Recent Progress of Stack Fixture Test at Pacific Northwest National Laboratory

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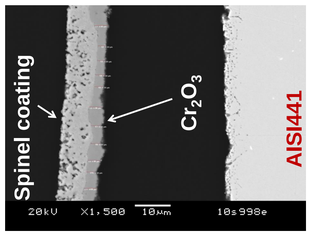
Introduction:

To bridge the gap between "button" cells and industrial stacks, a generic stack fixture was developed at PNNL. The objective was to validate SOFC candidate materials, processing, and testing methods. In FY14 research was focused on:

1. **Long-term validation and characterization of AISI441 interconnect with surface treatment**
2. **Effect of current density on the degradation of LSCF-based cells.**

1. Long-term test with surface-modified AISI441

AISI441 is the leading candidate for metallic interconnect; however, Cr-oxide scale would continue to grow even under protective (Mn,Co)-spinel coating and can lead to spallation upon thermal cycling. To enhance scale adhesion mechanical interlocking was proposed and formed by metal grit blasting. In FY14 AISI441 was tested in a generic stack fixture with surface blasting of grit #40 and #80 at 800ºC for 6000h.



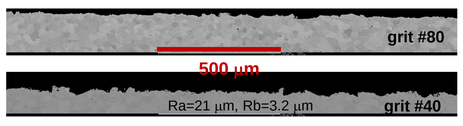
Cr-oxide scale de-bonded after ageing 4000h at 800ºC in air of the as-received AISI441 coated with (Mn,Co)-spinel.

Materials and Processing

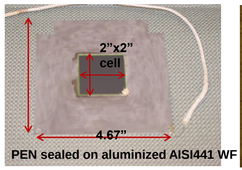
1. **Ce-(Mn,Co) spinel coating of surface-blasted AISI441**
2. **Aluminization of surface-blasted (#40, #80 grit) AISI441**
3. **AISI441 interconnect and window frame**
4. **Refractory glass seal for WF/PEN at 930ºC/2h**
5. **LSM20 or LSC20 and Ni paste + Ni mesh as contact**
6. **Final seal at 900-930ºC/2h and tested at 800ºC with fuel H_2:N_2=1:1 (3% H_2O) versus air at constant current mode**
7. **Impedance and IV sweep tests**
8. **Air side heat exchanger made of alumina (99%)**

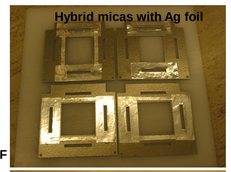
A commercial NiO-YSZ supported YSZ cell (5cm x 5cm) with LSM or LSCF cathode (16 cm^2) and compressive mica perimeter seal

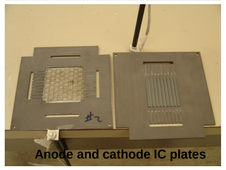
Cross-section of blasted AISI441

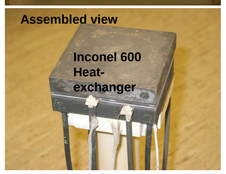


Generic Stack Test Fixture

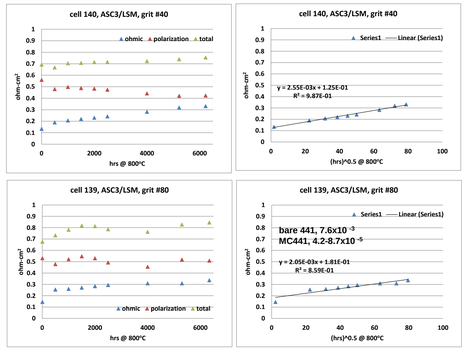




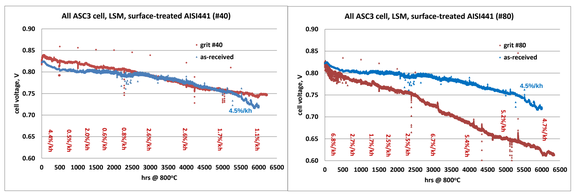




Impedance analysis

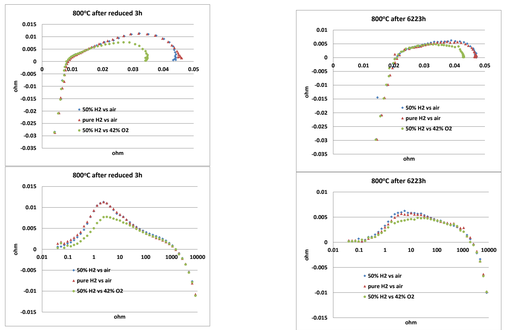


Long-term stability test at 800ºC

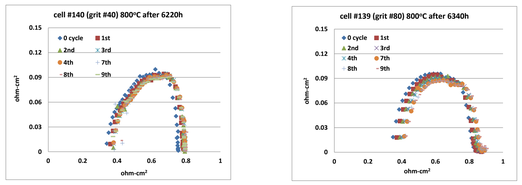


Cell with #40 grit-blasted showed stable/low degradation

EIS before and after~6300h stability test (grit #40)

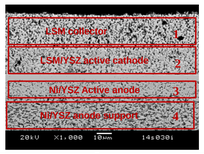


EIS during thermal cycling after ~6300h stability test



Minute increase in ohmic resistance suggests no substantial de-bonding of Cr-oxide scale

Post-test microstructure analysis



grit #40 grit #80

Area	O	Si	Cr	Mn	Ni	Sr	Zr	La
#1	60.67	0.23		19.04		4.68		15.64
#2	68.38			9.15	0.28		16.19	7.03
#3	46.88		0.04		31.32		21.97	
#4	48.15		0.03		29.81		22.40	

Area	O	Si	Cr	Mn	Ni	Sr	Zr	La
#1	58.39			20.60		4.72		16.29
#2	66.10			10.15		15.94	7.82	
#3	44.85				31.89		23.27	
#4	45.80				31.17		23.03	

Area	O	Si	Cr	Mn	Ni	Sr	Zr	La
#1	59.30	0.40		19.86		4.64		16.07
#2	67.27			9.99	0.33		15.83	7.59
#3	45.48				31.30		21.35	
#4	47.93				29.88		22.52	

Area	O	Si	Cr	Mn	Ni	Sr	Zr	La
#1	58.39			20.35		4.75		16.51
#2	65.98			10.03	0.29		16.17	7.53
#3	44.17				33.14		22.69	
#4	45.19				31.96		22.82	

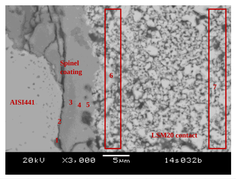
Area	O	Si	Cr	Mn	Ni	Sr	Zr	La
#1	59.22	0.39		19.82		4.74		16.13
#2	67.46			9.76	0.35		16.13	7.45
#3	45.18		0.04		33.57		21.32	
#4	47.76		0.02		29.99		22.46	

Area	O	Si	Cr	Mn	Ni	Sr	Zr	La
#1	57.71			20.81		4.99		16.48
#2	66.21			10.03	0.25		16.01	7.76
#3	42.36				35.42		22.22	
#4	45.13				31.82		22.85	

Area	O	Si	Cr	Mn	Ni	Sr	Zr	La
#1	58.84	0.41		19.89		4.78		16.34
#2	66.52			10.39		1.08	14.56	8.06
#3	42.74				36.38		20.93	
#4	46.70		0.01		31.00		22.61	

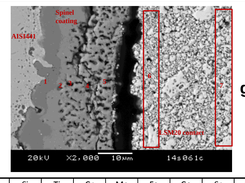
Area	O	Si	Cr	Mn	Ni	Sr	Zr	La
#1	57.88			20.51		5.20		16.43
#2	67.55			9.36		1.09	15.72	7.16
#3	43.60				33.86		22.54	
#4	45.24				31.95		22.81	

No Cr detected in electrode for both #40 and #80 grits



grit #40

Area	O	Si	Ti	Cr	Mn	Fe	Co	Sr	Nb	La	Ce
#1	15.12	1.09	6.27	22.98	1.39	52.37			0.78		
#2	65.51	1.60	0.33	27.57	1.08	3.92					
#3	60.32			28.71	4.26	0.59	6.13				
#4	54.92		0.29	1.16	29.45	2.73	10.27			0.56	0.62
#5	52.35				24.30	2.11	4.41	3.39		13.44	
#6	55.89				19.68	0.75	2.00	4.25		17.21	0.22
#7	58.80				19.77			4.36		16.83	0.24



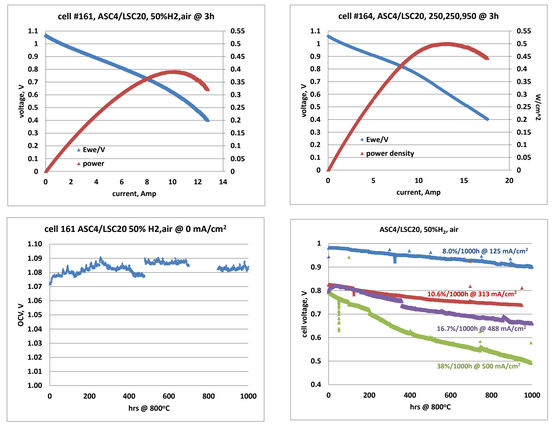
grit #80

Area	O	Si	Ti	Cr	Mn	Fe	Co	Sr	La	Ce
#1	64.28		0.15	34.3	0.81	0.33	0.12			
#2	36.87		0.89	3.15	36.84	3.06	19.19			
#3	61.77		0.48	0.43	20.89	2.46	13.97			
#4	33.94	3.51	0.55	2.01	35.20	3.44	20.80			0.55
#5	60.31		0.19	0.26	23.78	2.10	13.36			
#6	59.55				18.70		0.98	4.39	16.38	
#7	59.85				19.34			4.32	16.49	

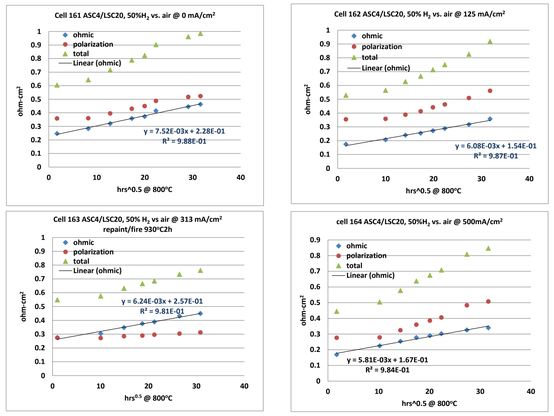
Cr-oxide scale showed good bonding to AISI441 for both #40 and #80 grits

2. Effect of current density on LSCF-based cell degradation

Five LSCF-based cells were tested at 0, 125, 313, and 500 mA/cm² at 800ºC



Impedance analysis



Summary and Conclusion

(A) Validation of surface treatment of AISI441 interconnect

1. **AISI441 with surface blasting to enhance the Cr-oxide scale was validated at 800ºC over 6000h. Results showed better cell performance for coarse grit #40 than finer grit #80. The cell performance of grit #40 was similar to the non-treated one.**
2. **EIS with various gases showed cathode side was rate limiting, and increase was primarily from ohmic part for both grits.**
3. **EIS of thermal cycling after 6000h test showed small increase in ohmic resistance, suggesting no Cr-oxide scale de-bonding. This is also consistent with SEM observation and EDS analysis of no Cr detected in electrode.**

(B) Effect of current density on LSCF-based cell's performance

1. **Five LSCF-based cells were tested at various current density (0-500 mA/cm^2) and the results showed increased degradation with increasing current density, consistent with small "button" cells.**
2. **All cells showed parabolic behavior of ohmic resistance with constants 2-3 times greater than LSM-based cells. The mechanism for this diffusion related process remains to be determined.**

About Pacific Northwest National Laboratory

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