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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 program 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 as a function of temperature and time.

## Technical Progress

### I. Sample Preparation and Acquisition

Powder samples used for phase equilibria studies including cordierite and aluminum titanate were prepared by sol-gel techniques. Cordierite was synthesized by mixing the appropriate amounts of aluminum tri-sec butoxide, tetraethyl orthosilicate, and magnesium nitrate in ethyl alcohol with constant stirring, followed by the addition of water. Pure cordierite phase was formed by firing the gel samples at 1350°C for 24 h. Aluminum titanate was prepared in a similar manner using the aluminum tri-sec butoxide and titanium butoxide, following the same heat treatment.

For the corrosion kinetics studies, high density, high purity silicon carbide, silicon nitride, zirconia, cordierite, mullite, alumina and aluminum titanate were purchased from commercial sources. Fireclay glass (76 $SiO_2$-16$Al_2O_3$-8$P_2P_5$ mol%) was prepared by solid state reactions of silicic acid, ammonium dihydrogen phosphate, and aluminum hydroxide at 1650°C. Homogeneity was achieved by repeated grinding and melting of the glass sample with sufficient length of heat treatment to eliminate essentially all bubbles.

## II. Phase Equilibria

The soda-cordierite compatibility relations were investigated in the region from 0-15 wt% soda and from 840-1030°C. Samples were prepared by grinding and mixing appropriate amounts of sodium carbonate and cordierite in acetone with a glass mortar and pestle, drying, and packing into platinum tubes. The samples were fired at 840 and 930°C in air for 90 and 63 h, respectively. The samples fired at 1030°C were first calcined at 800°C to remove the carbon dioxide, and then sealed in platinum tubes, followed by the heat treatment at 1030°C for 62 h. X-ray diffraction was used for phase identification.

Table 1. lists the phase assemblages resulting from the soda-cordierite reactions at the temperatures and compositions investigated. The results indicate that cordierite was decomposed by soda to form nepheline and forsterite according to the reaction

$$2MgO\cdot 2Al_2O_3\cdot 5SiO_2 + xNa_2O = x(Na_2O\cdot Al_2O_3\cdot 2SiO_2) + 0.5x(2MgO\cdot SiO_2) + (1\text{-}0.5x)(2MgO\cdot 2Al_2O_3\cdot 5SiO_2)$$

where x is less than 2. Therefore, the amount of soda necessary to completely decompose cordierite is equivalent to a soda content of 17.5 wt%. X-ray diffraction analysis show that the relative intensities of the forsterite and nepheline peaks increased with increasing alkali concentration while those of cordierite decreased. At 15 wt% soda, only traces of cordierite were detected at all temperatures, and the phase assemblage was mainly composed of nepheline and forsterite.

Table 1. Phase Analysis of Compositions Prepared in the Soda-Cordierite System

| Composition (wt%) | | | |
|---|---|---|---|
| Cordierite | Soda | Temperature (°C) | Phases |
| 95 | 5 | 840 | cordierite, nepheline, forsterite (trace) |
| | | 930 | cordierite, nepheline, forsterite (trace) |
| | | 1030 | cordierite, nepheline, forsterite (trace) |
| 90 | 10 | 840 | cordierite, nepheline, forsterite |
| | | 930 | cordierite, nepheline, forsterite |
| | | 1030 | cordierite, nepheline, forsterite |
| 85 | 15 | 840 | cordierite (trace), nepheline, forsterite |
| | | 930 | cordierite (trace), nepheline, forsterite |
| | | 1030 | cordierite (trace), nepheline, forsterite |

## III. Corrosion Kinetics

The corrosion kinetics of the materials exposed to alkali vapors will be initiated upon the arrival of the purchased specimens.

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