Mullite Formation

SEM image showing an alumina grain surrounded by a mullite network

Only few mineral phases have the same importance for refractoriness like the mullite phase. The mineral, named after the Scottish Isle of Mull, is rarely found in nature, but is a common mineral in fired alumina-silicate materials.

Mullite is the only stable high-temperature phase between alumina and silica, and the composition is commonly expressed as $3Al_2O_3 \cdot 2SiO_2$.

Mullite is frequently seen as a component of the high-temperature bonding phase in refractories.

It is known to give high refractoriness, moderate thermal expansion, good strength at elevated temperatures, and high creep resistance, giving good dimensional stability.

Mechanism of Mullite Formation

Mullite may be formed from alumina and silica in the bonding phase of refractory castables. The formation passes through several steps:

1300°C

At around 1300°C, alumina and silica form a liquid phase, normally together with calcia from cement.[1] When the bonding phase is partially liquid, the material softens and the hot strength is significantly reduced.[2]

1400°C

From this liquid, mullite may start to precipitate. To make it happen, the amount of silica in the liquid must be in excess as compared to calcia.

Although the amounts of silica and calcia are the most critical factors for mullite formation, elements like firing temperature, soaking time and total amount of impurities are also found to be important.

1500°C

The needle-like mullite crystals provide strength by bridging the aggregates together, forming a strong, highly refractory matrix. Depending on the amount of cement in the castable, this matrix may retain its strength at temperatures up to 1800°C. Impurities, in particular calcia from cement, will reduce the maximum operating temperature.

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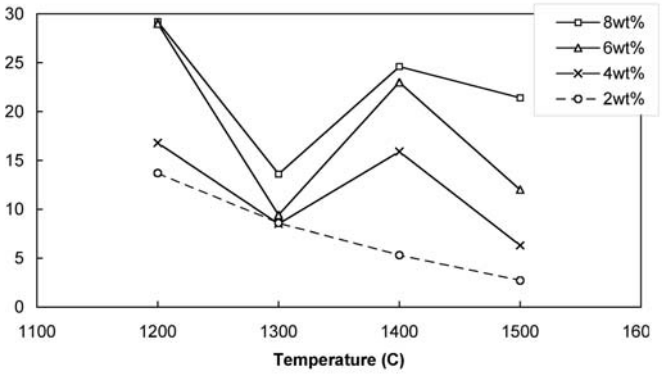
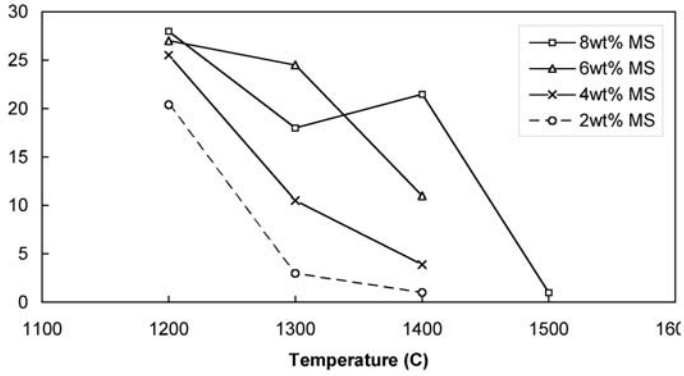
Effect of Mullite Formation in Low and Ultra-low Cement Castables



Hot-M.O.R. of ultralow cement (0.5wt% cement), fused alumina based castables as a function of temperature. Castables with different amounts of microsilica. 24hours at temperature. q=0.25, max. particle size 4mm, 13 vol% water for casting (3.8–4.2wt%).



Hot-M.O.R. of low cement (6wt% cement), fused alumina based castables as a function of temperature. Castables with different amounts of microsilica. 24hours at temperature. q=0.25, max. particle size 4mm, 13 vol% water for casting (3.8–4.2wt%).

Ultra-low Cement Castables (ULCC)

The increase in strength at 1400°C is a result of mullite formation in the bonding phase. Mullite formation is proportional to the microsilica content. At 1500°C, liquid phases appear, and the strength is reduced. The strength reduction is especially severe for samples with low contents of microsilica. With only 2wt% microsilica, there is not enough silica in the system for mullite formation. Consequently, there is no gain in strength at 1400°C for this sample.[2]

Low Cement Castables (LCC)

LCC's normally contain about 6–10wt% cement versus 0.5–2.5wt% in ULCCs. The figure shows that with higher content of cement, more silica is needed to obtain mullite formation. Only the sample with 8wt% microsilica has enough silica for mullite formation, as reflected by its increase in strength at 1400°C.[3]

References:

1) S.H. Risbud and J.A. Pask, J. Mater.Sci., 13 (11) 2449–2454 (1978)
2) B. Myhre, A.M. Hundere, H. Feldborg, C. Ødegård: «Correlation between mullite formation and mechanical properties of refractory castables at elevated temperatures», Proc. VIII Int. Met. Conf. Ustron, Poland. May 25–28, 1999, p. 163–172.
3) B. Myhre and B. Sandberg: «Mullite formation in tabular alumina based refractory castables with hydraulic alumina as binder», Acers 97th Annual meeting in Cincinnati, May 2, 1995.

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