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Impact damage behaviour of CVD-coated silicon nitride for gas turbines

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Impact tests were conducted on the silicon nitride substrates coated with Si_3N_4 and SiC by chemical vapour deposition (CVD). For both 100- and 200-μm-thick Si_3N_4-coated silicon nitride, Hertzian crack extension was reduced by debonding at the interface. Although Hertzian crack extension was not reduced for 100-μm-thick SiC-coated silicon nitride, it was reduced for 200-μm-thick SiC-coated silicon nitride. Theoretical calculations suggest that debonding at the interface consumed the fracture energy of Hertzian crack extension in the case of Si_3N_4 coatings, but it was observed that Hertzian cracks were not arrested at the interface.

1. Introduction

Particle impact damage and oxidation are the most serious problems seen in silicon nitride for gas turbine use [1, 2]. Oxidation is thought to cause fracture of the silicon nitride surface in a combustion environment, resulting in a strength degradation. Although the oxidation process has been studied and the associated phenomena have been identified recently [3, 4], impact damage behaviour is still not well understood. Fracture is thought to be caused by flying carbon particles which are generated in the combustion chamber and strike the turbine, resulting in complete failure.

Several studies have focused on impact damage problems and the damage process in silicon nitride applied to turbines [1, 2, 5, 6]. Impact phenomena have been studied using the Hertzian cone fracture theory [7] based on elastic stress analysis. Damage behaviour has also been analysed using elastic and plastic coefficients such as Young's modulus and hardness [2, 5]. However, a unified mechanism for explaining impact fracture has yet to be defined [8].

A chemical vapour deposition (CVD) method has been developed recently to overcome oxidation on ceramic components in a gas turbine project [9–11]. Chemically vapour deposited films are thought to be effective in preventing oxidation damage to silicon nitride [10, 11]. In this work, the impact damage behaviour of silicon nitride coated with CVD films was examined in preliminary experiments with reference to studies conducted on other coated materials systems [12, 13]. The impact damage behaviour of silicon nitride substrates coated with silicon nitride or silicon carbide CVD films was examined.

2. Experimental procedure

2.1. Materials

Commercially available gas-pressure-sintered silicon nitride (EC152; NGK Spark Plug Co., Komaki, Japan) was used in this study. Partially stabilized zirconia (Toso Co., Tokyo, Japan) spheres 1.0 mm in diameter were used in the spherical particle impact test. Silicon nitride specimens, 14 mm in diameter and 2 mm in thickness were polished with diamond paste (3 μm) to eliminate machining damage and to produce flat and parallel surfaces. Data furnished by the manufacturers on material properties are listed in Table I.

2.2. Chemical vapour deposition

CVD using a commercial process was performed by Mitsui Engineering and Shipping Co. Ltd. CVD [10, 11] was carried out under reduced pressure to form Si_3N_4 or SiC films that were 100 and 200 μm in thickness.

2.3. Impact test [5]

The experimental apparatus used is shown schematically in Fig. 1. A partially stabilized zirconia sphere was attached to the end of a sabot, and the sabot was set in a pistol. Helium gas was introduced to raise the gas pressure in the chamber to the specified level (3–5 MPa). When the diaphragm was broken by a

TABLE I Material properties[a]

Properties	Target	Spheres
	Silicon nitride	PSZ
Density ($g\,cm^{-3}$)	3.26	6.05
Poisson's ratio	0.26	0.3
H_V (GPa)	15.4	12.5
Young's modulus (GPa)	310	200
Bending strength (MPa) (4-point bend test)	920	1100
K_{IC} ($MPa\,m^{1/2}$)	6.0	7.5

[a] Data furnished by manufacturers.

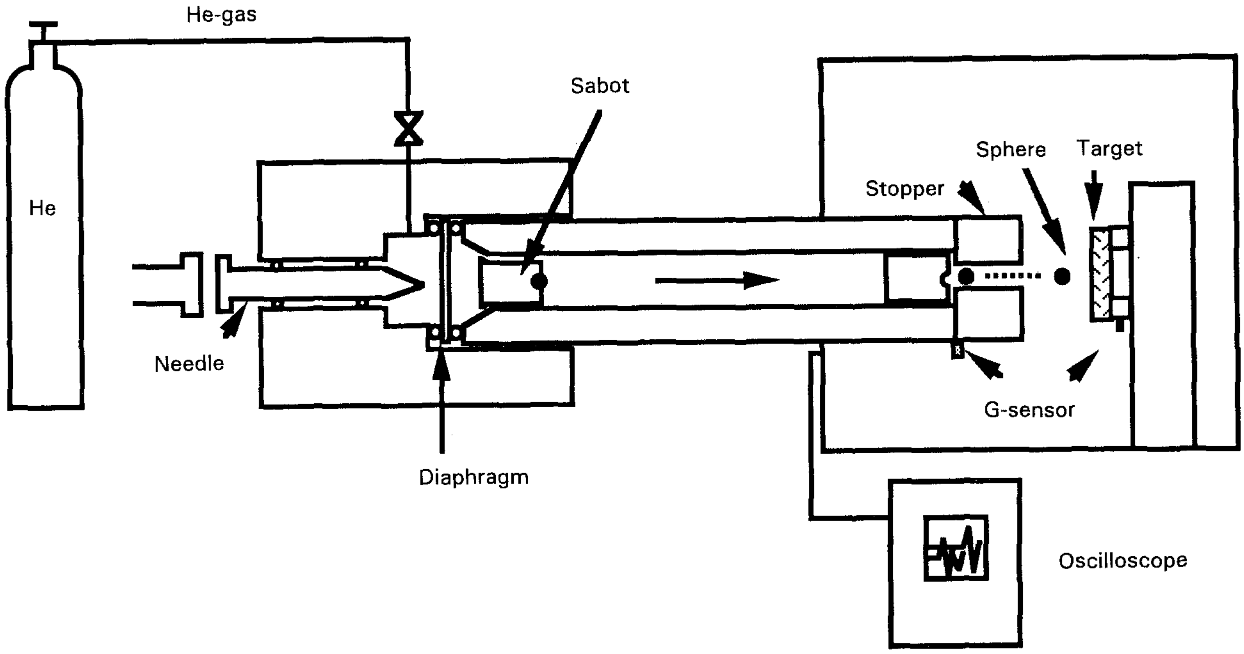


Figure 1 Impact test apparatus.

needle, the gas was released toward the pistol, thus driving the sabot to the end of a steel pipe where its immediate stop ejected the sphere toward the target. One impact was made under each set of conditions at room temperature. The velocity of the PSZ sphere was analysed by piezoelectric sensors attached to the stopper and specimen holder. The velocity was detected for each sabot firing, based on the time-of-flight principle.

2.4. Post-impact evaluation

After the impact test, surface damage to the silicon carbide- or silicon nitride-coated silicon nitride substrate was investigated using an optical microscope and a scanning electron microscope (SEM). Cross sections of the substrates under the impact site were also examined in the same manner.

3. Results and discussion

3.1. CVD films

The thickness of CVD films was 100 and 200 μm. The Si_3N_4 films had an α-type crystal structure, and the growth direction was found to be random by X-ray diffraction. On the other hand, SiC films showed a β-type crystal structure, and the growth direction of the crystals was confirmed as [1 1 1]. Fig. 2a and b shows typical surface morphologies of the CVD coating films, and Fig. 2c and d shows cross sections of the coated films. Large cracks are observed in the SiC film which were caused by thermal stress due to the mismatch in thermal expansion coefficients.

3.2. Surface damage

Typical examples of the surface damage observed after the spherical impact test are shown in Fig. 3a–c. Fig. 3a shows the surface damage of a silicon nitride substrate for comparison. Ring cracks and radial cracks were observed in the substrates near the impact site. Ring cracks were thought to be generated by radial stress during loading. Radial cracks were thought to be generated by tangential stress, when the stress state changed from the radial direction to tangential after sphere rebound [2].

Silicon nitride films showed debonding at the interface with the formation of radial and ring cracks at impact velocities over $400\ m\,s^{-1}$, and evidence of material removal was observed at a $500\ m\,s^{-1}$ impact velocity (Fig. 3b). On the other hand, SiC films showed signs of material removal at impact velocities greater than $500\ m\,s^{-1}$ and radial cracks were generated without any debonding at the interface (Fig. 3c).

3.3. Cracks generated under impact site

Test discs were cut from the impact site and polished using diamond paste (6 and 3 μm). Fig. 5 shows the crack depth from the surface based on microscopic observation (Fig. 4) for the specimens coated with films of two different thicknesses. Specimens coated with 100-μm-thick Si_3N_4 showed reduced crack extension at impact velocities under $500\ m\,s^{-1}$, and specimens coated with 200-μm-thick Si_3N_4 showed reduced crack extension at impact velocities under $600\ m\,s^{-1}$. On the other hand, SiC films did not exhibit the crack arresting effect, resulting in Hertzian crack extension. The reduction in Hertzian crack extension for Si_3N_4 film is thought to be explained by the debonding phenomena at the interface resulting in consumption of the crack extension energy [12, 13].

3.4. Direction of Hertzian crack extension

Fig. 6 shows the crack extension behaviour at the interface of both Si_3N_4- and SiC-coated substrates. Hertzian cone cracks extended from the impact site to the substrate, and the direction of crack extension did

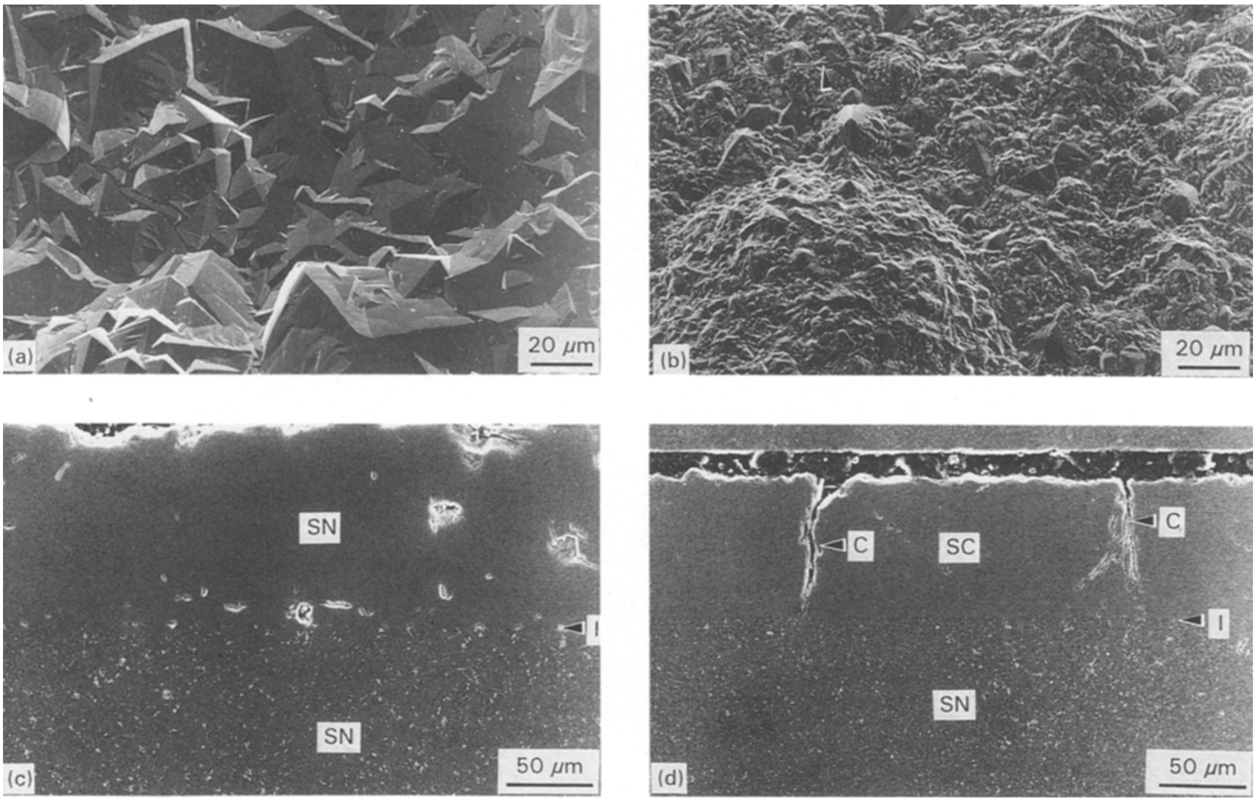
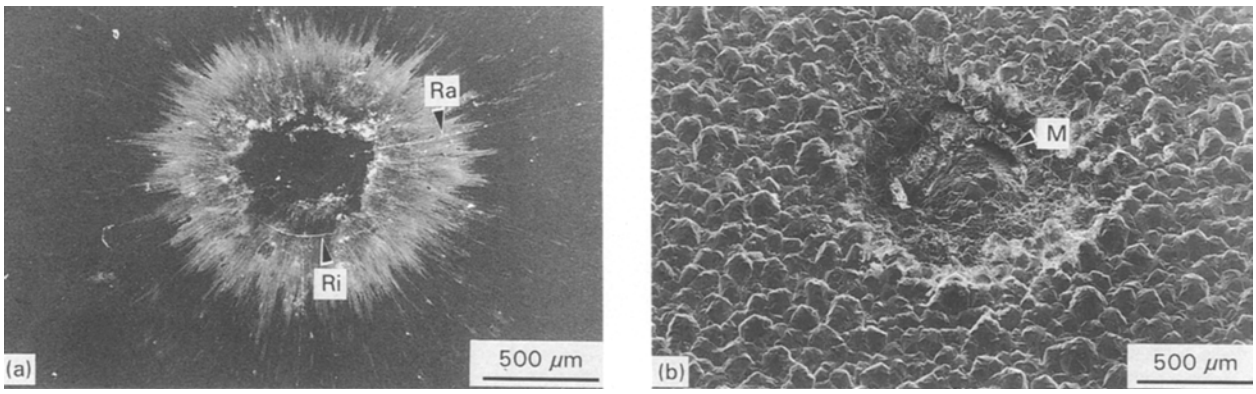


Figure 2 Scanning electron micrographs of CVD films. (a) Surface crystal morphology of Si_3N_4; (b) surface crystal morphology of SiC (arrow: crack formed by thermal expansion coefficient mismatch); (c) cross-section of Si_3N_4 film (SN: Si_3N_4, I: interface); (d) cross section of SiC film (SN: Si_3N_4, SC, SiC, I: interface).



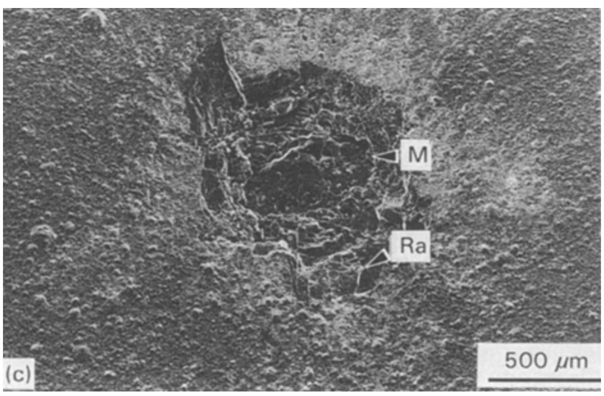


Figure 3 Surface damage morphologies. Silicon nitride substrate ($V = 500\ \mathrm{m\,s^{-1}}$) showing (a) ring cracks and radial cracks; (b) materials removal, M; (c) radial cracks (Ra) and lateral cracks.

not change at the interface, contrary to reports in the literature [12, 13]. Cracks in SiC films resulted from thermal stress due to the difference in the thermal expansion coefficient between the substrate and the film. The extension angle and length of Hertzian cone cracks were not influenced by the cracks caused by thermal stress.

3.5. Comparison of fracture behaviour

The initiated crack shape in both Si_3N_4 and SiC coatings was that of a Hertzian cone crack [7]. Debonding at the interface was observed for silicon nitride-coated substrates but not for SiC-coated ones. According to an analysis by Davis *et al.* [13], normalized energy release rate was calculated from the cone crack shape. The left side of the theoretical curve in Fig. 7 shows only Hertzian cone crack initiation, while the right side shows both Hertzian cone cracks and

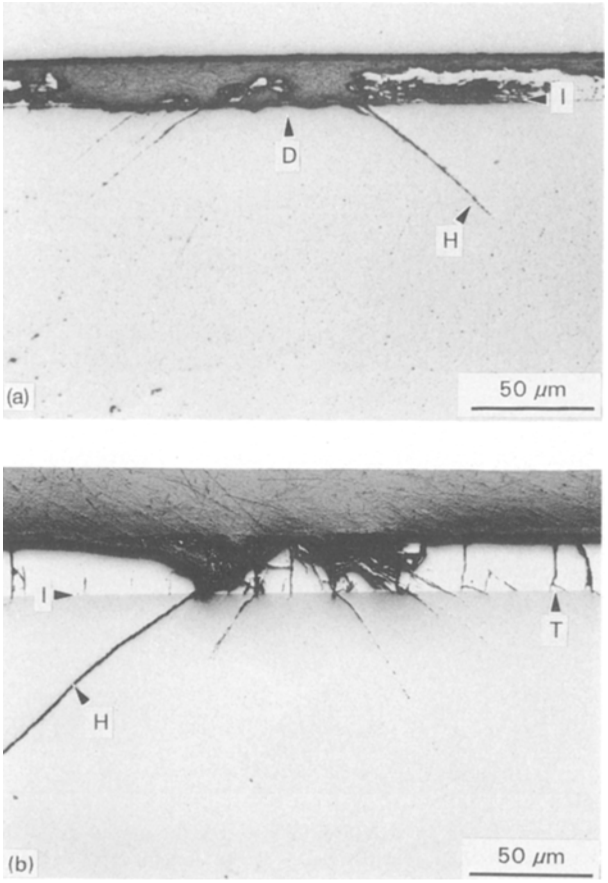


Figure 4 Optical micrographs of cross section. D, debonding; I, interface; H, Hertzian cone crack; T, crack by thermal expansion coefficient mismatch. Silicon nitride coating, (a) $v = 504\ \mathrm{m\,s^{-1}}$; (b) $v = 320\ \mathrm{m\,s^{-1}}$.

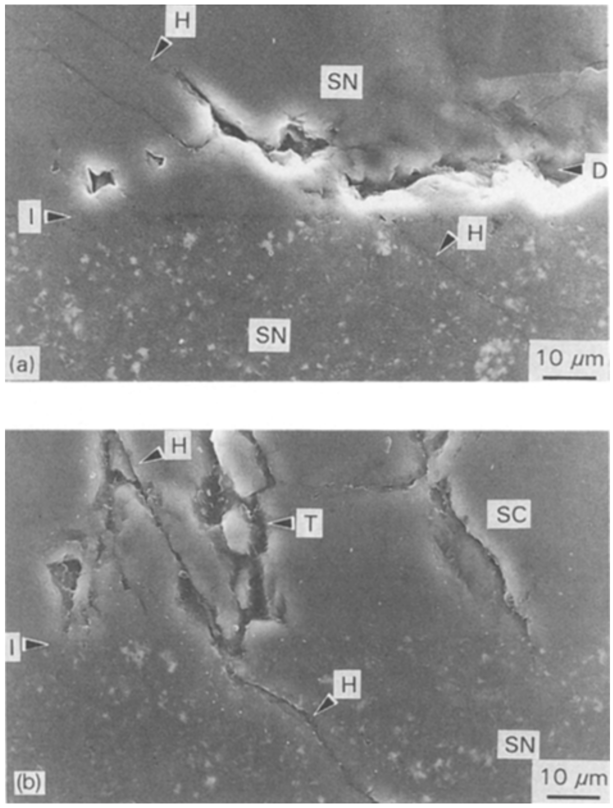


Figure 6 Scanning electron micrographs of cross section. SN: Si_3N_4, SC: SiC, D, debonding; I, interface; H, Hertzian cone crack; T, crack by thermal expansion coefficient mismatch. Silicon nitride coating, (a) $v = 504\ \mathrm{m\,s^{-1}}$; (b) $v = 320\ \mathrm{m\,s^{-1}}$.

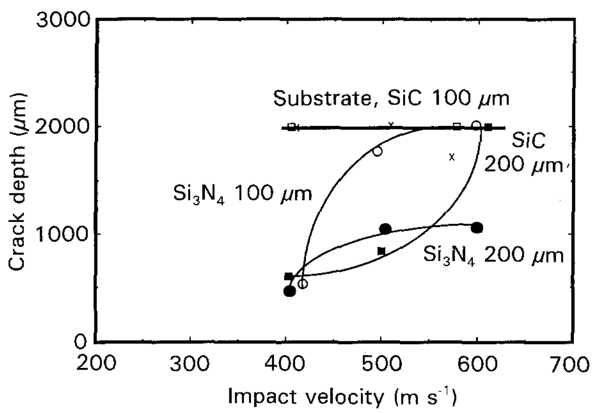


Figure 5 Cone crack depth against impact velocity for coating specimens of two thicknesses. Coating = ○, Si_3N_4, 100 μm; □, SiC, 100 μm; ●, Si_3N_4, 200 μm; ■, SiC, 200 μm; ×, substrate.

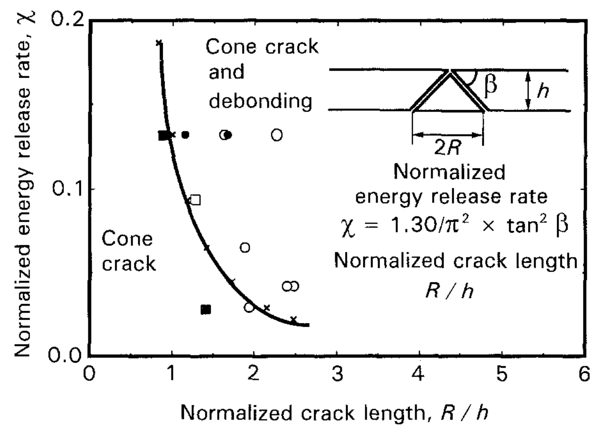


Figure 7 Crack behaviour curve from this experiment compared to those of Davis *et al.* [13]. Coating = ○, Si_3N_4, 100 μm; □, SiC, 100 μm; ●, Si_3N_4, 200 μm; ■, SiC, 200 μm; ×, theoretical curve from [13].

debonding at the interface. On the latter side, debonding behaviour consumes the fracture energy of the impact [13] but Hertzian cone cracks still extend to the substrate. This is because Hertzian cone cracks are typically initiated in 0.2–0.3 μs [14], and the strong bonding effect at the interface between the coating film and the substrate results in the generation of both debonding and Hertzian cone cracks.

4. Conclusions

Impact tests were conducted on silicon nitride substrates CVD-coated with Si_3N_4 or SiC. For both the 100- and 200-μm-thick Si_3N_4 films, Hertzian crack extension was reduced by the occurrence of debonding at the interface. In contrast, Hertzian cone crack extension was not reduced by debonding for the 100-μm-thick SiC films, although it was reduced for 200-μm-thick films. Theoretical calculations indicated that debonding should have occurred in the case of silicon nitride. However, as Hertzian cone cracks continued to extend beyond the coated films, it is presumed that strong bonding was present at the interface between the coating and the substrate.

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