LETTER

## Solid particle erosion of hot-pressed silicon nitride

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Many potential applications of silicon nitride involve the solid particle erosion at elevated temperatures. The typical examples of such applications are the key components in hot section of a gas turbine engine. Degradation of the components often occurs by the interaction between the erosion and hot corrosion processes by sodium sulfates. The hot corrosion attack is known to reduce the strength of silicon nitride by the formation of corrosion pits and dissolution of grain boundaries [\[1\]](#page-2-0).

The temperature dependence of solid particle erosion behavior has been reported for silicon nitride [[2–4\]](#page-2-0). The temperature increases the erosion rate of hot-pressed silicon nitride [\[2](#page-2-0)], whereas it decreases the erosion rate in the sintered [[3\]](#page-2-0) and reaction bonded silicon nitride [[4\]](#page-2-0). 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 solid particle erosion of hot-pressed silicon nitride was investigated for as-polished, pre-oxidized and pre-corroded specimens by sodium sulfates.

The target material used in this investigation is a hotpressed, high purity silicon nitride (with purity of greater than 98.5%) supplied in disc form with the following properties: density  $\rho = 3.2$  g cm<sup>-3</sup>; hardness  $H = 1800$  kg mm<sup>-2</sup>, fracture toughness  $K_{\text{IC}} = 5.5 \text{ MPam}^{1/2}$ . The discs were cut into pieces to give specimens of dimensions  $20 \times 20 \times$ 5 mm<sup>3</sup>. The specimens were mechanically ground and pol-

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ished successively using diamond paste up to  $3 \mu$ m. A typical microstructure of the specimens is shown in Fig. [1](#page-1-0). Some of polished specimens were then either oxidized or corroded in air at  $1000^\circ$  C for 60 h. Prior to this exposure, the specimens to be corroded were heated to  $200^{\circ}$  C 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 30–40  $\mu$ m thick remained on all samples after polishing.

Erosion tester used in this work consists of a screw type particle feeder, gas heater and resistance 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 3 zone furnace heated to test temperature. The particle velocity is controlled utilizing the gas pressure and measured using the rotating double discs [[5\]](#page-2-0).

Erosion tests were conducted under normal impingement condition at 22, 500 and 900  $^{\circ}$ C using 50 g of angular silicon carbide particles of mean diameter 100 µm. After completion of the test, the specimen was furnace cooled to room temperature 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.

Figure [2](#page-1-0) shows the variation in the erosion rate of hotpressed silicon nitride with temperature at an impact velocity of  $100 \text{ m s}^{-1}$ . The erosion rate of as-polished specimens tends to increase with increasing temperature in consistent with the previous results [[2\]](#page-2-0), but in contrast to the result of sintered and reaction bonded silicon nitrides  $[3, 4]$  $[3, 4]$  $[3, 4]$  $[3, 4]$ . The increase in erosion rate is moderate at 500 $^{\circ}$  C,

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Fig. 1 A typical microstructure of the silicon nitride specimen used in the present work  $(1900\times)$ 

but sharp at  $900^{\circ}$  C. The erosion rate at  $22^{\circ}$  C is approximately  $60\%$  of that at  $900^{\circ}$  C.

The temperature dependence of erosion rate for as-polished silicon nitride may be related with the variation of fracture toughness and hardness with temperature as theoretical models predict [\[6–8\]](#page-2-0). The fracture toughness of a similar hot-pressed silicon nitride has been reported to decrease moderately up to 600°C, then decreases sharply with increasing temperature [\[9](#page-2-0)]. Hardness also decreases with increasing temperature though the rate of change with temperature is dependent upon the temperature range [[10\]](#page-2-0). Dynamic model predicts that erosion rate of brittle material increases with decreasing fracture toughness or hardness following a power law relationship,  $E \propto (K_{\rm IC})^{-4/3} (H)^{-0.25}$ , where,  $K_{\text{IC}}$  and H are the fracture toughness and hardness of the target material, respectively. Thus, the temperature dependence of erosion rate can be explained by this model, but fracture surface needs to be examined for the applica-

-3.8  $-4.0$ Log(erosion rate, g/g) Log(erosion rate, g/g) -4.2 -4.4 -4.6 -4.8 0 200 400 600 800 1000 Temperature, °C

Fig. 2 Variation in erosion rate with temperature for as-polished  $(①)$ , pre-oxidized ( $\circ$ ) and pre-corroded ( $\nabla$ ) specimens eroded at a velocity of  $100 \text{ ms}^{-1}$ 

tion of this model to the prediction of erosion behavior of silicon nitride since the model was developed based on brittle erosion behavior.

The temperature dependence of erosion rate in preoxidized specimens is similar to that of as-polished specimens. The erosion rate tends to increase with increasing temperature, and the increase of erosion rate in this specimen is moderate at  $500^{\circ}$  C and larger at  $900^{\circ}$  C. An important point to be noted is that the erosion rate of preoxidized specimens is lower than that of as-polished specimens at  $22^{\circ}$  C, where-as it is higher at  $900^{\circ}$  C. For the investigation of this in detail, eroded surfaces of these specimens were examined by SEM. For as-polished specimens, erosion occurs by cutting and plowing both at 22 and  $900^{\circ}$  C as in Fig. 3, indicating that dynamic model is not supported by fractographs. On the other hand, for preoxidized specimens, erosion occurs by cutting and plowing at 22° C, while it occurs by lateral cracking in addition to cutting and plowing at  $900^{\circ}$  C as in Fig. [4.](#page-2-0) The lower erosion rate at 22° C in preoxidized specimen compared to as-polished specimen is presumably due to the blunting of surface flaws during oxidation, and the higher erosion rate in the this specimen at  $900^{\circ}$  C may be due to brittle lateral cracking. 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 [[11\]](#page-2-0). The beneficial blunting effect, however, may not be present at 900° C. Rather, chipping by lateral cracks may contribute significantly to the erosion process at this temperature. Lateral cracks are presumed to be generated from brittle glass phases in the pre-oxidized specimens at this temperature.



Fig. 3 SEM micrograph of a typical surface  $(3690\times)$  for as-polished specimens eroded at a velocity of  $100 \text{ ms}^{-1}$  at  $900^{\circ}$  C

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Fig. 4 SEM micrograph of a typical surface  $(1860\times)$  for pre-oxidized specimens eroded at a velocity of  $100 \text{ ms}^{-1}$  at  $900^{\circ}$  C

The pre-corroded specimens show different temperature dependence from that of as-polished or pre-oxidized specimens. The erosion rate of pre-corroded specimens decreases with increasing temperature. Erosion rate at 900 $\degree$  C was about 40% of that at 22 $\degree$  C. Main cause for this may be due to the 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. The liquid film is observed to seal off pores (Fig. 5a) which otherwise may induce stress concentration and facilitate crack initiation during erosion. Numerous small pores were observed in the specimens eroded at  $22^{\circ}$  C, but such pores were not observed for the specimens tested at  $900^{\circ}$  C. In addition, intergranular cracks were also observed in the specimens tested at  $22^{\circ}$  C (Fig. 5b). The presence of these cracks may also be responsible for the higher erosion rate of pre-corroded specimens at 22° C compared to that at 900° C. 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.

In summary, erosion rate of silicon nitride increases with increasing temperature for as-polished or pre-oxidized specimens. This can be explained by dynamic model, but it is not supported by fractographs. Erosion rate of preoxidized specimens is lower than that of as-polished specimens at  $22^{\circ}$  C, while it is higher at  $900^{\circ}$  C. Lower erosion rate at  $22^{\circ}$  C in the former specimens is attributed due to the blunting of surface flaws, and the higher erosion rate at 900° C is due to brittle lateral cracking. Erosion rate of pre-corroded specimens, however, decreases with



Fig. 5 SEM micrographs of a typical surface for pre-corroded specimens eroded at a velocity of  $100 \text{ ms}^{-1}$  at  $900^{\circ}$  C (1830 $\times$ ) (a) and at  $22^{\circ}$  C (3740 $\times$ )(**b**)

increasing temperature. Less erosion at  $900^{\circ}$  C than at  $22^{\circ}$  C is associated with the liquid corrosion products that seal off pores at  $900^{\circ}$  C as well as with the absence of intergranular crack propagation observed at  $22^{\circ}$  C.

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