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Effects of Oxidation and Hot Corrosion on the Erosion of Silicon Nitride

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The effect of oxidation and hot corrosion on the solid particle erosion was investigated for hot-pressed silicon nitride using as-polished, pre-oxidized and pre-corroded specimens by molten sodium sulfates. Erosion tests were performed at 22, 500 and 900 ℃ using angular silicon carbide particles of mean diameter 100 ㎛. Experimental results show that solid particle erosion rate of silicon nitride increases with increasing temperature for as-polished or pre-oxidized specimens in consistent with the prediction of a theoretical model. Erosion rate of pre-oxidized specimens is lower than that of as-polished specimens at 22 ℃, but it is higher at 900 ℃. Lower erosion rate at 22 ℃ in the pre-oxidized specimens is attributed due to the blunting of surface flaws, and the higher erosion rate at 900 ℃ is due to brittle lateral cracking. Erosion rate of pre-corroded specimens decreases with increasing temperature. Less erosion at 900 ℃ than at 22 ℃ is associated with the liquid corrosion products sealing off pores at 900 ℃ and the absence of inter-granular crack propagation observed at 22 ℃.

Keywords : *solid particle erosion, oxidation, hot corrosion, temperature dependence*

1. Introduction

Silicon nitride is a candidate material for the applications involving the solid particle erosion at elevated temperatures. The examples of such applications include the components in hot section of a gas turbine engine. The components are often degraded by the interaction between the erosion and oxidation or hot corrosion processes. Blunting of surface flaws occurs during oxidation and leads to strength increase in hot-pressed silicon nitride when thin protective oxide film is formed.[1)] On the other hand, the hot corrosion attack by sodium sulfates is known to reduce the strength of this material by the formation of corrosion pits and dissolution of grain boundaries.[2)]

The solid particle erosion behavior or the temperature dependence of erosion rate has been reported for silicon nitride.[3)-5)] The temperature increases the erosion rate of hot-pressed silicon nitride,[3)] whereas it decreases the erosion rate in the sintered[4)] and reaction bonded silicon nitride.[5)] This has been explained in terms of the variation in hardness and fracture toughness with temperature. The results, however, were obtained from polished specimens in the absence of oxidation or hot corrosion effects, and limited information is available for the combined effects of erosion and oxidation or hot corrosion in silicon nitride materials. In this work, the influence of oxidation and hot corrosion on the solid particle erosion of hot pressed silicon nitride was investigated using as-polished, pre-oxidized and pre-corroded specimens by sodium sulfates.

2. Experimental

A hot-pressed, high purity(greater than 98.5%) silicon nitride was used in this work. The material was supplied in disc form with the following properties: density ρ= 3.2 gcm^{-3}; hardness H=1800 kg/mm^2, fracture toughness K_{IC}= 5.5 $MPam^{1/2}$. The discs were cut into pieces to give specimens of dimensions 20 X 20 X 5 mm^3. The specimens were mechanically ground and polished successively using diamond paste up to 3 ㎛. Some of polished specimens were then either pre-oxidized or pre-corroded in air for 60 h at 1000 ℃. Prior to this treatment, the specimens to be pre-corroded were heated to 200 ℃ and coated with a thin sodium sulfate layer by air brushing a saturated solution. Corrosion products were then polished flat before erosion testing to provide a smooth surface. A smooth layer of glass 30 to 40 ㎛ thick remained on all samples after polishing.

Solid particle erosion tester used in this work consists of a screw type particle feeder, gas heater and resistance

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heated furnace. The carrier gas is air which is heated to the desired temperature and passes through acceleration tube, and then mixes with particles before reaching the 4 mm internal diameter alumina nozzle. The acceleration tube, nozzle and specimen holder are all housed in a three zone furnace heated to test temperature. The particle velocity is controlled utilizing the gas pressure and measured using the rotating double discs.[6]

Erosion tests were conducted at 22, 500 and 900 ℃ using 50 g of angular silicon carbide particles of mean diameter 100 ㎛. After completion of the test, the specimen was furnace cooled to room temperature, dried after ultrasonic cleaning and then weighed to an accuracy of 10^{-5} g using a microbalance. Erosion rate was calculated by dividing the specimen mass loss by the total mass of erodent particles impacted.

3. Results and discussion

Fig. 1 shows the results of as-polished silicon nitride with temperature at an impact velocity of 100 ms^{-1} under normal impingement condition. The solid particle erosion rate of this specimen increases with increasing temperature. The increase in erosion rate is moderate at 500 ℃, but sharp at 900 ℃. The erosion rate at 22 ℃ is approximately 60% of that at 900 ℃. The variation in the erosion rate of this specimen with temperature is in consistent with the previous results,[3] but in contrast to those of sintered and reaction bonded silicon nitrides.[4),5)]

The temperature dependence of erosion rate for as-polished silicon nitride may be associated with the variation of fracture toughness and hardness with temperature as theoretical models predict.[7)-9)] The fracture toughness of a similar hot-pressed silicon nitride has been reported to decrease moderately up to 600 ℃, then decreases sharply with increasing temperature.[9)] Hardness is also known to decrease with increasing temperature though the rate of change with temperature is dependent upon the temperature range.[11)] Thus, the increase of erosion rate with temperature can be predicted roughly by dynamic model.[7)] Erosion rate($\triangle E$) is proportional to the power law relationship by this model

$$\triangle E \propto V^n D^{2/3} \rho^p (K_{IC})^{-4/3} (H)^{-0.25}$$

where, V, D and ρ are the velocity, size(mean diameter) and density of erodent particle, respectively, and K_{IC} and H are the fracture toughness and hardness of the target material, respectively. For a fixed test condition in which V, D and ρ are kept constant like in this work, erosion rate is predicted to increase with decreasing fracture toughness or hardness.

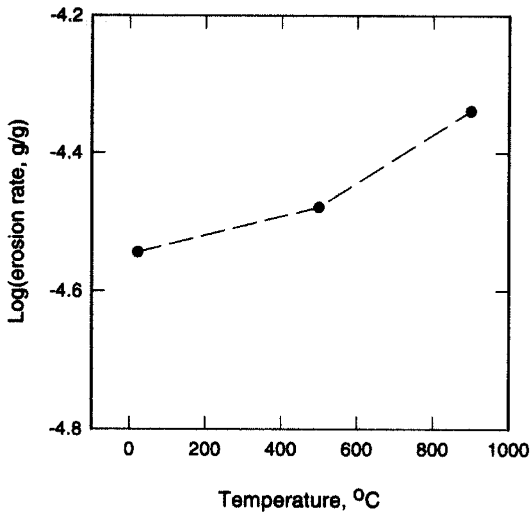


Fig. 1. Variation in erosion rate with temperature for as-polished pecimens eroded at a velocity of 100 ms^{-1} under normal impingement condition.

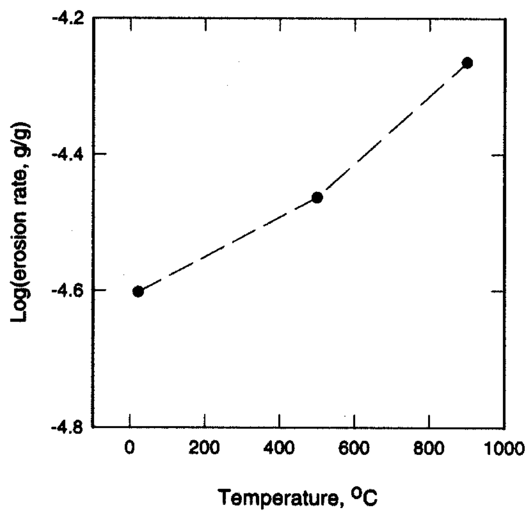


Fig. 2. Variation in erosion rate with temperature for pre-oxidized specimens eroded at a velocity of 100 ms^{-1} under normal impingement condition.

The variation of erosion rate with temperature in pre-oxidized specimens is similar to that of as-polished specimens as shown in Fig. 2. The erosion rate again increases with increasing temperature, and the increase of erosion rate in this specimen is moderate at 500 ℃ and large at 900 ℃. Comparison of this result with that of as polished specimen reveals that the erosion rate of pre-oxidized specimens is lower than that of as-polished specimens at 22 ℃, where-as it is higher at 900 ℃.

For the investigation of this in detail, eroded surfaces of these specimens were examined by SEM. For as-

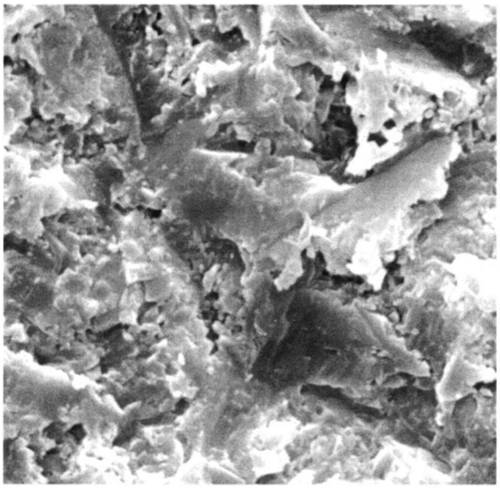


Fig. 3. SEM micrograph of a typical surface(3660X) for as-polished specimens eroded at a velocity of 100 ms^{-1} under normal impingement condition at 900 ℃.

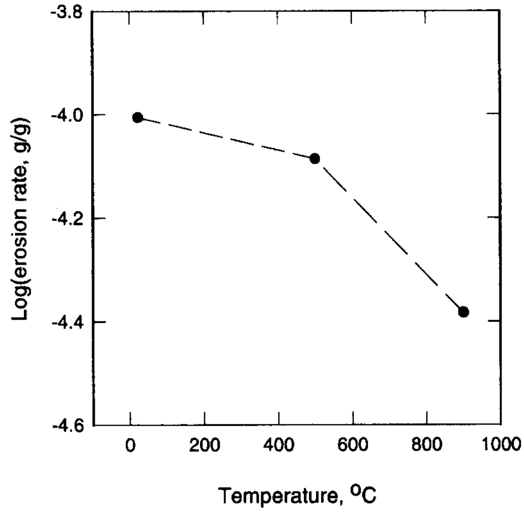


Fig. 5. Variation in erosion rate with temperature for pre-corroded specimens eroded at a velocity of 100 ms^{-1} under normal impingement condition.

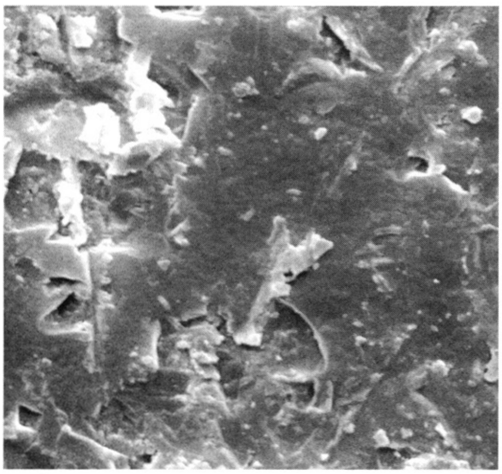
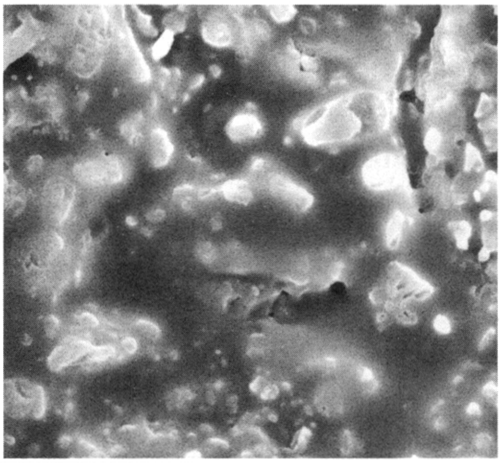


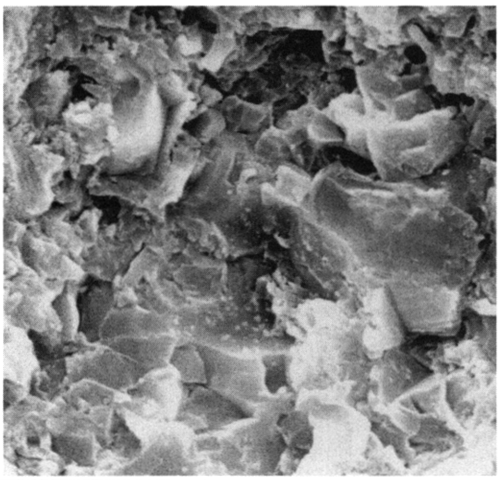
Fig. 4, SEM micrograph of a typical surface(1870X) for pre-oxidized specimens eroded at a velocity of 100 ms^{-1} under normal impingement condition at 900 ℃.

polished specimens, erosion occurs by cutting and plowing both at 22 and 900 ℃ as in Fig. 3. Cutting and plowing is a major erosion mechanism for pre-oxidized specimens, but erosion occurs also by lateral cracking at 900 ℃ for this specimen as in Fig. 4. The lower erosion rate at 22 ℃ in pre-oxidized specimen compared to as-polished specimen is presumably due to the blunting of surface flaws during oxidation though it is not observed in SEM micrographs. Blunting of strength limiting surface flaws is known to occur in hot-pressed silicon nitride when thin protective oxide film is formed on the surface mostly during initial stages of oxidation or following exposure at low temperatures.[1] However, the beneficial blunting effect may not be significant at 900 ℃. Rather, chipping by lateral cracks may contribute to the erosion process resulting in higher erosion. Lateral cracks are presumed to generate from brittle glass phases in the pre-oxidized specimens at this temperature.

The temperature dependence of erosion rate for pre-corroded specimens is different from those of as-polished or pre-oxidized specimens as shown in Fig. 5. The erosion rate of pre-corroded specimens decreases with increasing temperature. Erosion rate at 900 ℃ was about 40 % of that at 22 ℃. Main cause for this may be due to the presence of corrosion products such as sodium silicates that would melt and form thin layer at this temperature. Erosion may occur less effectively when the specimen surface is covered with liquid film as in many metals. The liquid film is observed to seal off pores(Fig. 6(a)), which otherwise may induce stress concentration and facilitate crack initiation during erosion. Numerous small pores were observed in the specimens eroded at 22 ℃, but such pores were not observed for the specimens tested at 900 ℃. In addition, inter-granular cracks were also observed in these specimens tested at 22 ℃(Fig. 6(b)). The presence of these cracks may also be responsible for the higher erosion of pre-corroded specimens at 22 ℃ compared to that at 900 ℃. This is consistent with the observation of grain boundary dissolution and resulting strength reduction in hot-pressed silicon nitride.[1] Thus, the effects of the variation of fracture toughness and hardness with temperature are not evident in the solid particle erosion of pre-corroded specimens unlike in as-polished and pre-oxidized specimens.



(a)



(b)

Fig. 6. SEM micrographs of a typical surface for pre-corroded specimens eroded at a velocity of 100 ms^{-1} under normal impingement condition at 900 ℃(1850X)(a) and at 22 ℃(1900X)(b).

4. Summary and conclusions

Conclusions drawn from solid particle erosion of silicon nitride in this work can be summarized as follows:

(1) Solid particle erosion rate of silicon nitride increases with increasing temperature for as-polished or pre-oxidized specimens in consistent the prediction of a theoretical model.

(2) Erosion rate of pre-oxidized specimens is lower than that of as-polished specimens at 22 ℃, but it is higher at 900 ℃. Lower erosion rate at 22 ℃ in the pre-oxidized specimens is attributed due to the blunting of surface flaws, and the higher erosion rate at 900 ℃ is due to brittle lateral cracking.

(3) Erosion rate of pre-corroded specimens decreases with increasing temperature. Less erosion at 900 ℃ than at 22 ℃ is associated with the liquid corrosion products that seal off pores at 900 ℃ as well as with the absence of inter-granular crack propagation observed at 22 ℃.

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