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### **Glass Optimization**

> No crystallization in Glass 102 after heat treatment at 800°C in air



Glass 102				
800C 500hr				
	500μm 			
800C 1248hr	· · ·			
	500μm 			
800C 2000hr				
	500µm			

	2500				
	3500				
	3000	_			
	2500	(	573		
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> XRD confirms no crystallization in Glass 102 after >2000 hr heat treatment at operational temperatures

# **High-Temperature Viscous Sealing Glasses for Solid Oxide Fuel Cells**

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> Develop glass compositions that exhibit stable thermomechanical/ thermochemical properties, including viscosity, for use as seals for SOFCs a) Long-term stability in viscosity (650-850°C) c)  $T_{soft}$ : < 650°C: requisite flow for re-sealing behavior d)  $T_{Lig}$ : < 800°C (as low as possible): stable, a small volume fraction of crystals e) CTE(RT-subT<sub>g</sub>): 10-12.5×10<sup>-6</sup>/°C (YSZ- SS441)

### **Promising compositions were identified**

Preferred Compositions Exhibit Promising Sealing Behavior

	Phase I		Phase II				
	Glass 2	Glass 4	Glass 28	Glass 73	Glass 75	Glass 77	Glass 102
Glass system	BaO-B <sub>2</sub>	O <sub>3</sub> -SiO <sub>2</sub>	$BaO-RO-Al_2O_3-B_2O_3$		BaO-RO-A	N <sub>2</sub> O <sub>3</sub> -B <sub>2</sub> O <sub>3</sub> -Si	O <sub>2</sub>
T <sub>g</sub> (°C) measured from CTE curve	619	599	581	624	623	625	604
Dilatometric T <sub>s</sub> (°C)	650	632	615	640	650	656	639
CTE 40-500°C (/°C)	8.19x10 <sup>-6</sup>	7.32x10 <sup>-6</sup>	7.48x10 <sup>-6</sup>	8.48x10 <sup>-6</sup>	8.17x10 <sup>-6</sup>	9.25x10 <sup>-6</sup>	7.25x10 <sup>-6</sup>
Liquidus T (°C)	805	790	795	800	810	810	Non-Crystallizing

### **Stable Viscosity**

> Viscosity measurements provide valuable performance information



# **Re-Sealing Tests (ex-situ)**

Seal originally found Glass seal deliberately cracked by Crack healed after re-heating high cooling rate quench (>25°C/s) to 725°C for 2 hrs



**Glass Optimization-cont.** 





Stable viscosity of Glass 102 after 2000 hr heat treatment at 800°C

# **Re-Sealing Tests-cont. (ex-situ)**

Temperature (°C)	Time (hr)	Observation	Viscosity log(η) (Pa-s)
800	2	Healed	3.7
750	2	Healed	5.1
725	2	Healed	5.9
700	2	Healed once, but not a second time	6.9

Glass	Fitt Paran	T <sub>g</sub> (°C)	
	m	T <sub>g</sub> (°C)	Dilatometri
Glass 73 as-cast	64.1	606	624

# **Glass Optimization-cont.**

Glass 102 coupon seal heat treated 2280 hr at 800°C in air > Excellent wetting and bonding to both aluminized metal and YSZ

- Glass is homogeneous
- > No crystals in glass
- $\geq$  BaAl<sub>2</sub>Si<sub>2</sub>O<sub>8</sub> layer at glass/metal interface



- High temperature measurements (1-10<sup>4</sup> Pa-s) by the rotating spindle technique
- Low temperature measurements (10<sup>5</sup>-10<sup>11</sup> Pa-s) by the parallel plate technique
- Viscosity-temperature curves fit using the MYEGA viscosity model (JC Mauro, PNAS, 2009)



> No significant elements from metal or ceramics diffusing into glass





### Long-Term Reactivity Characterization

Glass 73 reaction couple: 103 Thermal cycles (750°C to RT) Excellent wetting and bonding to both aluminized metal and YSZ Man Munderman





> No major Cr or Fe migration to glass seal Some Al migration to the interface of glass seal or to ceramic substrate

# **On-going & Planned Work**

- > Determine electrical stability of viscous seals
- Study long-term viscous behavior
- Characterize long-term thermochemical reactions
- Hermeticity and 're-sealing' behavior

### Acknowledgements

- > SECA
- DOE SBIR Phase II Contract # DE-SC0002491
- > DOE Project Officer: Dr. Joseph Stoffa, NETL
- > Dr. Yeong-Shyung Matt Chou/Dr. Jeff Stevenson, PNNL



### **DOE SBIR Phase II Contract # DE-SC0002491**

