

National Energy Technology Laboratory



SOFC Interconnect Work at NETL

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Office of Fossil Energy



RE Surface Treatment Results

- **Oxidation resistance of Crofer improved by 3x (800C/3% water/4,000h)**
 - Appears to minimize internal silica layer formation
- **Improved ASR (800C/500h)**
 - Button cell tests underway
- **Oxidation resistance of type 430 vastly improved**
 - Eliminated spall (800C/3% water/4,000h)
- **Effectively makes 12 Cr alloys perform as 22 Cr**



Improving Oxidation Resistance with Rare Earths

- **Well established that small amounts RE improve oxidation resistance (RE: Ce, La, Y)**
- **Melt addition**
 - + Elements added during ingot production (single manufacturing step)
 - Difficulty in melting (react with crucibles)
 - Surface concentration limited by solubility and diffusivity
- **Surface treatments**
 - + Rare Earth concentrated where needed (at surface and have most benefit)
 - “Extra” manufacturing step.
 - ? Long term effectiveness (as with any coating or surface treatment)



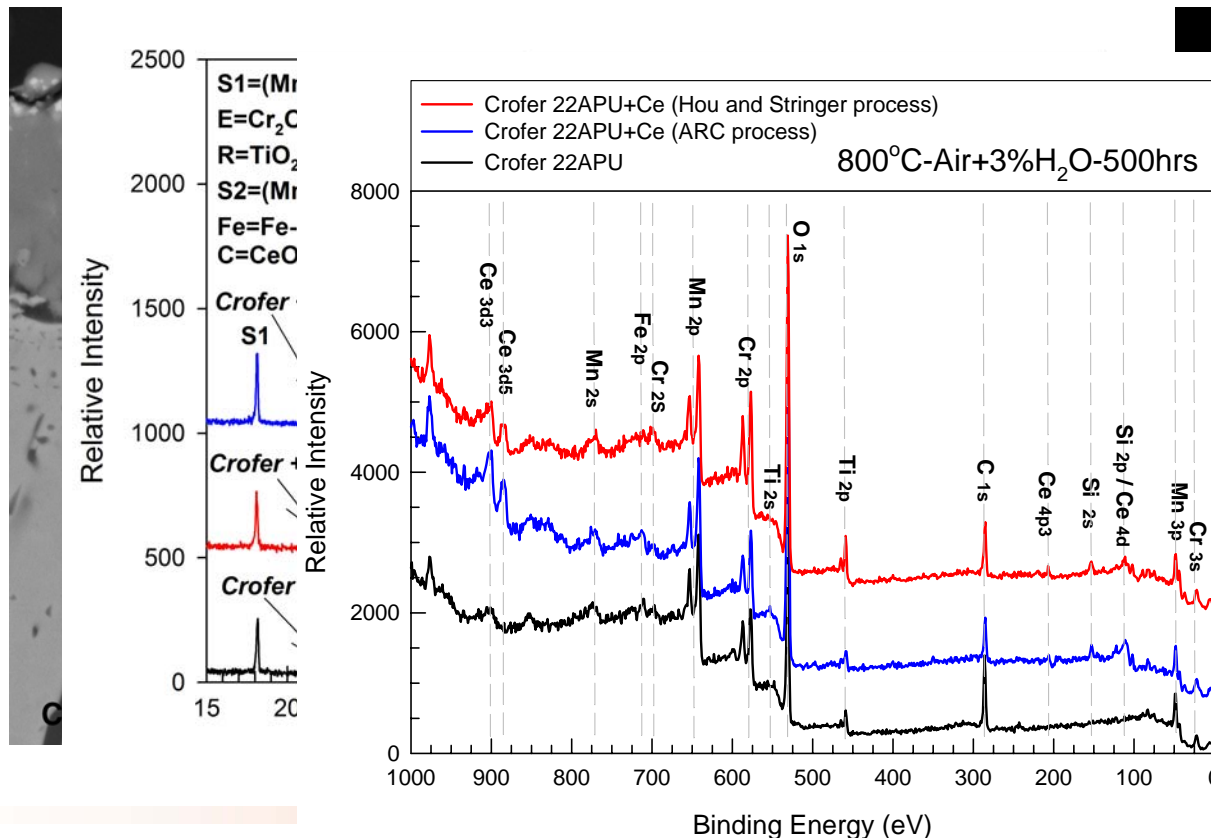
Research Goal

- Investigate rare earth surface treatment for improving oxidation resistance of alloys for SOFC applications.
- Two different surface treatments investigated
 - Developed at NETL
 - Similar to pack cementation: coated with a powder mixture containing CeO_2 or CeN and halide activator followed by heating in a controlled atmosphere (900°C -12 hrs), after which residual “pack” coating is washed off the surface.
 - Patent application filed with USPTO in September, 2005.
 - Applied to over 50 alloys of interest to FE.
 - Described in a paper by P.Y. Hou and J. Stringer (H/S)
 - J. Electrochem Soc., Vol 134, No. 7, July 1987, pp. 1836-1849
 - Coupons heated to 200°C were coated with a cerium-nitrate slurry (10w/o nitrate adjusted with HNO_3 to $\text{pH}=2$), followed heating in air at 400°C to decompose to CeO_2

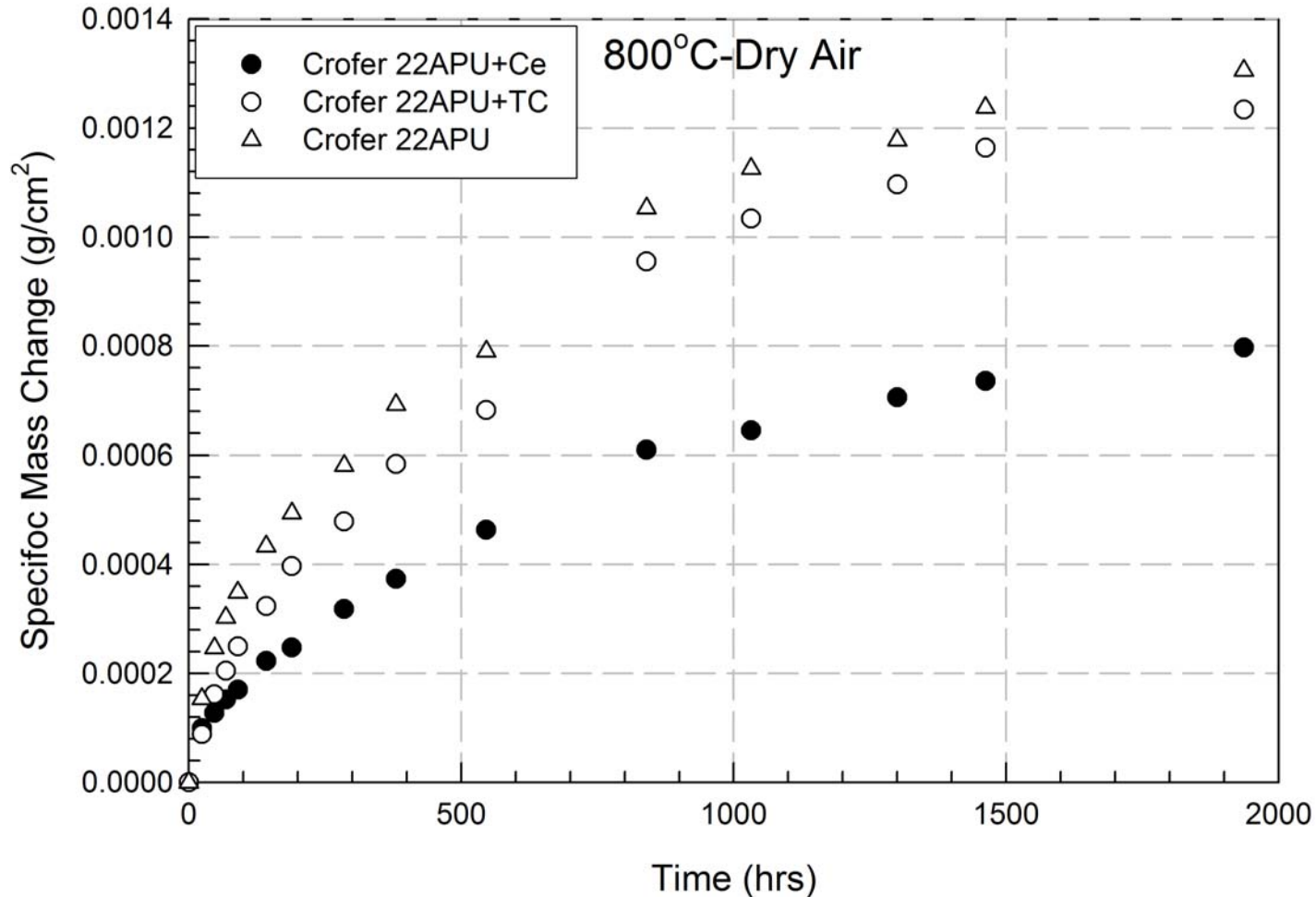


Surface Treatments to Improve Oxidation

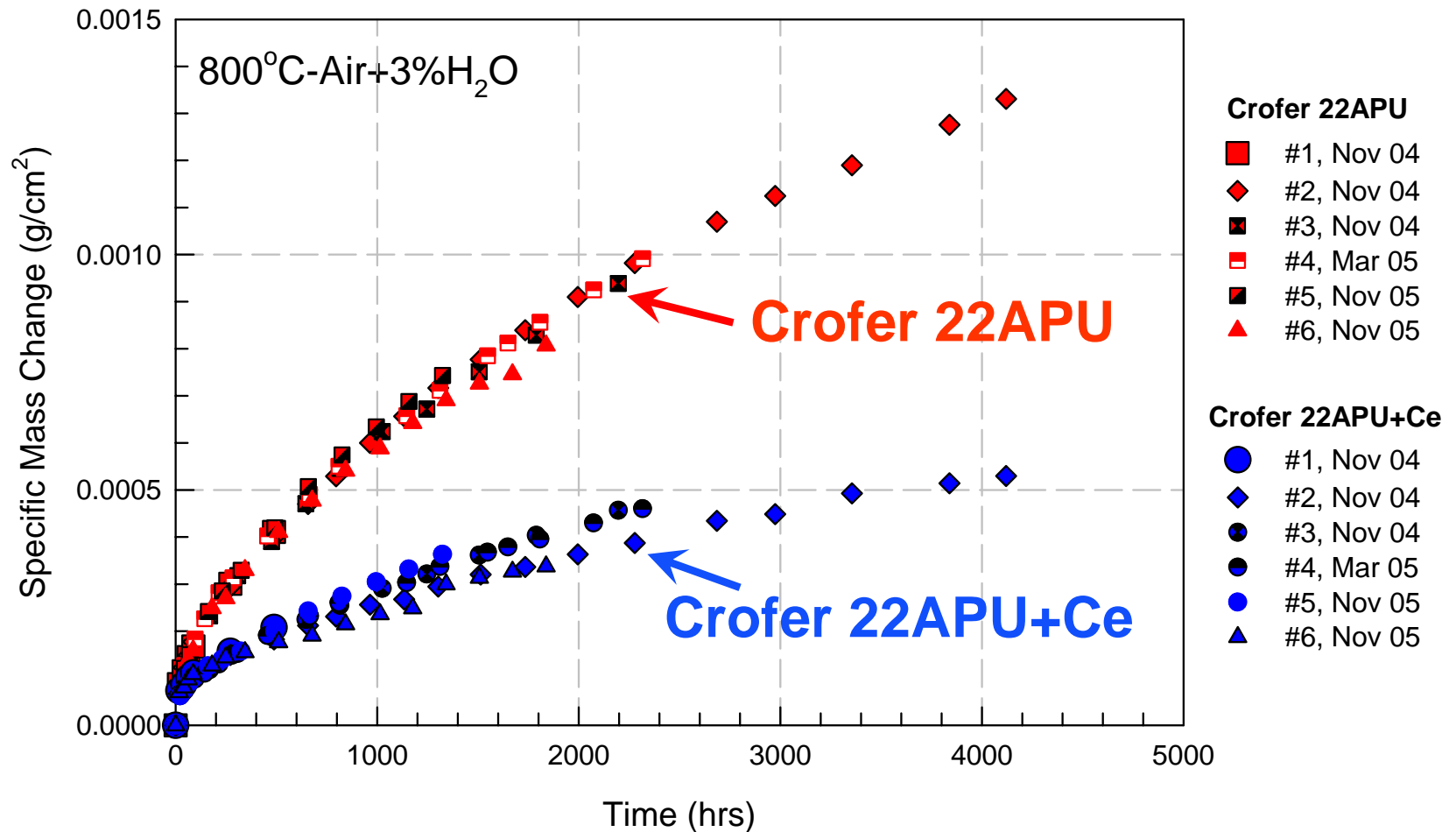
- As part of NETL-Albany's SOFC research effort, we have utilized surface treatments to incorporate reactive elements (Ce, La, Pr) into the surface of metallic alloys for development of metallic interconnects.



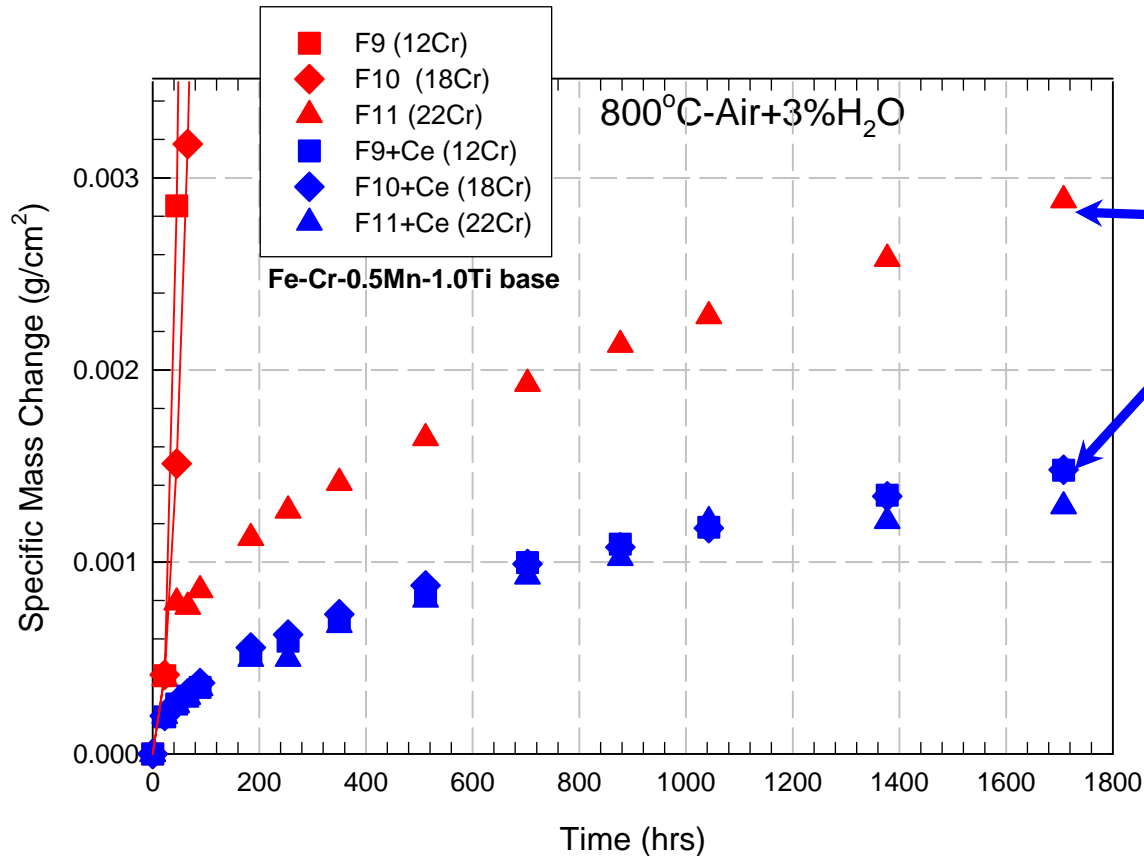
Thermal Treatment (TC) Only is *Ineffective*



Repeatable Behavior

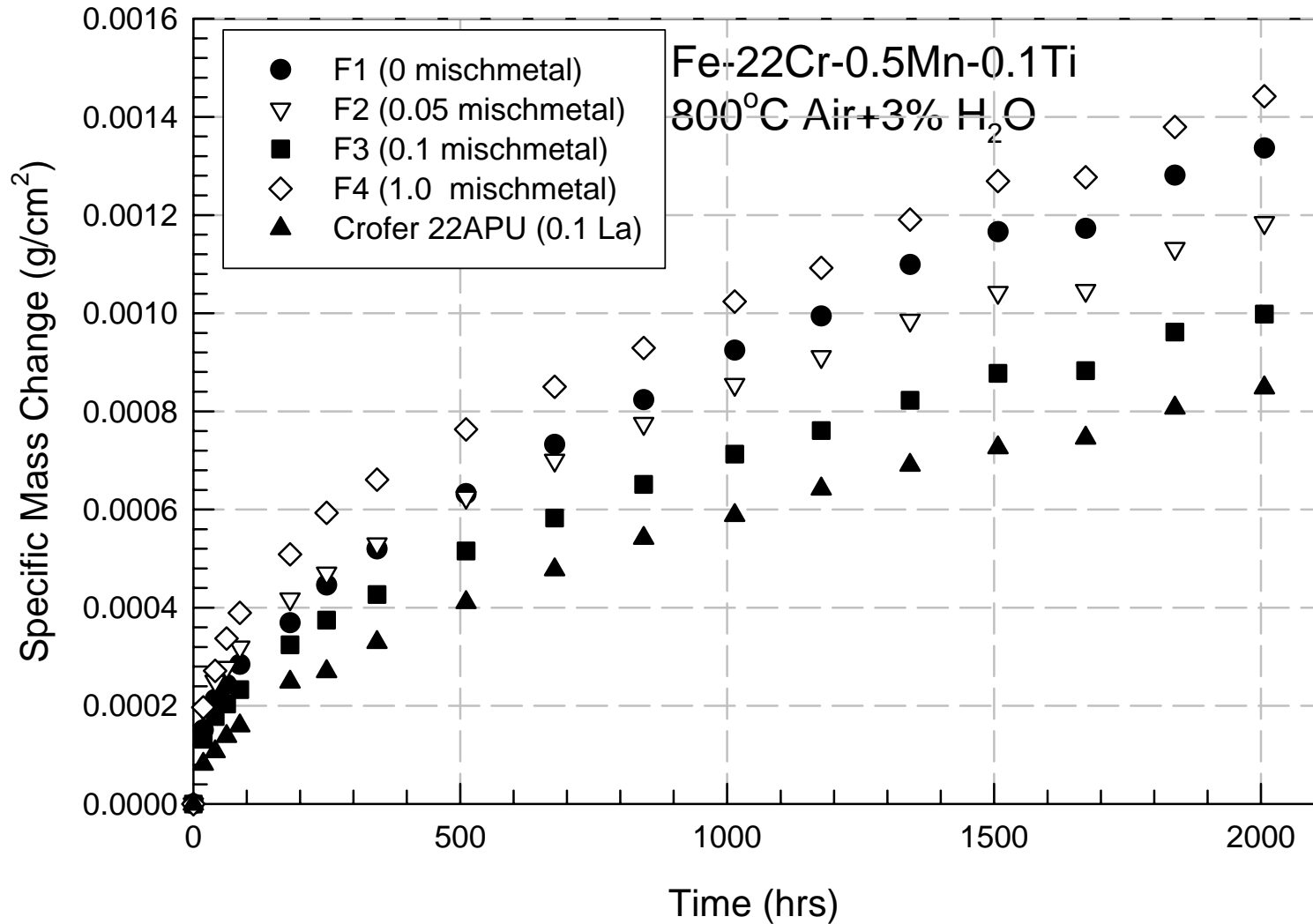


Effect of Ce Treatment: Fe-Ce-0.5Mn-1Ti

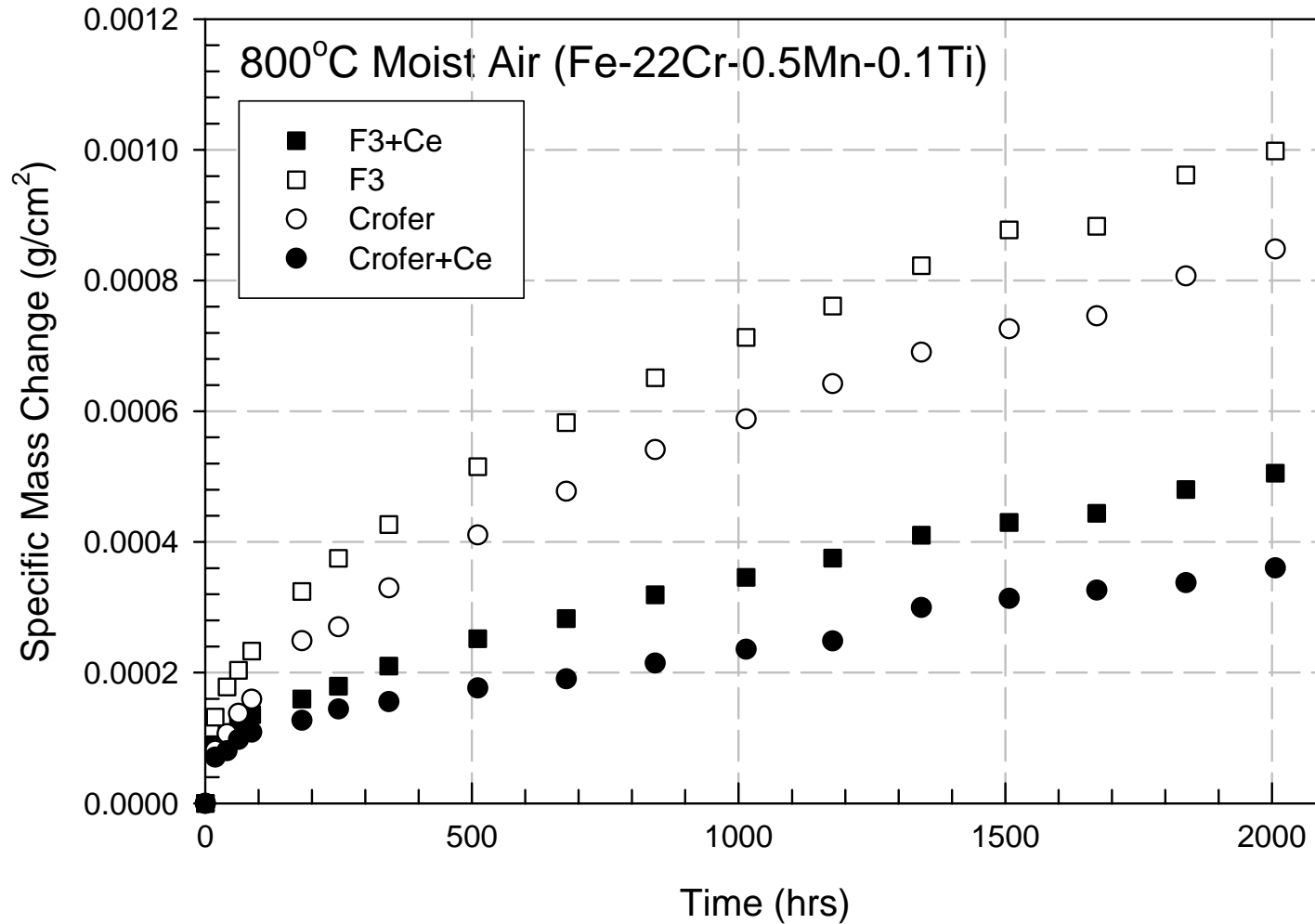


Fe-12Cr + Ce alloy
superior
performance to
an Fe-22Cr alloy

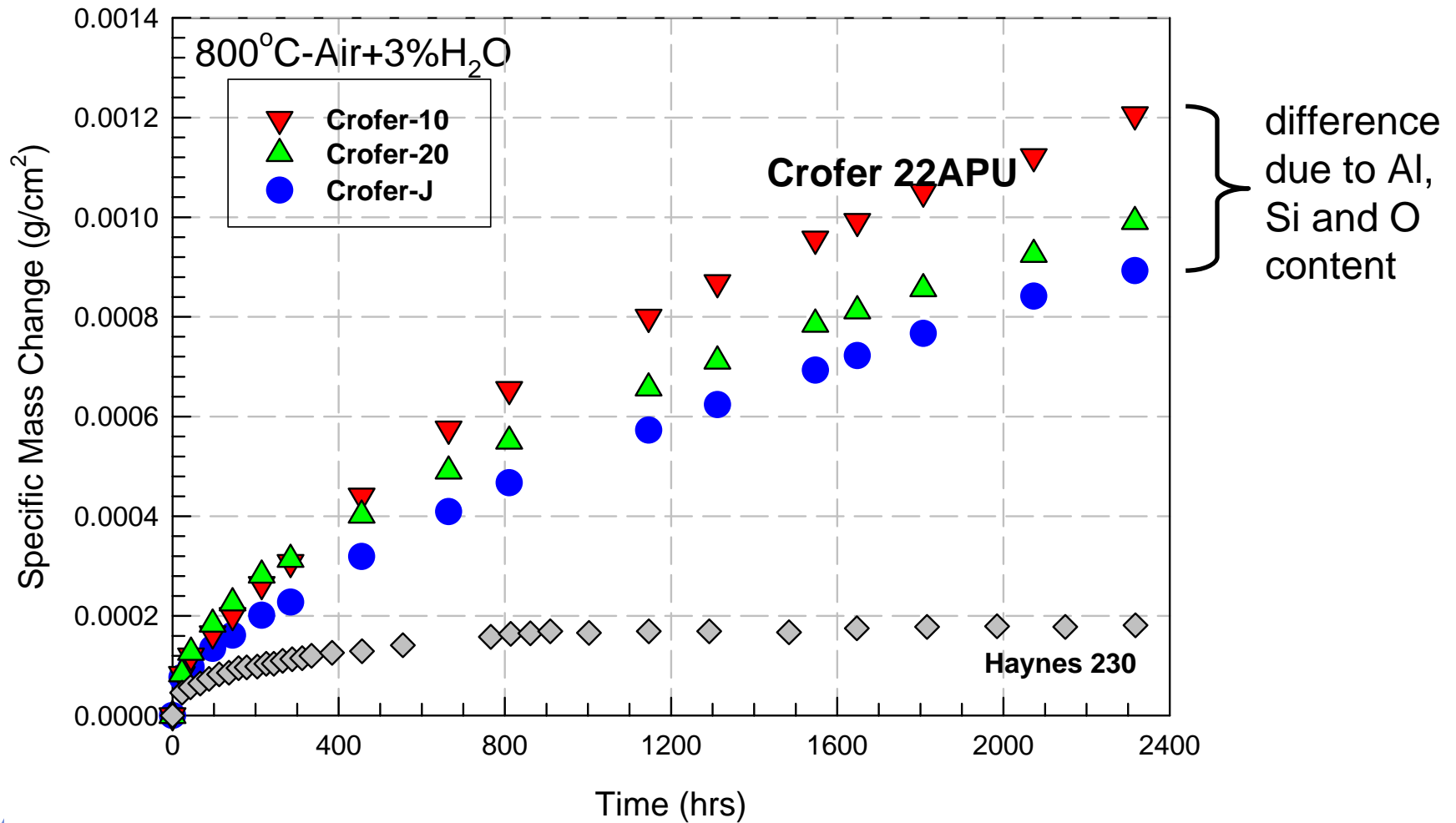
In Melt Additions of RE



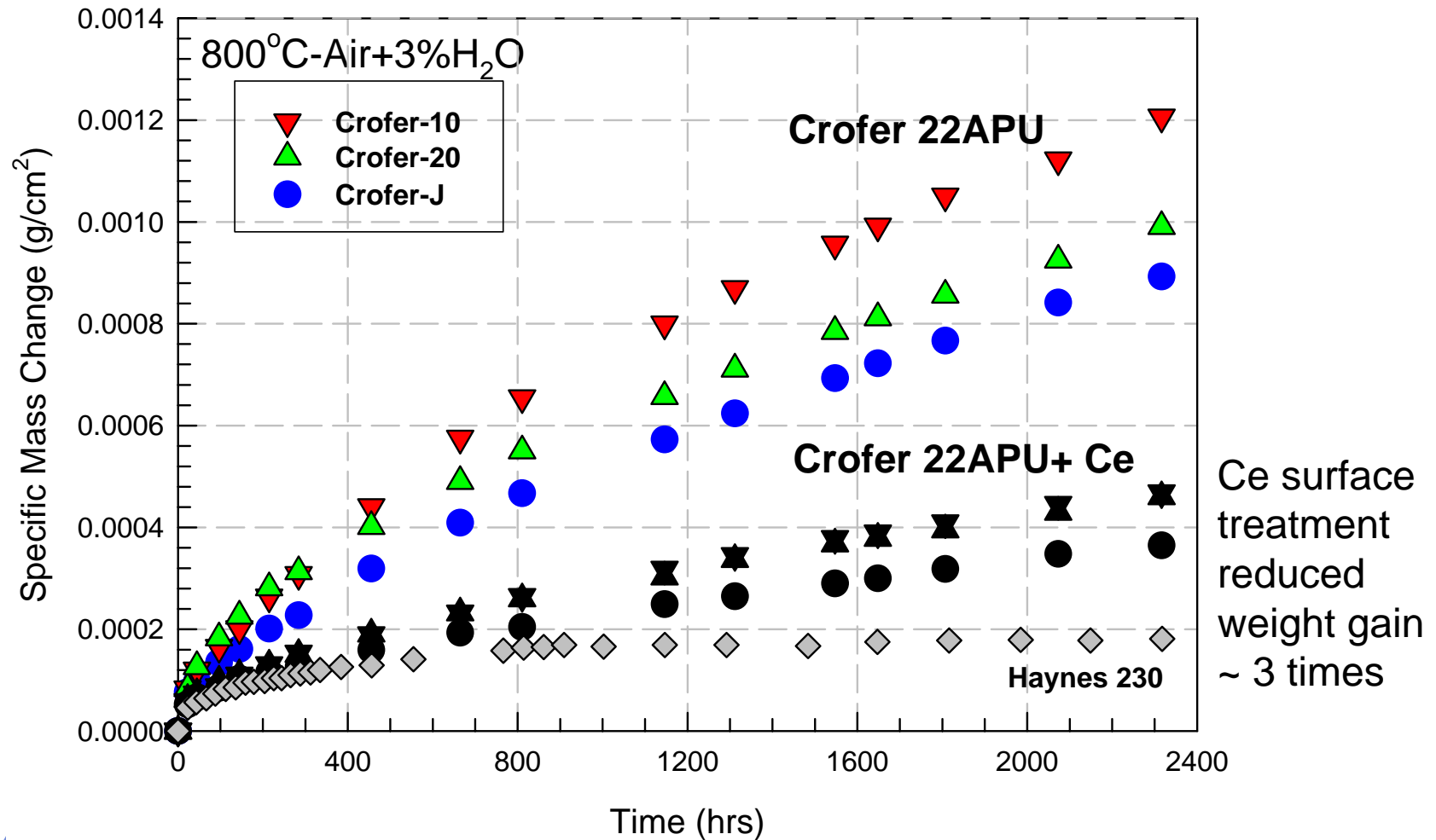
In Melt + Surface Treatment



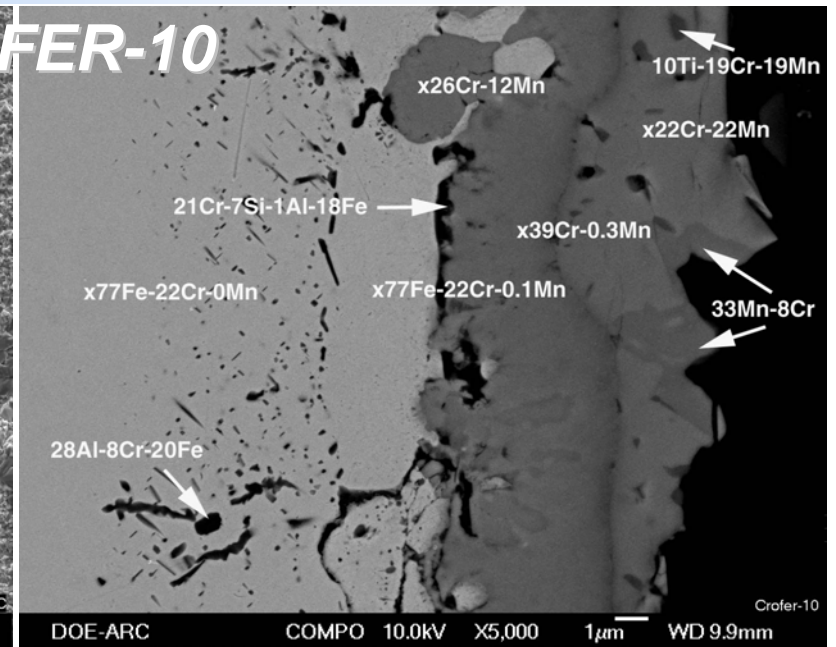
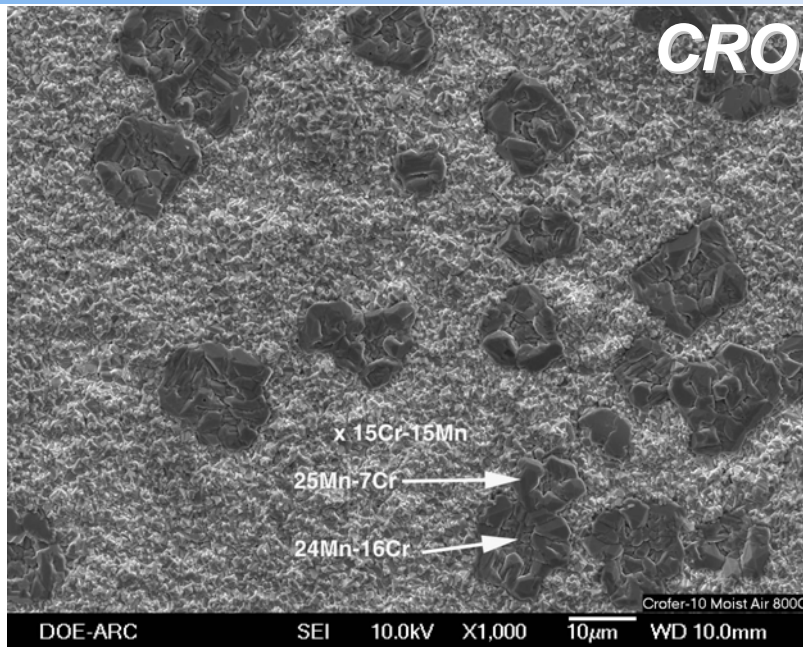
Minor Element Effect



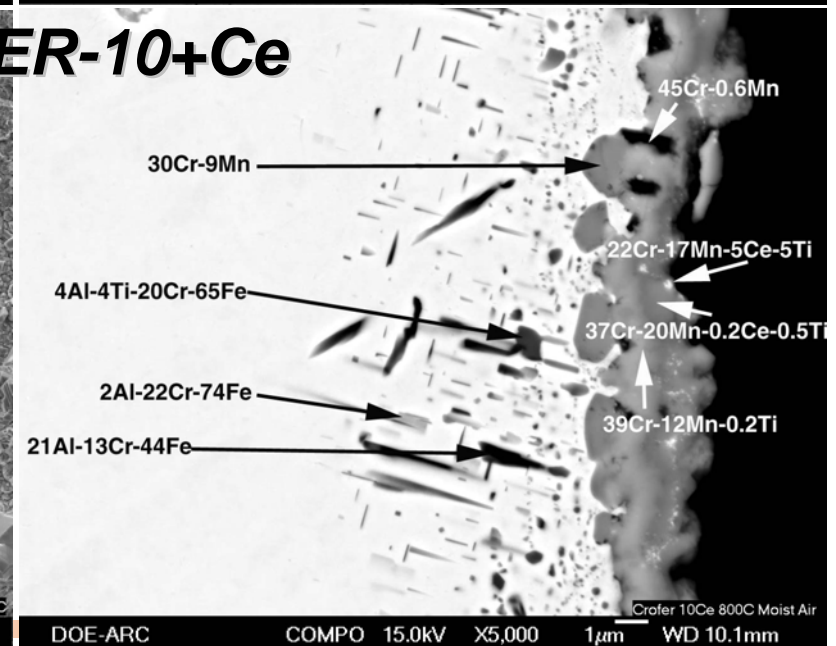
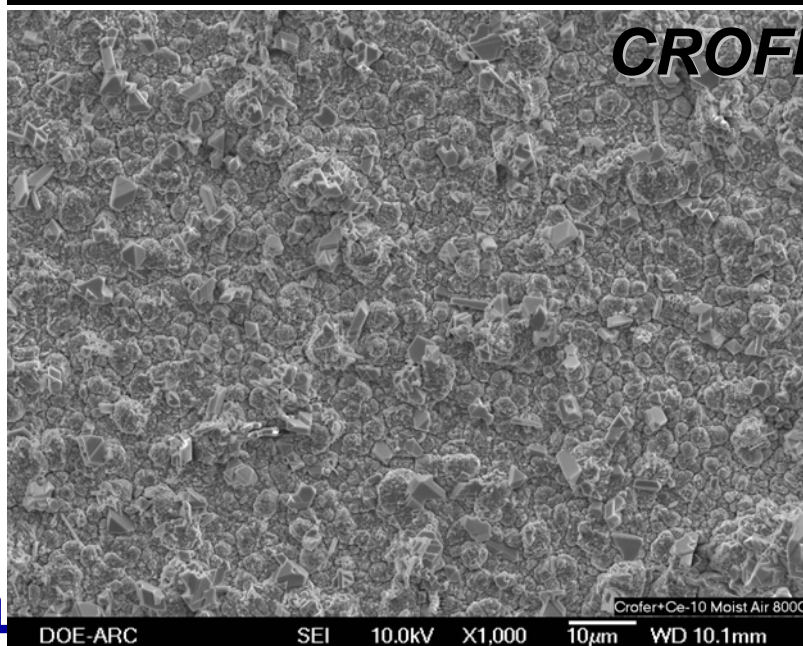
Ce Surface Treatment on Crofer 22APU



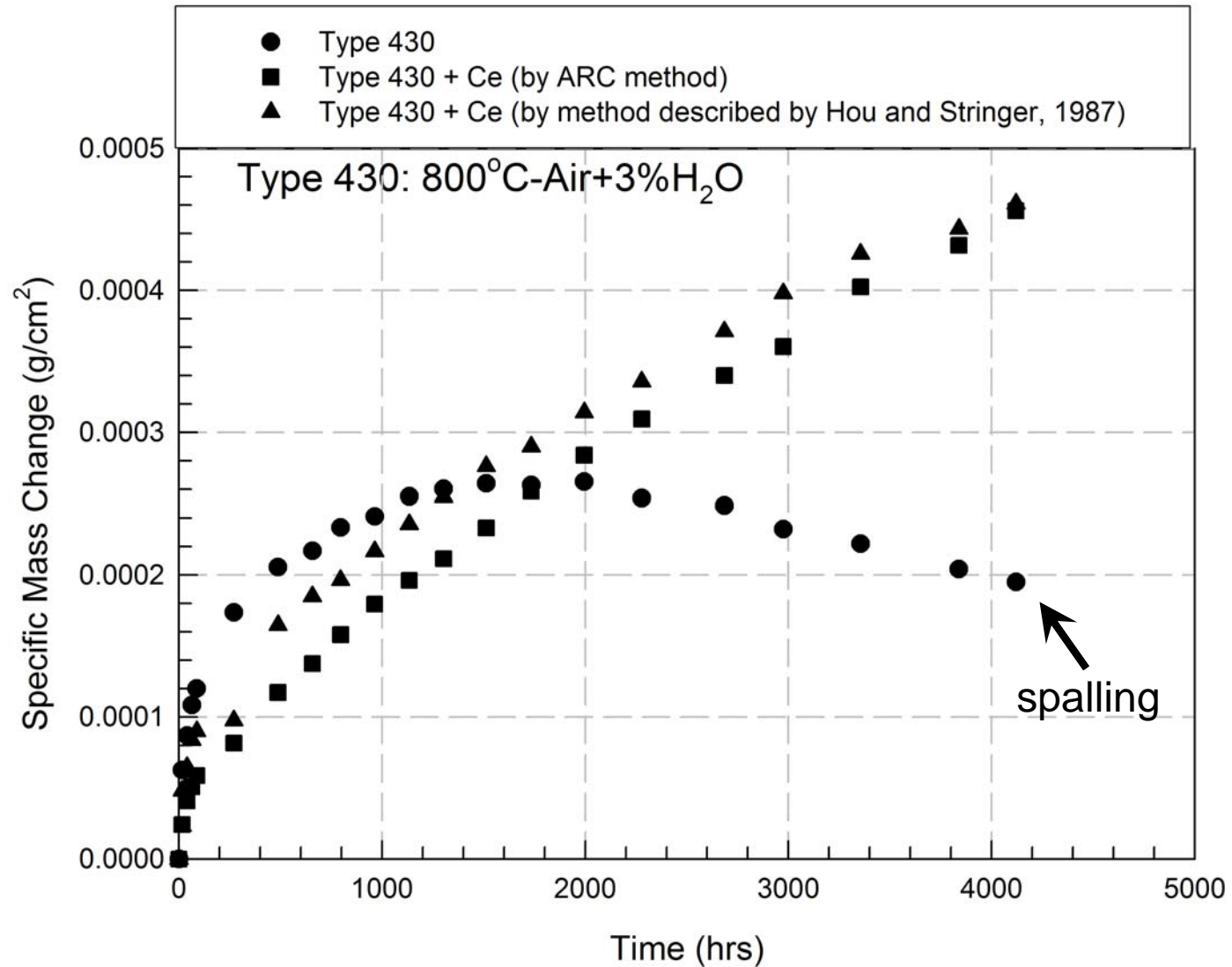
CROFER-10



CROFER-10+Ce

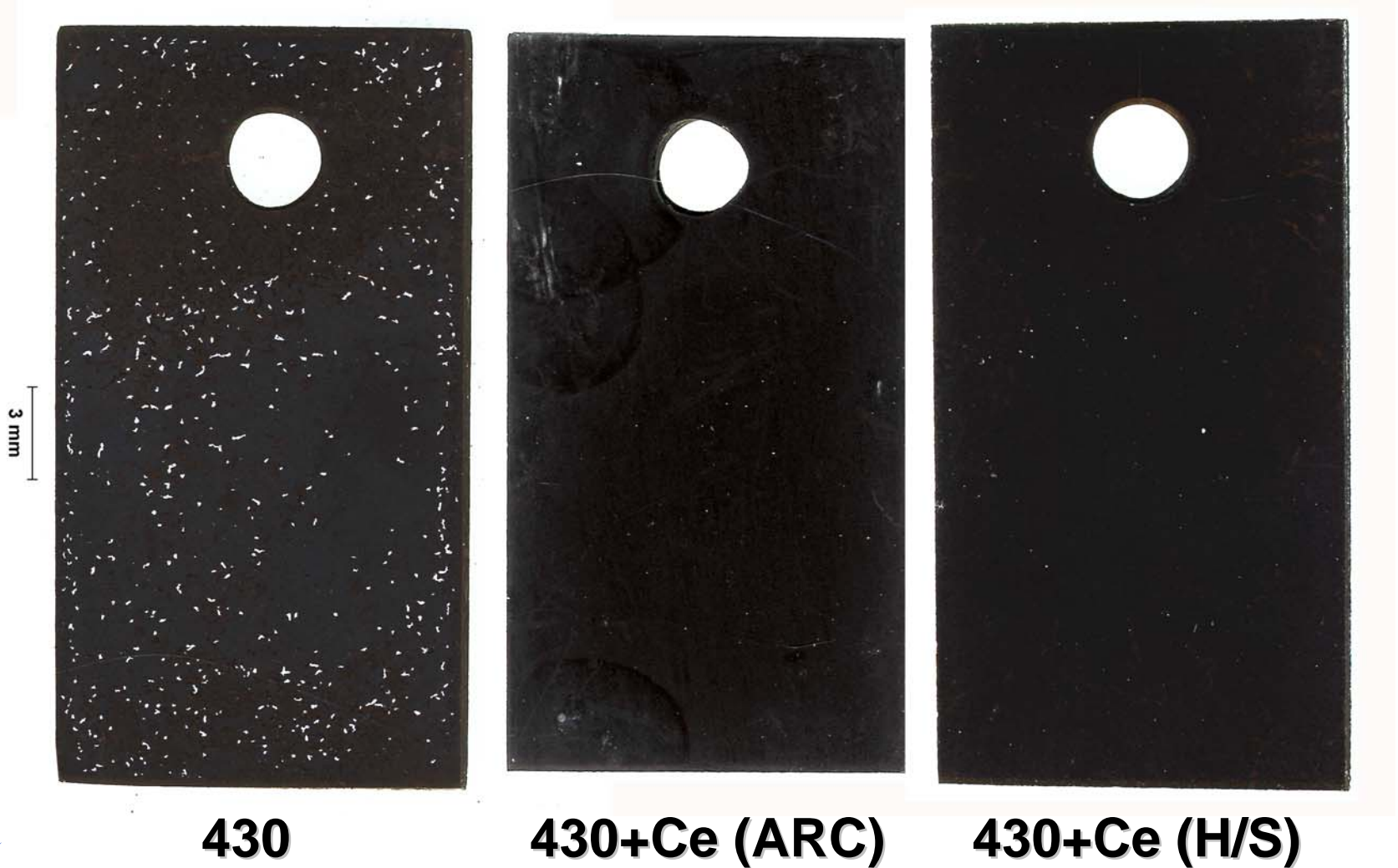


TYPE-430



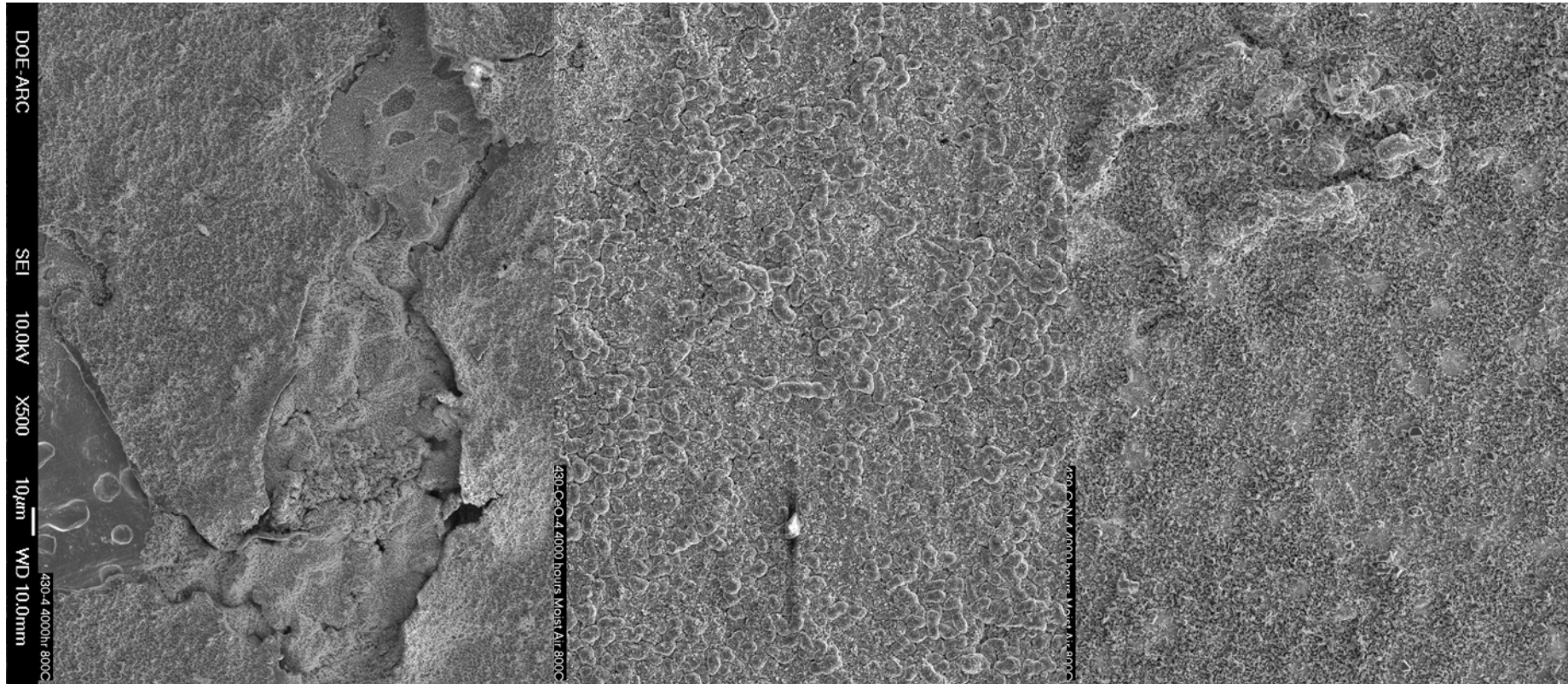
Surface: Type-430

800°C-4000hrs-Air+3%H₂O



Surface: Type-430

800°C-4000hrs-Air+3%H₂O



Type 430

Type 430 + CeO
(NETL)

Type 430 + CeO
(H/S)

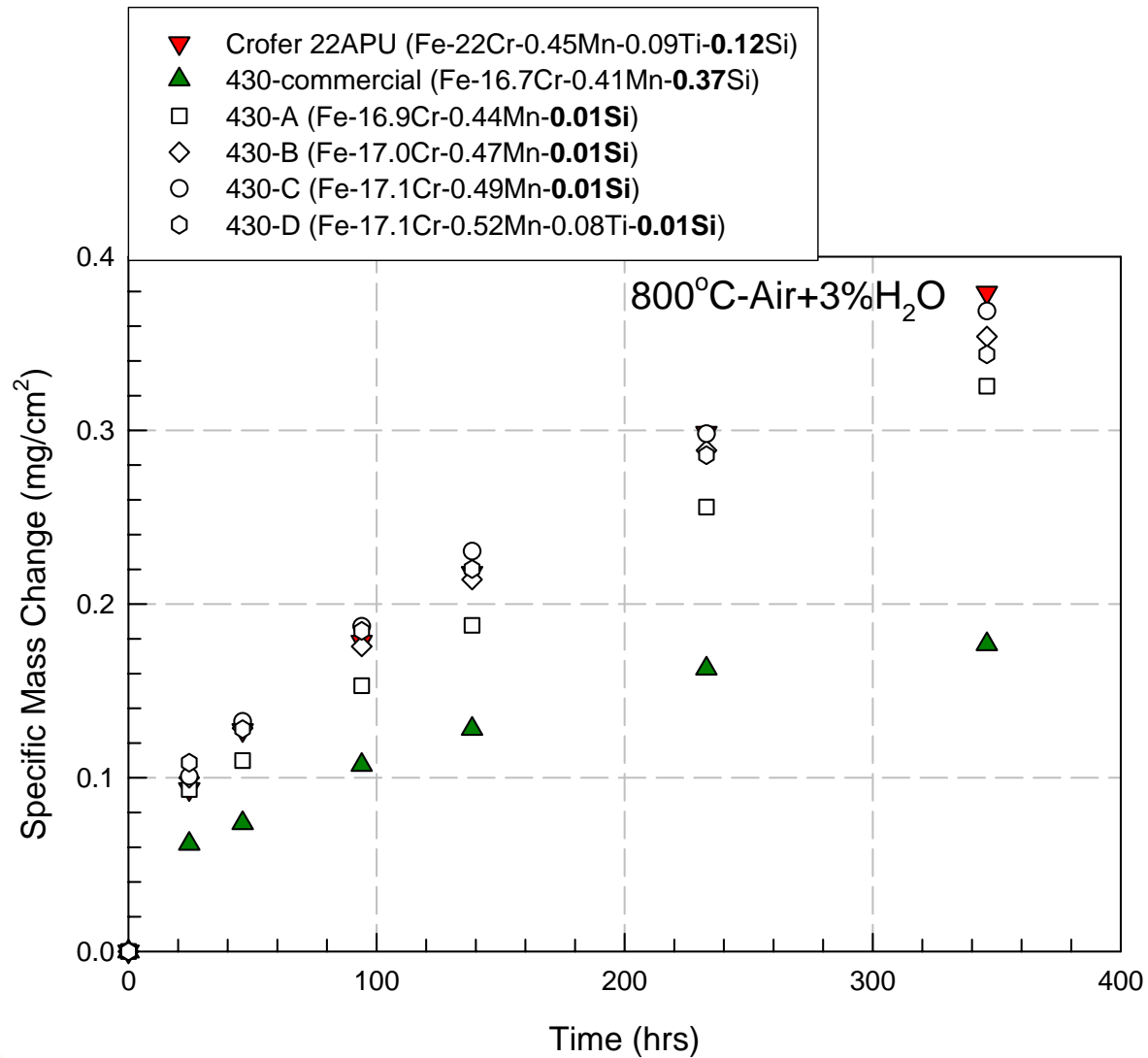
Stainless Steel Alloys

	430-A	430-B	430-C	430-D	430-std	Crofer22APU
Fe	Bal	Bal	Bal	Bal	Bal	Bal
Cr	16.85	17.03	17.13	17.11	16.6	22.42
Ti	<0.001	<0.001	<0.001	0.080	<0.001	0.092
Al	<0.001	<0.001	<0.001	<0.001	0.016	0.13
Si	<0.01	<0.01	<0.01	<0.01	0.37	0.12
Mn	0.44	0.47	0.49	0.52	0.42	0.45
C	0.005	0.05	0.1	0.01	0.06	0.02

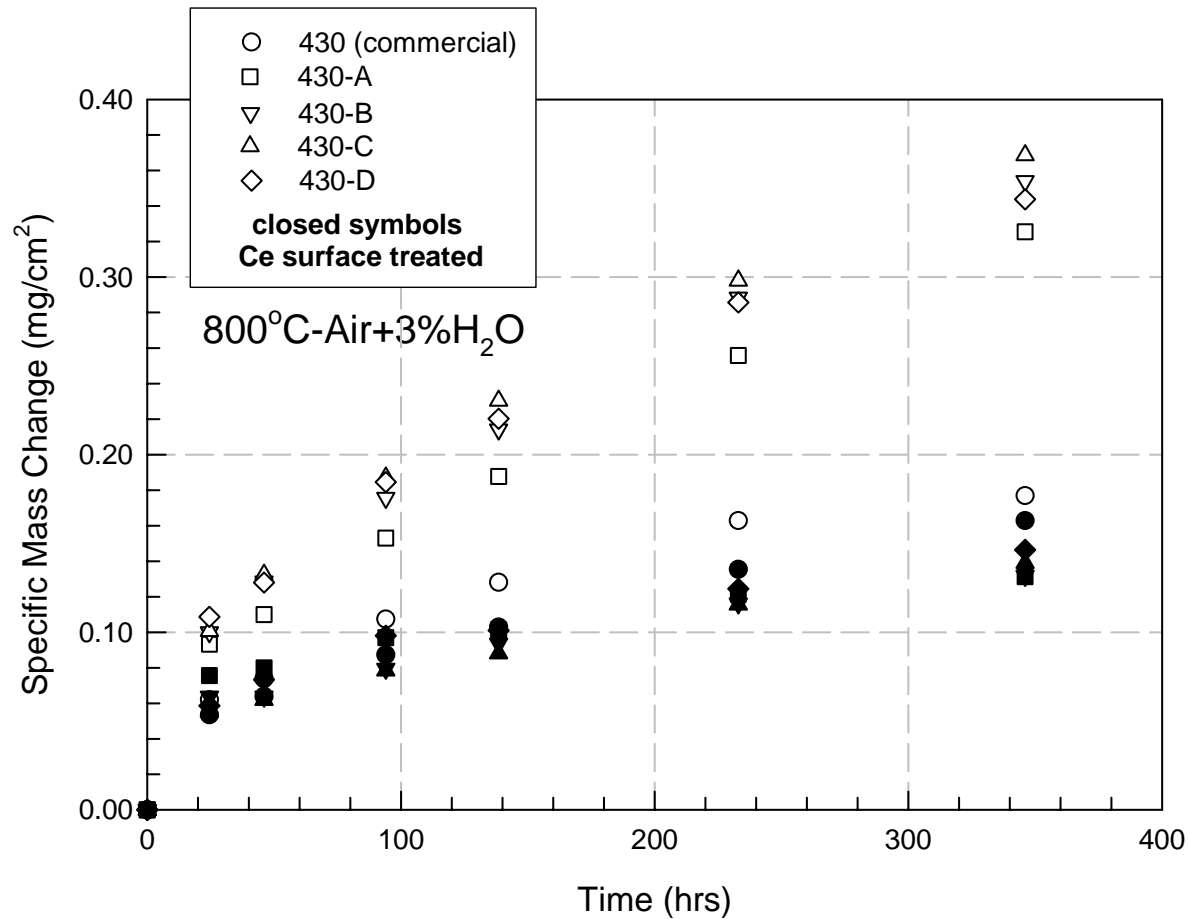
Alloys 430A-D were melted and fabricated at NETL



Low Si 430 Alloys



Surface Treated Low Si 430 Alloys



Summary

- Oxidation results of a simple surface treatment have been presented.
- The Cerium surface treatment improves the oxidation performance of type 430.
- The Cerium surface treatment improves the oxidation performance of RE containing alloys such as Crofer 22 APU.
- Improved ASR results have been achieved with RE treated Crofer 22 APU.

Acknowledgements

- **Gordon Xia and Z. Gary Yang from PNNL for performing the ASR measurements.**
- **Chris Johnson from NETL-Morgantown for assistance with initial SOFC button cell testing.**
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- **Ed Argetsinger, Marisa Arnold, Richard Chinn, Keith Collins, Paul Danielson, Dan Davis, Neal Duttlinger, Mike Hayes, Al Hunt, Steve Matthes, and Dave Smith from NETL-Albany for assistance with experiments.**
- **Margaret Zoimek-Moroz, Tom Adler, Omer Dogan, Gordon Holcomb, Karol Schrems, Rick Wilson, John Dunning and Paul King from NETL-Albany for contributions to the SOFC effort at Albany.**



Experimental Alloys and RE Surface Treated Materials Available For Evaluation by SECA Participants



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