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HIGH TEMPERATURE ALKALI CORROSION OF CERAMICS

IN COAL GAS

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Objective/Scope

High temperature alkali corrosion has been known to cause premature failure of ceramic components used in advanced high temperature coal combustion systems such as coal gasification and clean-up, coal fired gas turbines, and high efficiency heat engines. The objective of this research is to systematically evaluate the alkali corrosion resistance of the most commonly used structural ceramics including silicon carbide, silicon nitride, cordierite, mullite, alumina, aluminum titanate, zirconia, and fireclay glass. The study consists of identification of the alkali reaction products (phase equilibria) and the kinetics of the alkali reactions.

Technical Progress

Mullite Alkali Corrosion Kinetics

The alkali corrosion kinetics of mullite (Superior Technical Ceramics, Inc.) were investigated in a constant partial pressure of sodium nitrate. As previously described, the constant alkali vapor pressure was generated by flowing compressed air (filtered to remove carbon dioxide and moisture) at 200 ml/min over the sodium nitrate salt maintained at 715°C and monitoring the weight loss of the salt. After passing through the salt chamber, the gases flowed into the reaction chamber where the samples were

held at the appropriate temperature. The concentration of sodium nitrate was calculated to be approximately 0.90 vol%. The mullite pellets (approximately 3x3x6 mm) were placed vertically in an alumina holder containing alumina powders to support the specimens and were isothermally heated at the appropriate temperatures from 2 h to 15 h. The thickness of the mullite samples before and after the alkali corrosion was measured using a micrometer (Starrett model 230M). The reaction layer thickness was calculated as the difference between the thickness after and before corrosion. The reaction products were identified using x-ray diffraction (XRD). The microstructure of the corroded specimen was examined and compared to that of the as-received specimen using a scanning electron microscope.

The reaction thickness of the mullite in the alkali-containing atmosphere versus reaction time is shown in Figure 1. The reaction layer thickness appears to be linearly related to the reaction time for the reaction temperatures tested so far. The linear reaction rate constants are 9 $\mu\text{m/h}$ at 1000°C and 25 $\mu\text{m/h}$ at 1050°C respectively. X-ray diffraction analysis of the as-received material showed only mullite and a trace of corundum were present. No cristobalite was detected. Density measurements (using archimedes method) gave a true density of 3.004 and a bulk density of 2.998 g/cc for the as-received material. Elemental analysis using EDAX (energy dispersive analysis of x-rays) measurements of the samples (which were cut on a diamond saw) before corrosion show only aluminum and silicon as the major constituents with trace iron, copper and potassium.

EDAX measurements after corrosion at 1000°C for 8 h show only aluminum, silicon and sodium. X-ray diffraction analysis of the mullite specimens after reaction in the alkali-containing atmosphere revealed the presence of sodium aluminate and carnegieite. The peaks in the diffraction pattern shift slightly as a function of reaction time at 1000°C suggesting that there might be some solid solution of the sodium aluminate and the carnegieite phases.

Figure 2 shows the SEM micrographs of the as-received mullite and the specimen corroded at 1000°C for 8 h. The reaction layer exhibits a large number of microcracks as can be seen in Figure 2b. It is not certain whether these cracks form during the reaction process as a result of the differences in volumes of the substrate and the corrosion products or whether they form during the cooling period due to differences in the thermal expansions of the substrate and the corrosion layer. The 950°C and 1100°C alkali reaction kinetics for mullite and the alkali reaction kinetics for cordierite and zirconia between 950°C and 1100°C will be investigated for the next reporting period.

Figure 1. Mullite reaction layer thickness versus time

a.)

b.)

Figure 2. SEM micrograph of (a) as-received mullite and (b) after 8 h at 1000°C

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