Creep of hot-pressed silicon nitride

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Creep tests were undertaken on hot-pressed silicon nitride in the temperature range 1200 to 1400°C. The activation energy for creep was determined to be 140 kcal mol⁻¹ and the stress exponent of creep rate was 1.7. The creep behaviour is ascribed to grain-boundary sliding accommodated by void deformation at triple points and by limited local plastic deformation. Electron microscopic evidence supporting this mechanism is presented.

1. Introduction
In recent years there has been considerable interest in hot-pressed silicon nitride because of its high strength. It has emerged as a prime candidate material for high efficiency gas turbines, and its creep behaviour is an essential design parameter.

Little or no creep data have been reported in the open literature on silicon nitride. In 1961 the creep characteristics of a low-strength material were studied by Glenny and Taylor [1], and Stokes et al. [2] made passing mention of the relative creep resistance of high- and low-strength silicon nitride materials in 1972. More recently, tensile and bend data generated by Westinghouse [3] have become available.

Creep of materials at high temperatures and low stresses can generally be divided into three creep-rate regimes. The initial deformation is rapid (stage I), and the creep rate subsequently decreases until the second stage (II) is reached, wherein the creep rate remains constant. Finally, in stage III, the creep rate increases again, producing cracks followed by failure. Most ceramics show the first two stages. This work was undertaken to investigate stage II or "steady state" creep of hot-pressed silicon nitride and the microstructural changes associated therewith.

2. Experimental
2.1. Material characterization
The Si₃N₄ material was characterized by spectrographic analysis, bulk density measurements, grain size determinations and X-ray analysis. Samples for spectrographic analysis were ground in a silicon carbide mortar and pestle and dissolved in HF.

Spectrographic analysis revealed that the material was 97% silicon nitride. The major impurities were Ca (0.04 wt %), Mg (0.7 wt %), Fe (0.4 wt %), and Al (0.4 wt %). The density was found to be 98% of the theoretical value. Sections for grain size determination were mounted in plastic and rough polished with 400 grit diamond. Final polishing was undertaken on 0.25 μm diamond. After polishing, the samples were removed from the mount and etched in a mixture of HF, HNO₃, and H₂O₂ (1:1:3) at 80°C for 15 min. Shadowed replicas of the etched surface, made by the two-stage carbon replica technique, were examined in the electron microscope and grain sizes determined by the intercept method. About 85% of grains were equi-axed with diameter in the range 0.5 to 2 μm. The remainder were elongated with lengths in the range 1 to 5 μm large. X-ray diffraction showed that the β and α phases of silicon nitride were present. The energy-dispersive X-ray analysis technique was used to identify the intergranular glassy phase. It was found to be a compound of Mg, Ca, and Fe silicates.

2.2. Creep testing
All specimens were taken from a single hot-pressed billet to eliminate variables such as different chemical compositions and thermal history. The material was received in the form of (1 1/2 × 1 1/2 × 4) in. bars.

Creep tests were conducted in four-point bending with silicon carbide knife edges and a
templet was used to centrally locate the samples. The desired load was applied by lowering appropriate slotted weights on to a load plate located on top of the sample loading column. Counter weights were used to balance the load-free column weight. A silicon carbide clamshell furnace was used to heat the sample to $1400 \pm 5^\circ$C and a Pt/Pt-Rh thermocouple measured the specimen temperature. Samples were creep tested between 1200 and $1400^\circ$C at stresses between 8000 and 25 000 psi*. Maximum deformation was limited to about 3.0 % and an LVDT utilized to monitor the creep deflection. The LVDT was mounted rigidly under the load plate.

A series of isothermal creep tests was undertaken for times up to 250 h (standard creep tests). In addition, individual specimens were creep tested by (a) stress cycling at constant temperature and (b) temperature cycling wherein the stress was held constant and the temperature changed incrementally. The former was used to evaluate the stress exponent of the creep rate and the latter to determine the activation energy for the creep process.

### 2.3. Electron microscopy

Samples for the electron microscopic analysis were taken from the tension edge of crept specimens. The first step involved the production of a geological thin section by grinding to 20 $\mu$m on a diamond wheel. The material was then ion-thinned until a small perforation was observed in the centre of the foil. A graphite film was evaporated onto the specimen before introduction into the transmission electron microscope.

### 3. Results

Creep data for the specimens tested are summarized in Table I. Typical standard creep curves at 1260 and $1300^\circ$C at a stress of 10 000 psi are shown in Fig. 1. The regimes of creep are readily distinguishable. The transients in all the creep tests were quite long (40 to 50 h) and, therefore, long-time tests (up to 250 h) were conducted.

The outer fibre stress and strain in bending specimens were calculated using the Timoshenko [4] elastic equations. The use of elastic equations for the evaluation of the plastic strain is questionable but it has been shown in a previous study [5] that for low plastic strains up to 3 %, the elastic equations are valid.

Fig. 2 shows the log-log plot of steady state creep rate versus stress. The relationship between the two is given by the empirical law:

$$\dot{\varepsilon} = A \varepsilon^{-E/R T} \sigma^n$$

(1)

where $E$ is the activation energy.

From the slope of these plots, the stress exponent ($n$) of the creep rate was determined and is given in Table II. The exponent was also found by changing the stress level during a test and using Equation 1. The result of a stress change experiment is illustrated in Fig. 3.

The activation energy for the creep process was also determined in two ways, i.e. constant stress,