

# Interconnect Lifetime Prediction from Interfacial Indentation



Pacific Northwest  
NATIONAL LABORATORY

Proudly Operated by **Battelle** Since 1965

Elizabeth V. Stephens, Zhijie Xu, Brian J. Koepfel, and Jeffry W. Stevenson

## OBJECTIVE

Develop a modeling approach to predict the life of spinel-coated interconnect materials under typical SOFC operating conditions.

## TECHNICAL APPROACH

In this work, an integrated experimental and modeling approach is utilized (Fig. 1). A model based on experimental data from interfacial nano and microindentation performed on coated, surface modified, 441 stainless steel (SS) specimens exposed to 800°C is used to predict the interconnect lifetime under isothermal cooling conditions.

The model considers buckling driven blistering of oxide scale on the interconnect surface as the main failure mechanism where relationships between the energy release rate,  $G$ , the material interface toughness,  $\Gamma$ , and the Mode I stress intensity factor,  $K_I$ , are established to determine the critical oxide thickness for failure to occur. Lifetime can then be estimated from the oxidation kinetics and the critical thickness.

## INTERFACIAL ANALYSIS

Nano and microindentation was performed where a Vickers indenter was applied on the cross section of surface blast and surface ground modified specimens to generate and propagate a crack along the oxide scale and the 441 SS substrate interface.

The crack length,  $a$ , the local oxide thickness, and the half-diagonal of each indent were measured optically as illustrated in Fig. 2 to determine the critical load,  $P_c$ , and crack length,  $a_c$ , for which no cracks propagate (Fig. 3). Once these parameters are determined,  $K_I$  may be defined and input to the model.

$$K_I = 0.015 \frac{P_c}{a_c^{3/2}} \left(\frac{E}{H}\right)_I^{1/2} \text{ where } \left(\frac{E}{H}\right)_I^{1/2} = \frac{\left(\frac{E}{H}\right)_S^{1/2}}{1 + \left(\frac{H_S}{H_C}\right)^{1/2}} + \frac{\left(\frac{E}{H}\right)_C^{1/2}}{1 + \left(\frac{H_C}{H_S}\right)^{1/2}}$$

## MODEL DEVELOPMENT

A thin film of thickness  $h$  on a thick substrate are considered where both the film and substrate are isotropic materials. During isothermal cooling, two dominant failure modes are commonly observed with one being compressive stress-driven blistering (Fig. 4). The energy release rate,  $G$ , is used to describe the observed buckling (J.W. Hutchinson and Z. Suo, (1992), *Adv Appl Mech* 29:63):

$$G = \frac{(1-\nu^2)h\sigma^2}{2E} \left(1 - \frac{\sigma_c}{\sigma}\right) \left(1 + 3\frac{\sigma_c}{\sigma}\right) \quad G \text{ increases with } \sigma \text{ and } h$$

where  $\sigma_c$  is the critical buckling stress. When  $\sigma \geq \sigma_c$ , buckling occurs and drives the blister to propagate.

$$\sigma_c = \frac{\pi^2 E}{12(1-\nu^2)} \left(\frac{h}{b}\right)^2 \quad b \text{ is the half width of blister}$$

The interface cracking tends to be mixed mode (Mode I and II). This mode mixity is represented by the phase angle  $\Psi$ :

$$\tan \Psi = \frac{4 + \sqrt{3}\xi \tan \omega}{-4 \tan \omega + \sqrt{3}\xi} \quad \text{where } \xi = \sqrt{\frac{4}{3} \left(\frac{\sigma}{\sigma_c} - 1\right)}$$

$\omega = \omega(\alpha_D)$  function of Dundur's mismatch parameters

## CRITICAL THICKNESS CALCULATION

Interfacial fracture toughness,  $\Gamma$ , is defined as the minimum value of  $G$  needed to propagate the crack. When  $G > \Gamma$ , the film fails.

$$G(h, \alpha_D, \sigma, b) > \Gamma(\Psi(h))$$

$$\Gamma(\Psi) = \Gamma_I \left(1 + \tan^2[(1-\lambda)\Psi]\right) \quad \Gamma_I = \frac{1-\nu^2}{E} K_I^2 \quad \text{From interface indentation experiments}$$

Combining expressions yields the critical thickness  $h_c$  (Fig. 5):

$$\frac{h_c \sigma^2}{K_I^2} F(h_c/b, \sigma, \alpha_D) = 2 \quad \text{where } F(h_c/b, \sigma, \alpha_D) = \frac{(1-\sigma_c/\sigma)(1+3\sigma_c/\sigma)}{1 + \tan^2[(1-\lambda)\Psi]}$$

If  $h > h_c$ : film will fail due to isothermal cooling  
If  $h < h_c$ : film will survive during isothermal cooling

## CONCLUSIONS

- Based on the experimental measurements for 441SS at 800°C,  $K_I = 2.0$  to  $2.9 \text{ MPa}\sqrt{\text{m}}$ , and the current model predicts the critical scale thickness in the range of  $4.1 \mu\text{m}$  to  $8.5 \mu\text{m}$ .
- The lifetime predicted from  $K_I$  for surface blast specimens was longer than surface ground specimens consistent with observations from the long term oxidation studies in progress.

## FUTURE WORK

- Predict lifetime of surface modified specimens exposed to 800°C.
- Determine  $K_I$  for unmodified, spinel-coated 441 SS specimens exposed to 800°C to verify the model predicts shorter lifetime.
- Determine  $K_I$  for surface modified specimens exposed to 850°C.
- Benchmark methodology with possible known standards.
- Evaluate effects of surface roughness on the methodology and data scatter.

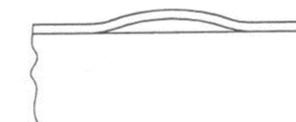


Figure 4. Schematic of buckling-driven interface delamination with mixed Mode I and II.

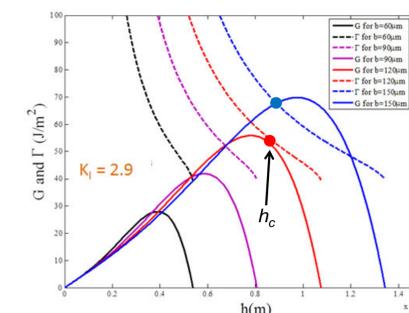
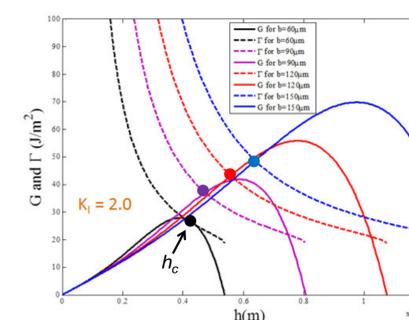


Figure 5. The predicted thicknesses where no blister delamination is expected based on  $K_I$  experimental measurements.

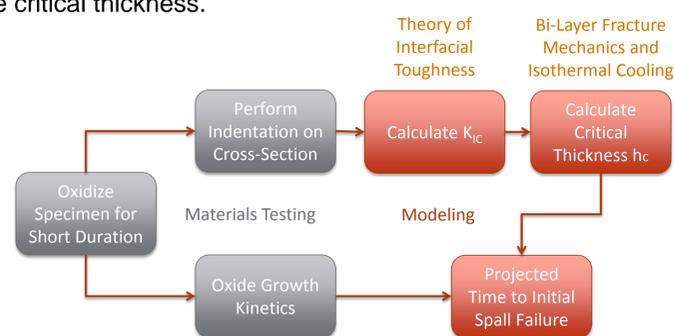


Figure 1. Schematic of proposed predictive methodology.

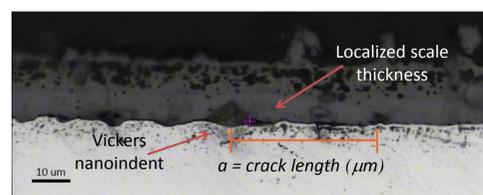


Figure 2. Nanoindentation performed on a surface ground specimen exposed to 10,000 hour at 800°C.

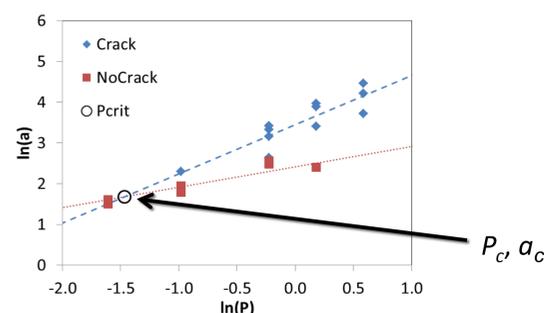


Figure 3. Plot of the interfacial analysis results of a surface blast specimen exposed to 14,000 hour at 800°C and its corresponding  $P_c$  and  $a_c$  point.

## ACKNOWLEDGEMENT

This work was funded as part of the Solid-State Energy Conversion Alliance Core Technology Program by the U.S. Department of Energy's National Energy Technology Laboratory.



For more information on the science you see here, please contact:

**Brian J. Koepfel**  
Pacific Northwest National Laboratory  
P.O. Box 999, MS-IN: J4-55  
Richland, WA 99352  
brian.koepfel@pnnl.gov

PNNL-SA-97132