444 - a formulation for thermal shock resistance

Kenneth Andrew Domann*, Ceramic Engineer and Larry Finn**, Laboratory Manager discuss a formulation that has helped solve the issue of thermal shock in applications such as expendables. Research included a collaboration with the local university as well as testing in glass plants.

Over the past five years we have worked to solve the issue of thermal shock, particularly in feeder tubes. Our customers had continually asked for this customisation. Initially, the work showed increases in thermal shock resistance negatively affected the corrosion resistance. This is the normal trade off for refractories development. So, our task was twofold: increase the thermal shock resistance, while maintaining corrosion resistance.

We followed the basic scientific method in four to five directions, then focused on the most promising direction, the formulation of what would later become 444. With our direction down to one path, we set up a design of experiment to optimise the mix, and now add a third variant, manufacturability.

First sintering aids were investigated. Several different dopants including titania were assessed at different concentrations and the resulting parts were tested for basic properties, including MOR, and MOR after thermal shock cycles. After testing, these dopants were disregarded from future development of this mix.

University collaboration

After hundreds of laboratory batches and numerous collaborations with the local university, Missouri S&T, we were able to get a mix for trial. For two years we tested this mix with many customers.

The results took a long time to acquire, due to the necessity of post-mortem evaluation. However, at the end of this period we concluded that this mix has an excellent resistance against thermal shock, while also maintaining a long lifetime compared with the competitors and our traditional mixes.

Following the success at our customer trials we have now offered this new mix formulation to all.

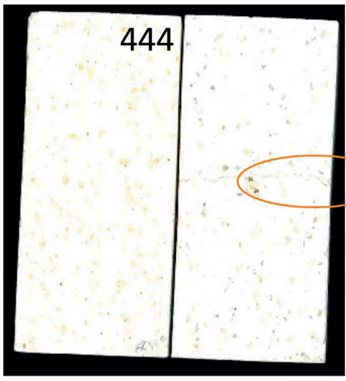

Fig 1. Cross Section of two thermal shock samples.

The thermal shock resistance of each specimen is rated based on how well they hold up to thermal cycling. Visual inspections and impulse excitation testing are used to rate the samples after radical, extensive thermal cycles. Specimens are heated up to 2500°F and removed from the furnace and allowed to air quench to room temperature.

This is repeated five times. ***Fig 1*** shows two such specimens that were exposed to these thermal cycling conditions. As seen in ***Fig 1*** the 444 specimen remained intact while the standard specimen developed a crack across the centre.

Corrosion testing

Corrosion testing was also done on specimens made of 444. Under an accelerated corrosive environment the specimens held up to chemical attack just as well as the standard materials.

Once the mix was finalised, a firing DOE was implemented. Samples of 444 were fired at various temperatures, and then specimens were taken from the samples and tested under thermal shock and static and dynamic corrosion. These tests were to ensure that the optimal firing temperature was implemented when sintering this material. The final firing temperature was determined from these tests, and sample parts were cast and trialed at glass manufacturing facilities. This gave us a good idea of how the parts preformed in an industry setting. The trials went very well and the parts were observed post-mortem, to check the wear and conditions of the parts.

No cracks or unforeseen failure mechanisms were present in the trial parts, finalising the testing period of 444 and facilitating the release of the new mix. The superior thermal shock resistance of 444 makes it the ideal choice for high thermal shock applications, including but not limited to expendables. ■

*Ceramic Engineer,
**Laboratory Manager,
Bucher Emhart Glass, Cham, Switzerland
www.bucheremhartglass.com

Thermlac Tubes.