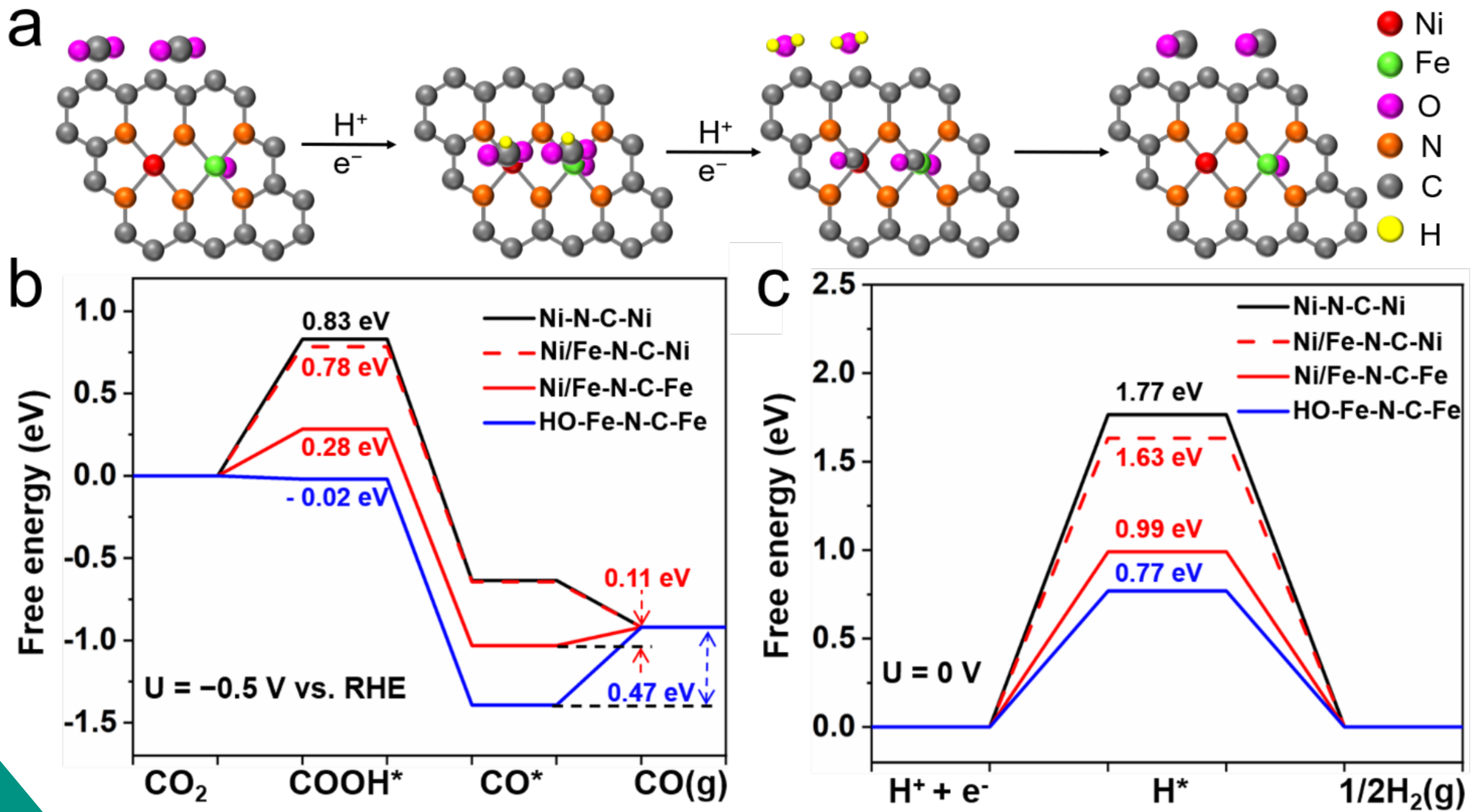
(a) Total current density
(b) FE of CO at various potentials
(c) Current density for CO production
(d) FE of H2 at various potentials
(e) Current density for H2 production
(f) Stability test of Ni7/Fe3-N-C at −0.50 V vs. RHE.

Maximum of 98% FE for CO production at −0.50 V (vs. RHE) with a low overpotential of just 390 mV

Energy Environ. Science, **14**, 4847 (2021)

1. Ni-Fe SACs for CO2 RR

DFT Studies of CO2 RR



(a) Supposed pathway of CO2 reduction to CO in DFT calculations

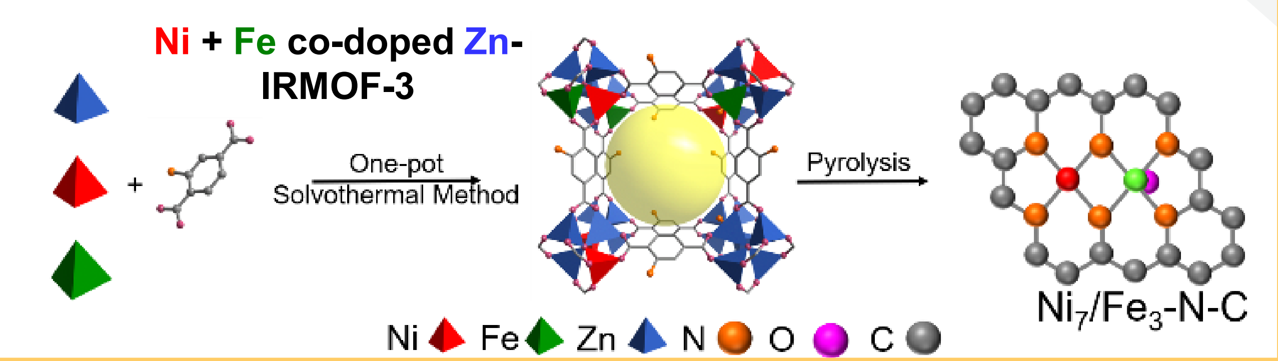
Free energy profiles for:

(b) CO2 RR to CO at −0.50 V (vs. RHE)

(c) HER at 0 V (vs. RHE) on simulated models

Energy Environ. Science, **14**, 4847 (2021)

1. Ni-Fe SACs for CO2 RR



The adjacent Ni and Fe active sites act as a nano-reactor, affecting different reaction steps in comparison to two separate active sites during CO2 RR, thus, enhancing the overall activity.

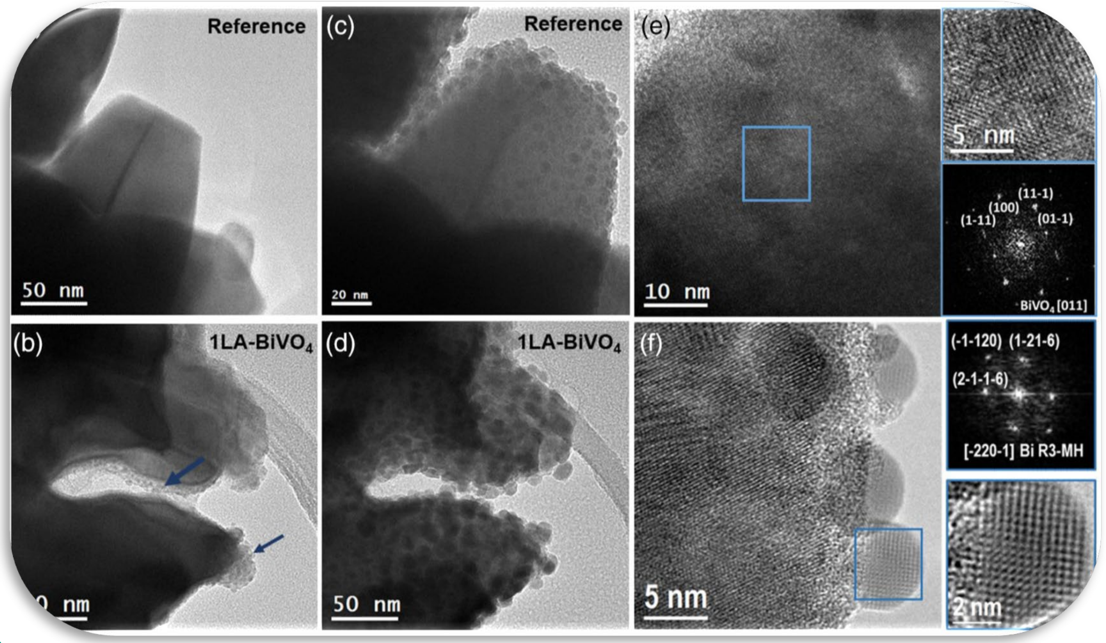
These bimetallic adjacent sites weaken the bonding energy of CO2 RR intermediates while limiting the competitive HER.

With: IREC Institut de Recerca en Energia de Catalunya Catalonia Institute for Energy Research

Energy Environ. Science, **14**, 4847 (2021)

5. BiVO4 photoanodes aging

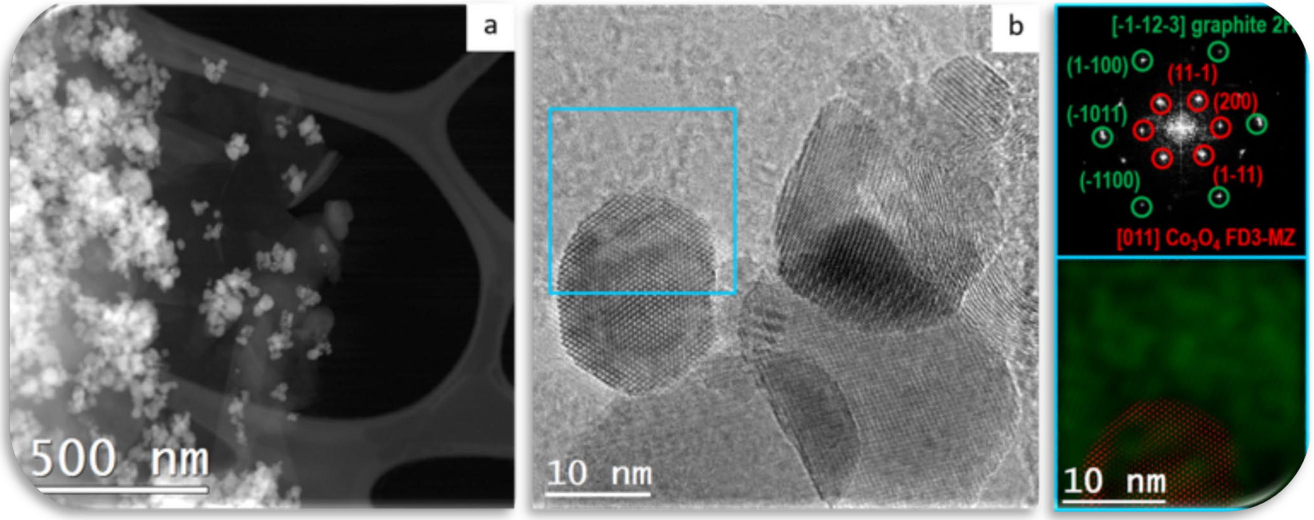
Direct observation of the chemical transformations in BiVO4 photoanodes upon prolonged light-aging treatments.



Solar RRL, **6**, 2200132 (2022)

With: UNIVERSITAT JAUME I

Sustainable oxygen evolution electrocatalysis in aqueous 1 M H_2SO_4 with earth abundant nanostructured Co_3O_4



Nature Commun., **13**, 4341 (2022)

With: ICIQ UNIVERSITAT JAUME I eman ta zabal zazu UPV EHU

Thanks for your attention!

CONTACT:
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See more at: ***www.gaen.cat***