High Throughput, In-line Coating Metrology Development for Solid Oxide Fuel Cell (SOFC) Manufacturing

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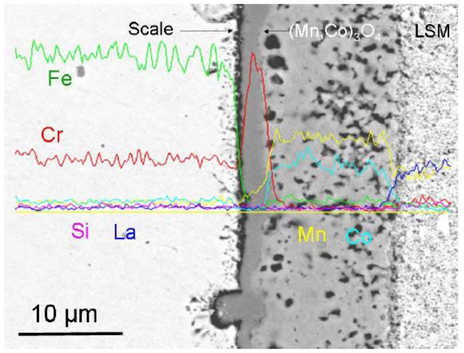
Introduction

Coating and interconnect cross-section



PNNL report ID: PNNL- 17568, May 2008

Protective coating on interconnect:

- Prevent electrode Cr poisoning: Barrier to Cr transport
- Prevent interconnect oxidation: Barrier of oxygen migration

$(Mn,Co)O_4$ (MCO) is a commonly used barrier coating layer

Defects in coating inhibit coating and SOFC performance

→ Need manufacturing-scale defect detection techniques

Metrology techniques state-of-the-art

	Technique	Measured parameter	Automation for interconnect	Speed for large area scan	Non-destructive
Most common	Tape peel test	Film adhesion	Yes	Fast	Yes
	Mass	Film thickness	Yes	Fast	Yes
	Scratch test	Film adhesion	Yes	Slow	No
	SEM/EDS/TEM	Cracks, pores, film uniformity, subsurface defects /composition	No	Slow	No
	XRF	Composition	Yes	Slow-Medium	Yes
	Indentation	Mechanical properties	Yes	Slow	Possibly
	Ellipsometry	Film thickness	No, requires uniform substrate	Fast	Yes
	X-ray tomography	Microstructure	Yes	Slow	Yes
	X-ray diffraction	Composition	Yes	Slow	Yes
	Raman Spectroscopy	Local atomic arrangement	Yes	Slow	Yes

Project Goals and Approach

- *Identify key interconnect coating and substrate defects that lead to coating failure;*
- *Assess capabilities of in-line metrology techniques, e.g., optical profilometry (Redox) and thermography (NREL), to probe defects;*
- *Demonstrate improved long-term performance of SOFC stacks*

Technique	Measured parameter	Automation for interconnect	Speed for large area scan	Non-destructive
Optical Profilometry	Cracks, pores, film uniformity	Yes	Fast	Yes
Optical Reflectance	Cracks, pores, film uniformity	Yes	Fast	Yes
Thermography	Cracks, pores, film uniformity, subsurface defects	Yes	Fast	Yes

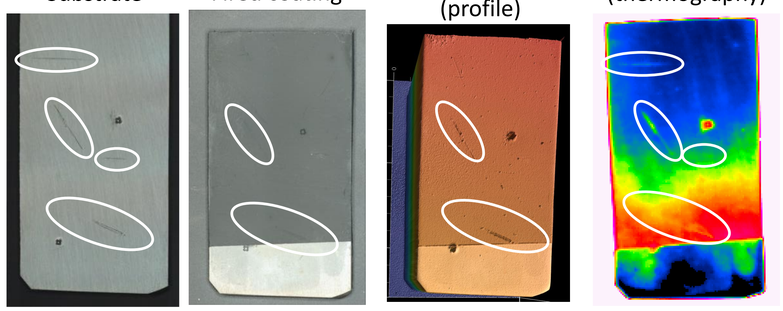
Optical profilometry capabilities at Redox (Keyence VR and VHX)

High throughput *High resolution (1000x)*

- High throughput and high magnification microscopy available at Redox
- Additional optical and thermography instrumentation at NREL
- Sample fabrication and characterization tools at Redox

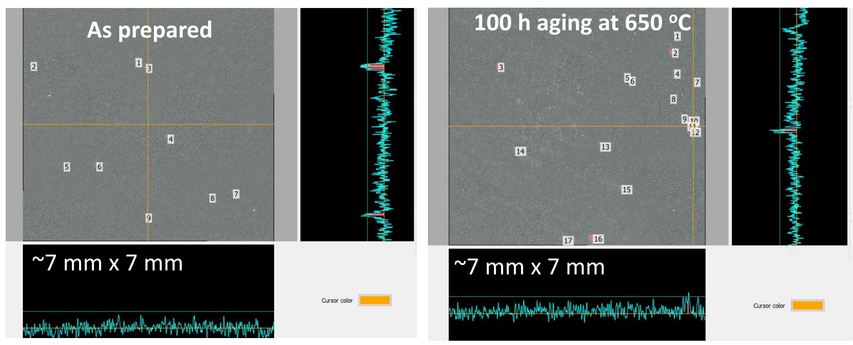
Results

Intentionally scratched substrate coated with MCO



- 4 scratches in stainless steel substrate (~2 cm x 3 cm) intentionally introduced
- Optical and height profile mapping can only detect two scratches
- *Thermography detects all 4 scratches!*

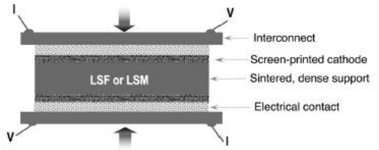
Peak identification using a height threshold



- *Twice as many peaks uncovered in aged sample!*
- Source of peaks and methods for mitigation under examination
- Currently evaluating detection scale-up to larger sample images

Area-specific-resistance (ASR) measurement of coating

Example setup from PNNL



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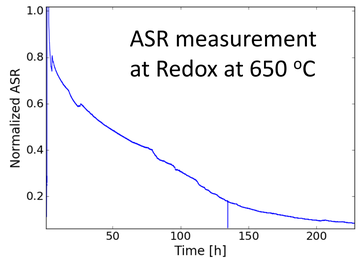

4-channel setup at Redox

- ASRs of ~50 mΩcm² at 650 °C consistent with literature
- ASR typically exhibits a decrease with time – may be related to thermally induced coating densification

Future Work

- Evaluate thermography and optical techniques ability to characterize intentionally and unintentionally added defects (e.g., scratches, thickness gradients, porosity)
- Continue to identify key defects that form during SOFC-like annealing and correlate with processing conditions
- Demonstrate scale-up of defect detection techniques to in-line manufacturing conditions

Summary and Conclusions

- Determined preliminary defects of interest, includes: film thickness uniformity, bumps and dips, film porosity, and film cracks/scratches
- Demonstrated ability to detect large scratches with optical microscopy and profilometry
- Demonstrated ability to detect fine scratches in substrate not optically visible using thermography
- Used Keyence analysis software to detect and quantify peak/bump defects on coating using height threshold
- Observed a 2x increase in the number of peaks after aging film in SOFC-like conditions
- Demonstrated ASR measurement of coatings, with results consistent with literature

Acknowledgement

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