



Characterization of High Pressure High Temperature Cell Media



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BACKGROUND

The manufacture of polycrystalline diamond (PCD) simultaneously may involve pressures in excess of 8 GPa and temperatures over 1400°C. Generating these extreme conditions in an environment large enough to produce PCD for high wear applications is a serious materials challenge.

A cubic press allows large volume production of PCD. This works by placing the required sintering components within a high pressure high temperature (HPHT) compatible cube. As the anvils compress, the cube gasket material seals in the pressure while simultaneously insulating the anvils from the high temperatures at the core of the cell.

Gasket properties are critical to obtaining higher pressures and preventing damage to the cubic press. *In situ* experimentation with new cube materials is undesirable due to the high risk of causing costly damage to the press. Therefore, we must use *ex situ* material characterization to minimize *in situ* damage.

A given gasket material must be:

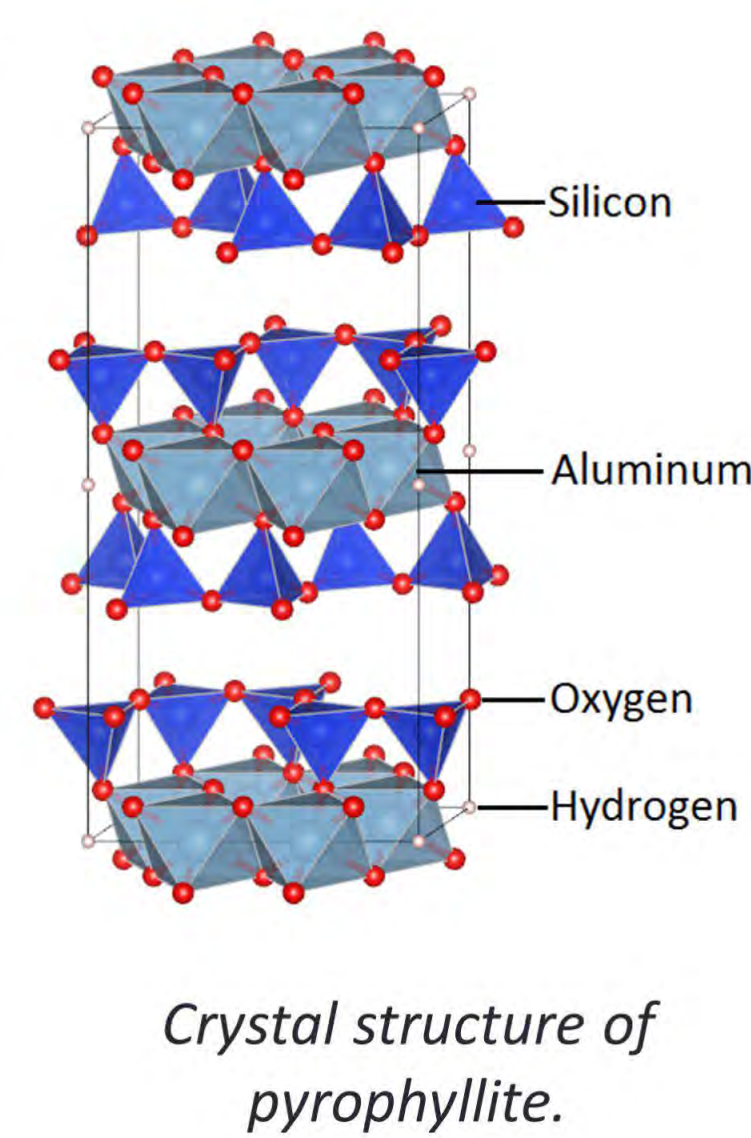
- Mechanically robust
- Capable of extreme plastic deformation without fracture
- A thermal barrier

The risk of new material experimentation can be significantly mitigated by *ex situ* assessment of the material's performance in each of the above categories.

HYPOTHESIS

Mechanical/Rheological Testing:

- Reducing free moisture content should strengthen the gasket material. The crystal structure of phyllosilicates, such as pyrophyllite (right), contains crystalline moisture that can be released at high enough temperature. Bake temperatures should be kept low enough to avoid a change in crystal structure.
- A new fixture could be designed to simulate cubic press gasket geometry for *ex situ* testing. Axial force versus axial displacement curves will show the material's ability to initially resist flow, the point of failure, and its ability to act as a gasket.

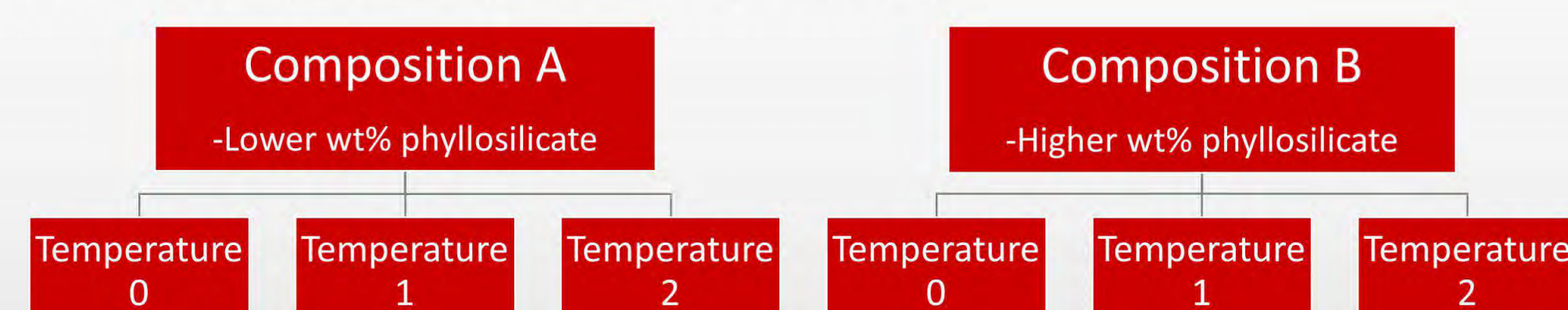


Thermal Barrier:

- Low thermal conductivity is required to protect the anvils from the high temperatures. Modeling has shown the material should have a thermal conductivity of 4 W/m*K or lower [1].

MATERIALS & METHODS

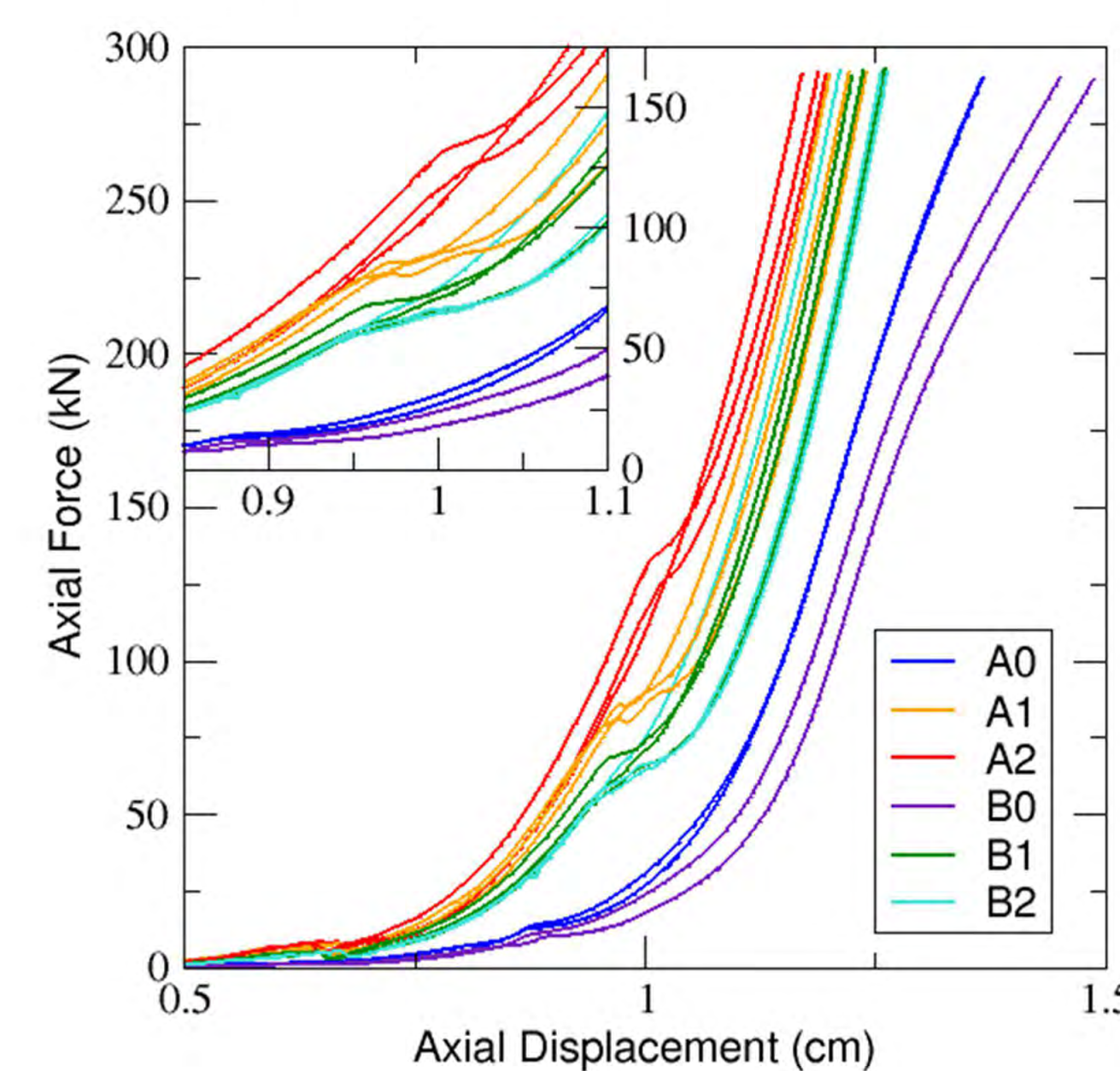
- Material variables: composition and bake temperature.
 - Temperature 0 < Temperature 1 < Temperature 2
- Samples were prepared by uniaxial pressing.



*Compositions and temperatures are associated with proprietary information and will not be specifically identified.

RESULTS

RHEOLOGICAL PROPERTIES

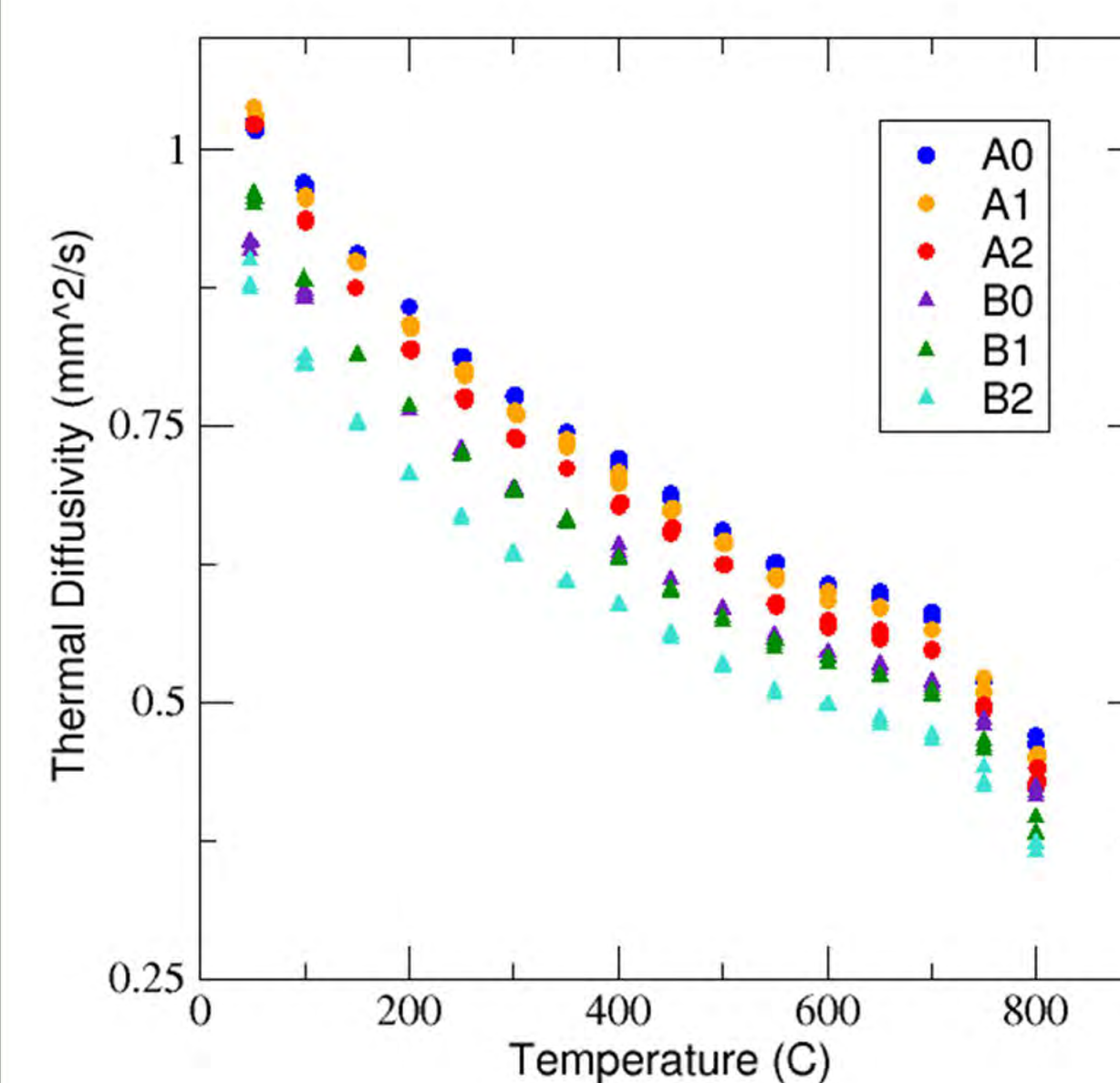


Axial force vs axial displacement curve (left) shows rheological properties are distinguishable and capable of being customized based on alterations of temperature or composition. The designed rheology fixture (right).

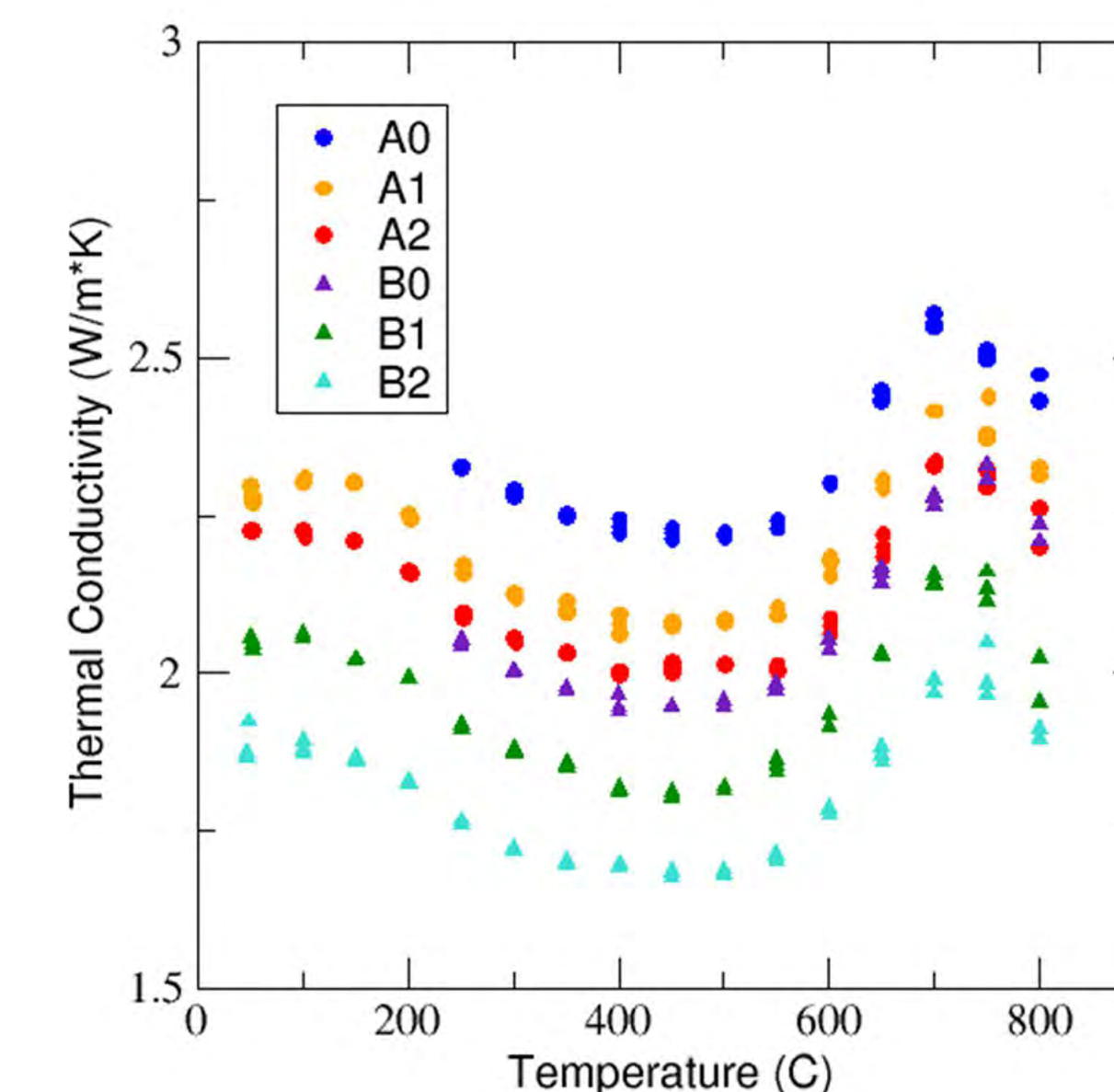
Force vs. Displacement:

- Unique rheological response for different samples.
- Ability to resist flow increases with higher bake temperatures.
- Is this test valid?
 - Contents in the extrusion cavity were soft, whereas gaskets in the press are hard, indicating the test may not fully simulate gasket formation.

THERMAL PROPERTIES



Thermal diffusivity as a function of temperature.



Theoretical thermal conductivity calculated using Neumann Kopp method.

Thermal Diffusivity:

- Phase change observed beginning at approximately 700°C correlating to crystalline moisture.
- Thermal diffusivity decreases as bake temperature increases.
- Thermal diffusivity decreases with increasing phyllosilicate content.

Thermal Conductivity:

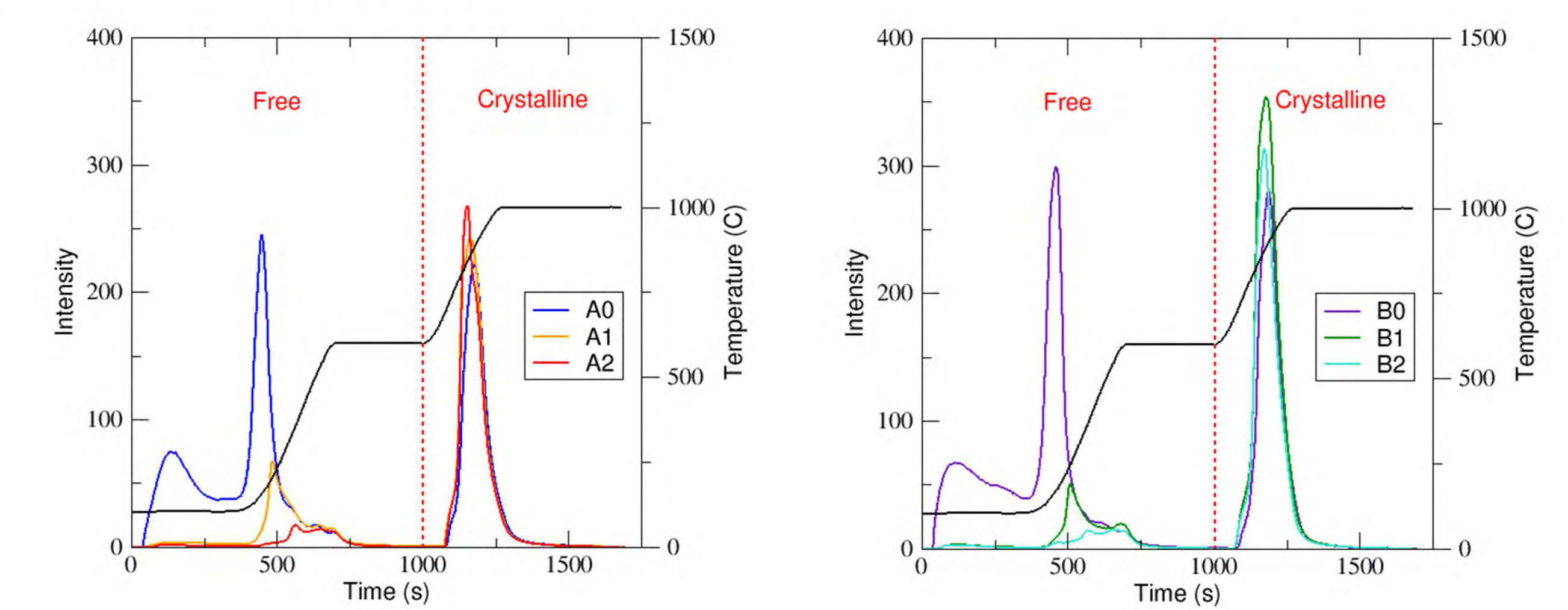
- Specific heat capacity calculated via Neumann Kopp method.
- Thermal conductivity is well below required 4 W/m*K.

$$\alpha = \frac{\kappa}{\rho C_p}$$

α - thermal diffusivity (mm²/s)
 κ - thermal conductivity (W/m*K)
 ρ - density (g/cm³)
 C_p - specific heat capacity (J/g*K)

RESULTS (CONTINUED)

MOISTURE CONTENT



Total moisture content of Composition A (left) is lower than Composition B (right) due to a lower phyllosilicate content. Both graphs show unchanged peak size in the crystalline moisture regime.

- Two distinct sets of peaks: free vs. crystalline moisture. Initial moisture loss is coming from the free moisture in the binding material and not chemically altering the phyllosilicates.
- Trapped intergranular moisture due to uniaxial pressing of samples causes the free moisture peak to extend past 105°C and into the 600°C ramp.

CONCLUSIONS

- The force vs. displacement curves developed with the rheology fixture indicate that new compositions could be directly compared and screened for use on a cubic press even though the test does not fully replicate the cubic press environment.
- Rheological and thermal properties can be altered by either composition or by bake temperature.
- All results show definite distinctions between compositions for mechanical robustness, flow, moisture, and thermal conductivity. Thus, the *ex situ* measurements may enable customization of experimental compositions to mitigate the risk of damage to the cubic press.

FUTURE WORK

- Correlate data to:
 - Mechanical strength
 - Coefficient of internal friction
- Determine coefficient of friction for die wall interaction.
- Build more representative test that includes elevated temperature.

ACKNOWLEDGEMENTS/REFERENCES

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- Thanks to US Synthetic for sponsorship and the use of their testing facilities.

[1] A. Carte, 'Thermal constants of pyrophyllite and their change on heating', *British Journal of Applied Physics*, vol. 6, no. 9, pp. 326-328, 1955.