

# Micro-Indentation Mechanical Properties of G18 Sealant

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## Introduction

The sealant has an important role in solid oxide fuel cells (SOFCs) because it separates the compartments of the fuel cell and the reactant chemicals. The life of SOFCs can be greatly limited by their sealant. SOFCs operate at high temperatures, and thermal cycling causes expansion and contraction of the components of the fuel cell. The sealant is stressed as it attempts to expand and contract at the same rate as these components. This stressing can lead to sealant damage and failure. Because sealant replacement is difficult, this will usually end the life of the fuel cell.

The Pacific Northwest National Laboratory has developed a sealant for SOFCs called G18. G18, a glass-ceramic, is barium-calcium-aluminosilicate-based with added boron oxide. It is a good sealant because it is chemically stable when introduced to SOFC components and chemical fuel and because it has a high coefficient of thermal expansion. This high coefficient of thermal expansion allows it to expand and contract at the same rate as the other components of the fuel cell, preventing cracks and damage.

As the fuel cell ages, enduring more thermal cycles, certain phases in the sealant increase in crystallization, making it more brittle and vulnerable to failure. The objective of this project was to use a micro-indentation device to examine the mechanical properties of G18 by creating and studying load-deflection curves. The curves were used to find the stiffness of G18 at different indentation rates, ages, and temperatures, modeling operating conditions in a fuel cell. The elastic modulus can be calculated from this data.

## Procedure

Micro-indentation trials were run on G18 samples that had been aged at 750°C for durations ranging from 1 hour to 4 days. The aging was meant to model thermal cycling in fuel cell operation. The indentation tip was cone-shaped and made of tungsten carbide (WC). The angle of the tip was 30°. The samples were mounted to a stage that was heated by a resistive heating cartridge. Each sample was tested at temperatures of 22, 200, 400, and 680°C, and at indentation rates of  $5.67 \times 10^{-5}$ ,  $3.87 \times 10^{-4}$ ,  $8.02 \times 10^{-4}$ , and  $1.32 \times 10^{-3}$  in/s. The indentation trials were used to produce load-deflection curves. The stiffness of the material is given by the slope of the linear elastic regions of these curves.

## Results and Discussion

Figure 1 shows the dependence of the curves on indentation rate. This data came from a sample that was aged for 10 hours. The trials were run at 22°C.

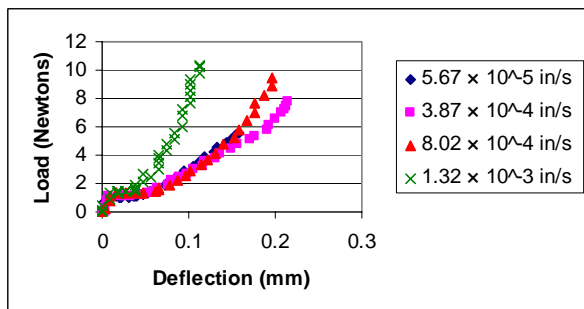


Figure 1. Load vs. Deflection at Varied Indentation Rate

The data shows that the load capacity of the sealant increased greatly when indented at  $1.32 \times 10^{-3}$  in/s but was not affected much by changes at lower indentation rates. This shows that G18 is strain rate dependant, which is characteristic of viscoelastic materials.

Figure 2 displays the change in stiffness as temperature changes on a sample that was aged for 1 hour.

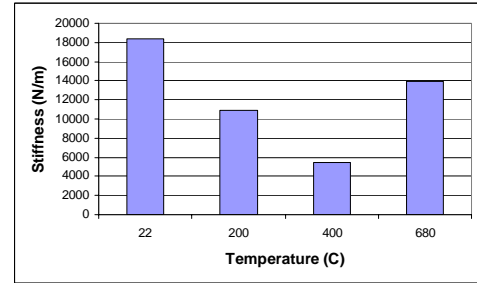


Figure 2. Stiffness at Varied Temperature (Age = 1 Hour)

Below the glass transition temperature of 619°C, the data shows a drop in stiffness as temperature rises. This is consistent with previous data collected by Penn State University in a dynamic resonance for un-aged G18 study. The rise in stiffness above the glass transition temperature may be due to further crystallization of some phases.

Figure 3 is representative of the dependence of stiffness on temperature in samples of older ages. This sample was aged for 4 days.

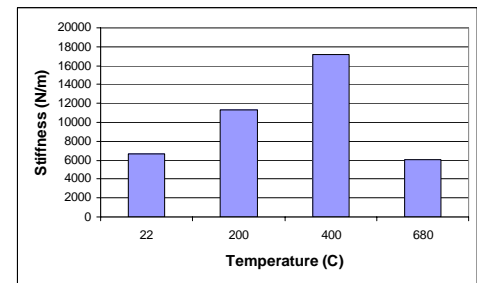


Figure 3. Stiffness at Varied Temperature (Age = 4 Days)

The longer-aged data is inverted compared to that of the un-aged. Below the glass transition temperature, the stiffness increases as temperature rises. The stiffness decreases after the sample reaches the glass transition temperature. These results are also consistent with the dynamic resonance data.

As seen in Figure 4, there is a moderately positive correlation between age and stiffness at high temperatures. This data was collected from samples at 400°C. This was expected, because aging causes further crystallization of some phases in G18. Surface cracks and pores could have altered the stiffness of the samples, preventing a clearer correlation. It is likely that the higher amount of crystalline phases caused cracking during cooling.

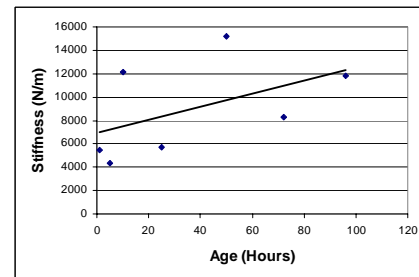


Figure 4. Stiffness at Varied Age. (400°C)

## Conclusion

The micro-indentation device produced quality load-deflection curves. Indentation rate made a large impact on the curves. The largest indentation rate created a great increase in the load capacity. The dependence of stiffness on temperature was consistent with previous data on samples of both young and old age. Samples of older ages displayed greater stiffness than the younger aged samples. In further testing, polishing the surface of the samples could prevent cracks and pores from harming the clarity of this correlation.