

## SEM Evaluation of Advanced Refractory Failures

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The SEM is an invaluable tool in the evaluation of advanced refractories and their failure. A reaction vessel's refractory liner, at minimum, must protect the reaction vessel from elevated temperatures, corrosive slag and thermal cycling. To understand the failure mechanisms ARC staff had first to determine how an advanced chrome rich refractory was attacked by various components that make up a slag. Refractory cups were made from the refractory of interest and various compounds that can be found in a slag such as CaO, SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, NaCl were placed into the test cups and fired for 24 hours at the required temperature with the desired atmosphere. Fig 1.

The cups are prepared for examination by embedding in epoxy and cross sectioning. SEM examination revealed how various slag compositions attacked and penetrated the refractory. The slag could corrode, free refractory grains or react with the refractory and form a new compound. Fig. 2. It was found that the only way to measure slag component penetration was with multiple elemental X-ray maps. SiO<sub>2</sub> penetrated deeply and in many instances moved through the cup. Fig 3.

The knowledge of slag refractory interactions gathered during cup testing was applied to actual spent refractory from reaction vessels. Obtaining samples from the reaction vessel itself proved difficult due to time constraints imposed in relining. Samples were selected based on spent brick shape, color or location in the heap of spent refractory Fig. 4. Sample preparation affected the results dry, water or oil coolant during cutting may dissolve reaction products. The complex reactions between the slag and refractory made for very interesting and time consuming evaluation. Elemental X-ray maps at low and high magnification combined with point analysis aided in locating regions of interest. Crystals were found growing in voids and appear to be from vapor deposition Fig. 5. Other crystal structures are from the slag refractory interaction. Knowledge gathered from this and other supporting research resulted in a new patented refractory composition that resists slag penetration.

### References

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FIG. 1: Fired cup test with slag penetration.

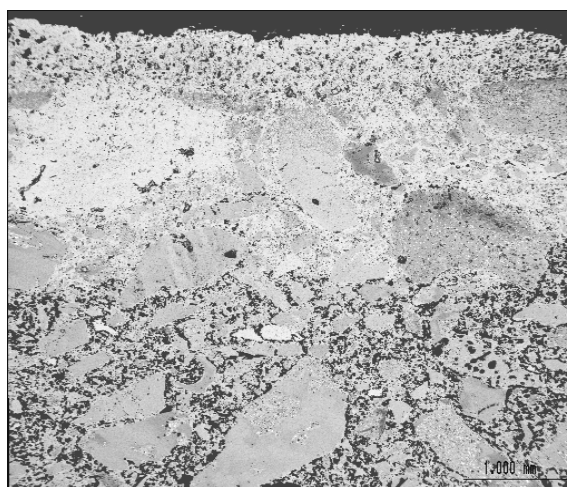


FIG. 2: BSE micrograph of CaO cup test CaO severe attack of the refractory.

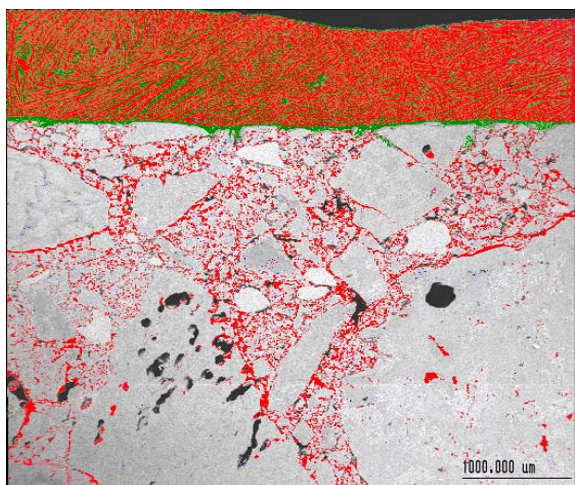


FIG 3: Si and Ca K $\alpha$  elemental X-ray maps overlaid. Note how deep the Si penetrates.



FIG. 4: Spent refractory

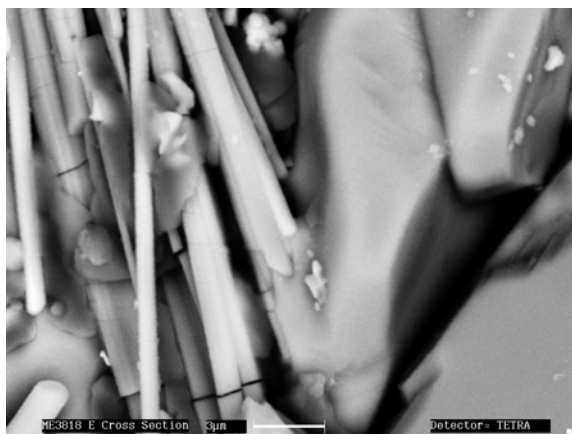


FIG. 5: Crystal growth from vapor deposition.