Two types of hot-pressed silicon nitride, one having an amorphous grain-boundary phase (6 wt% yttria, 3 wt% alumina) and the other having a predominantly crystalline grain-boundary phase (8 wt% yttria, 1 wt% alumina), were tested on a split Hopkinson pressure bar with a momentum trap, such that, in each test, the sample was subjected to a single predefined stress pulse and then recovered without being subjected to any other loads. The specimens were loaded repeatedly with a triangular pulse of 3.2 GPa amplitude at a strain rate of approximately 400/s. The dynamic fatigue life of amorphous grain-boundary-phase silicon nitride was observed to be higher than that of the corresponding fatigue life of crystalline grain-boundary-phase silicon nitride. The difference in fatigue lives is correlated to the microstructural damage occurring in both materials.

I. Introduction

Ceramics such as silicon nitride are currently being studied as potential materials for high-temperature structural and mechanical components. Since structural and mechanical components often fail under fatigue, it is important to understand the static, quasi-static, and dynamic fatigue properties of silicon nitride.

To achieve densification in silicon nitride below its dissociation temperature, sintering aids such as CaO, MgO, Al₂O₃, and Y₂O₃ are added to silicon nitride powder. The microstructure of silicon nitride depends upon the characteristics of the starting powder, types and amount of sintering aids, and processing and sintering conditions. Since the properties of silicon nitride vary widely, depending upon its microstructure, it is important to correlate fatigue properties to the corresponding microstructure.

Different types of fatigue tests, such as four-point bending tests, cantilever bending tests, rotating bending tests, and compression-tension tests, have been conducted on silicon nitride. The effects of compression stress on the fatigue life in four-point bending tests was studied by Nikkila and Mantyla. They evaluated the fatigue lives of sintered and hot-pressed silicon nitrides under three different types of loading cycles: (a) sinusoidal, \( R = -1 \) (tension-compression); (b) sinusoidal, \( R = 0 \) (tension); and (c) positive cut sinusoidal, \( R = 0 \) (tension). In type (c) tests no load was applied during the compression portion of the sinusoidal cycle, whereas a complete cycle in (b) consisted of the positive half of the sinusoidal cycle only. For the same maximum tensile stress, the highest fatigue strength values were obtained for the positive cut sinusoidal cycle, and the lowest fatigue resistance was found for the tension-compression cycle. Thus, compressive stresses acting on a material cannot be ignored in determining the fatigue life of a mechanical component. Fatigue failure in these tests was found to originate from preexisting flaws.

The fatigue life data under tension-compression and bending fatigue tests are more scattered than similar fatigue data in metals. This large scatter in the fatigue data is attributed to differences in flaw size and type, in otherwise similar samples. In some of these experiments, the fatigue lives of two identical samples tested under similar conditions differed by as much as a factor of 10, and in another study they differed by as much as 10³ times.

Fatigue crack growth, for silicon nitride fluxed with yttria and alumina, under vacuum in four-point bending tests was studied by Okazaki et al. Their results indicated that it is possible to propagate fatigue cracks in silicon nitride under vacuum. Higher crack growth rates were observed when the material was tested in air compared to vacuum, suggesting that stress-corrosion cracking plays an important role in the fatigue crack growth in silicon nitride, but it alone cannot account for the observed crack growth rates. Based on experimental evidence, it was proposed that fatigue crack growth in ceramics is closely related to the wedge effects created by the intergranular nature of crack propagation, resulting in the formation of rough fracture surfaces, and by the presence of debris particles on the fracture surfaces.

Most of the above-mentioned tests were performed quasi-statically (low strain rate), and the effect of microstructure (grain-boundary phase, fraction of elongated grains, etc.) was not studied in detail. Even though numerous tests have been conducted to study fatigue in silicon nitride, additional tests are needed to investigate static, quasi-static, and dynamic fatigue properties under different loading conditions, and to correlate the observed fatigue behavior to the relevant microstructural features. The results of the above-mentioned tests clearly indicate that compression stress cannot be neglected in determining the fatigue life of silicon nitride.

The purpose of this research was to investigate damage evolution in hot-pressed silicon nitride under dynamic compression fatigue. The modified split Hopkinson pressure bar was used to repeatedly test hot-pressed silicon nitride at a strain rate of approximately 400/s. The modified split Hopkinson pressure bar prevents the reloading of the sample by the stress pulse which would reflect off the free end of the incident bar. This modification allows for the correlation of microstructural damage with the number of fatigue cycles.

The effect of the grain-boundary phase (microstructure) on the dynamic compression fatigue was evaluated by testing two different types of silicon nitrides, one having an amorphous grain-boundary phase, and the other having a predominantly crystalline grain-boundary phase. Apart from the nature and composition of the grain-boundary phase, the microstructure of both materials is nearly the same. Based on the observation of damage in the microstructures, the effect of the grain-boundary structure on the dynamic fatigue life of hot-pressed silicon nitride is explained.

G. Grathwohl—contributing editor

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II. Experimental Procedure

(1) Materials

The two different types of hot-pressed silicon nitrides were provided by Cercom Inc., located in Vista, Ca. Both of the hot-pressed silicon nitrides were fluxed with yttria and alumina in different proportions. One of the materials had an amorphous grain-boundary phase, hereafter called ABP silicon nitride (6% yttria, 3%-alumina-doped material), and the other material with 8% yttria and 1% alumina dopants had a predominantly crystalline grain-boundary phase, hereafter called CBP silicon nitride. The grain-boundary phase refers to the phase that resides between the “pure” grains of silicon nitride; nearly all of the dopants reside within the grain-boundary regions. CBP silicon nitride was made by nitriding a mixture of silicon, yttria, and alumina, and subsequently hot-pressing the nitrided compact. ABP silicon nitride was made by hot-pressing a mixture containing silicon nitride, yttria, and alumina.

(2) Specimen Preparation

For both types of silicon nitride, mechanical test specimens were cut with the compression axis perpendicular to the hot-pressing direction. The dimensions of the test samples were 4 mm by 4 mm by 7.6 mm. Test samples were mounted and rough polished on 40, 30, and 15 μm diamond wheels, and then polished on a cloth with 6 μm diamond paste. Final polishing was performed on a polishing cloth with 1 μm diamond paste. All six faces of the samples were polished in succession by breaking the samples out of the mounts and remounting and polishing. Before conducting a test, the axial length of each sample was measured along four corners. If the difference in the axial length measured at any two corners was more than 0.005 mm, the sample was discarded. A simple stress calculation, using the properties from Table I, shows that if the length of one long edge of the sample is greater than the others by 0.005 mm, that edge experiences approximately 205 MPa higher stress than the other edges. Therefore, in order to keep the stress uniform over the cross-sectional area, it is important for the samples to have flat surfaces, with opposite sides parallel to each other, and adjacent sides perpendicular to each other.

Transmission electron microscope (TEM) foils were prepared from 3 mm diameter rods cut ultrasonically along axial and transverse directions from the tested specimens. These rods were sliced into disks by a thin diamond blade. The slices were hand-ground to less than 70 μm on a silicon carbide paper, and subsequently dimpled on one side to approximately 20 μm. The dimpled samples were then thinned to perforation on an ion-mill using 6 kV argon ions incident on the specimen surface at an angle of 14°. To prevent charging in the TEM, thin carbon was deposit onto the specimen surfaces. Finally, the samples were polished on a cloth with 6 μm diamond paste. Final polishing was conducted at 300 kV on a Philips CM30 microscope.

Scanning electron microscopy (SEM) was performed on fractured samples at 20 kV on a Cambridge S360 microscope, after ultrasonically cleaning the samples in acetone and sputtering them with gold to prevent charging in the SEM.

(3) Dynamic Fatigue Experiments

To study the damage mechanisms which lead to fatigue failure, it is important to correlate the microstructural changes with the number of cycles to which the sample is subjected. Split Hopkinson bars are commonly used to obtain the stress–strain relations for materials over a broad range of high strain rates. In the classical split Hopkinson compression bar, the sample is placed between two identical long bars of circular cross section, and then subjected to a stress pulse which is produced in one of the bars by impacting it with a striker bar at its free end. The tensile pulse which reflects off the sample is subsequently reflected from the free end of the incident bar as a compressive pulse which reloads the sample. Thus, this technique suffers from the drawback of uncontrolled reloading of the sample.

To overcome this limitation, a split Hopkinson compression bar, modified with a momentum trap apparatus, was used here to study damage evolution in silicon nitride at high strain rates. The schematic diagram of the apparatus is shown in Fig. 1. It consists of two 12.7 mm diameter maraging steel bars (incident and transmission bars), a striker bar which is propelled from a gas gun at a predetermined speed, an incident sleeve that is used as a momentum trap, and a reaction mass. The sample is sandwiched between the incident and the transmission bars. The details of the apparatus used are described by Nemat-Nasser et al., Sharma et al., and Subhash et al.

In the modified Hopkinson bar technique, the sample is subjected to a single stress pulse of a predefined profile and then recovered without the sample having been subjected to any additional loads. Hence, it allows correlation of microstructural damage to the exact number of fatigue cycles.

In a typical dynamic fatigue test employed here, the peak stress to which the samples were subjected exceeded the compressive yield strength of maraging steel (2.4 GPa). To ensure that the incident and transmission bars remain elastic, pieces of tungsten carbide (compressive strength greater than 5 GPa) of appropriate diameter and length were placed between the bars and the sample. The diameter of the tungsten carbide pieces was chosen to match the effective impedance between the bars and the tungsten carbide inserts. Matching effective impedance between bars and inserts ensures that no reflection of the stress pulse occurs when it reaches the interface between the bar and the insert.

In a Hopkinson bar experiment, the stress in the sample is determined from the time-dependent transmitted pulse, whereas the time-dependent reflected pulse gives a measure of the strain rate (and hence strain) in the sample, as long as the effective impedance of the sample is considerably less than that of the incident and transmission bars. The effective impedance of hard ceramics, such as silicon nitride, approaches the impedance of the bars, and hence, the reflected pulse gives an inaccurate measurement of the sample strain. To measure the strain in the sample, strain gauges were mounted directly on the sample, and the output of these gauges gave the sample’s time-dependent strain.

For silicon nitride, which behaves elastically at room temperature, a constant strain rate implies a constant stress rate in the material. Thus, the transmitted pulse should increase linearly with time. On the other hand, for a constant strain rate in the specimen, the difference between the transmitted and incident strains should be constant. Hence for a constant strain rate, the time-dependent incident pulse should increase linearly with time. To achieve a ramped incident pulse, a copper pulse shaper of appropriate diameter and thickness is placed between the striker bar and the incident bar. When the striker bar im-

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**Table I. Composition and Measured Mechanical Properties of ABP and CBP Silicon Nitrides**

<table>
<thead>
<tr>
<th></th>
<th>CBP silicon nitride</th>
<th>ABP silicon nitride</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition of boundary phase</td>
<td>8 wt% yttria, 1 wt% alumina</td>
<td>6 wt% yttria, 3 wt% alumina</td>
</tr>
<tr>
<td>Young’s modulus (GPa)</td>
<td>319</td>
<td>304</td>
</tr>
<tr>
<td>Shear modulus (GPa)</td>
<td>126.2</td>
<td>120.7</td>
</tr>
<tr>
<td>Poisson’s ratio</td>
<td>0.264</td>
<td>0.259</td>
</tr>
<tr>
<td>Density (kg/m³)</td>
<td>3247.8</td>
<td>3232.6</td>
</tr>
</tbody>
</table>
pacts the incident bar, the pulse shaper deforms gradually, creating an incident ramp pulse. The exact shape of the stress pulse depends upon the length and speed of the striker bar, as well as on the geometry of the copper pulse shaper.

The fatigue lives of both ABP and CBP silicon nitrides were determined for a peak stress of 3.2 GPa and at a strain rate of 400/s. Each time the split Hopkinson compression bar experiment was conducted, the sample was subjected to a single dynamic compressive pulse (compression-zero cycle). These experiments were then repeated until the samples failed. In this sense, these experiments were different from the conventional fatigue tests where samples are continuously loaded and unloaded.

(4) Ultrasonic Measurement of Wave Speeds

To measure longitudinal and shear wave speeds in silicon nitride, ultrasonic tests were conducted on a Matec’s MB 8000 system, along with a Marconi Instruments signal generator (2022 A), and a Tektronix dual beam oscilloscope. Two appropriate transducers were used to measure the pulse echo sound speed in longitudinal and shear modes. One of the transducers was used to transmit the signal from the controller to the sample; the other transducer was used to receive the transmitted signal after it had passed through the sample. The longitudinal and shear speeds are simply measured by recording the transit time and computing the speed according to

\[ \text{velocity} = \frac{(\text{thickness of the sample})}{(\text{transit time})} \]

The material properties (for isotropic materials only) are related to the shear wave speed \((V_s)\) and the longitudinal wave speed \((V_L)\) through the following relations:

\[
\text{Poisson ratio } (\nu) = \frac{1 - 2(V_s/V_L)^2}{2 - 2(V_s/V_L)^2} \tag{1}
\]

\[
\text{Young’s modulus } (E) = V_L^2 \frac{(1 + \nu)(1 - 2\nu)}{1 - \nu} \tag{2}
\]

\[
\text{Shear modulus } (G) = V_s^2 \rho \tag{3}
\]

where \(\rho\) is the density of the material.

During each fatigue cycle, dislocations and microcracks may be generated in the material, thereby degrading its mechanical properties. By comparing longitudinal and shear wave speeds in a damaged material with corresponding speeds in a pristine material, the average density of the microcracks in the material can be estimated. The average crack density, which is a function of the number of fatigue cycles, can be used as a measure of damage in the material. Because of the difficulties in measuring small changes in shear wave speed, only longitudinal wave speeds were measured in our experiments.

III. Experimental Results and Discussion

(1) Microstructure of Silicon Nitrides

The microstructures of CBP and ABP silicon nitrides differ only in the nature and composition of the grain boundary phase. Figure 2(a) shows a two-beam lattice image of three grains of silicon nitride and the grain-boundary phase. The presence of lattice fringes in the grain-boundary phase indicates its crystalline nature. Figure 2(b) shows a similar two-beam lattice image of ABP silicon nitride. The absence of lattice fringes in the grain-boundary phase suggests its amorphous nature, which was also verified by microdiffraction. The crystallized grain-boundary phase of CBP silicon nitride was identified, using X-ray diffraction, to be a combination of \(Y_3Si_2O_7N_2\) and \(YSiO_2N\).

Figures 3(a) and 3(b) show the microstructures of CBP and ABP silicon nitride, respectively, at a somewhat lower magnification. The microstructures shown in Figs. 3(a) and (b) were oriented perpendicular to the axial direction. The microstructures in the perpendicular direction to those shown in Figs. 3(a) and (b) were similar. Both materials had nearly the same grain size and grain morphology. The dark regions in both figures represent the grain-boundary phase. The \(\alpha/\beta\) ratio for both types of silicon nitride was close to zero, suggesting that the majority of silicon nitride grains were of the type \(\beta\). The microstructure basically consisted of a bimodal distribution of \(\beta\)-silicon nitride grains. The average grain size for both materials was less than 0.5 \(\mu\)m. Some of the large rod-shaped grains were greater than 10 \(\mu\)m in their longest dimension. It is well established that the elongated \(\beta\)-silicon nitride grains are formed during the solution–diffusion–precipitation phase of the sintering process, and their growth is favored when the concentration of \(\beta\) particles in the starting silicon nitride powder is decreased and the amount of sintering aid is increased.\(^{15}\)

The lattice image of CBP silicon nitride (see Fig. 2(a)) does not reveal any amorphous-phase grain-boundary triple points. If the amorphous phase exists in this region, it is probably less than 1 nm thick. A thin (1–8 nm) amorphous phase, in CBP (5 wt% \(Y_2O_3\), 2 wt% \(Al_2O_3\)) hot-pressed silicon nitride, was found at all grain boundaries by Ahn and Thomas.\(^{19}\) The CBP material used in the present study has a higher weight percent of yttria and a lower weight percent of alumina compared to the material studied by Ahn and Thomas. Increasing the yttria content and decreasing the alumina content increases the viscosity of the grain-boundary phase,\(^{20}\) which has a greater tendency to crystallize compared to a low-viscosity phase. The CBP material (8 wt% \(Y_2O_3\), 1 wt% \(Al_2O_3\)) studied in this work crystallized upon cooling after the sintering operation. On the other hand, post-sintering heat treatment was necessary to crystallize \(Si_3N_4\), fluxed with 6 wt% \(Y_2O_3\), 2 wt% \(Al_2O_3\),\(^{21}\) whose grain-boundary phase has a higher viscosity than 5 wt% \(Y_2O_3\), 2 wt% \(Al_2O_3\) fluxed \(Si_3N_4\). Falk and Dunlop\(^{21}\) also found the presence of a thin vitreous phase between \(\beta\)-\(Si_3N_4\) grains and...
Thus, the absence of an amorphous phase in the CBP material is likely due to the composition of the grain-boundary phase that has a higher tendency to crystallize.

Since ABP and CBP silicon nitrides differ only in the relative amounts of yttria and alumina in the grain-boundary phase, their elastic constants are expected to be nearly the same, especially when the volume fraction of the grain-boundary phase is small. Table I lists the measured properties for ABP and CBP silicon nitrides. ABP silicon nitride had slightly lower moduli and Poisson’s ratio compared to CBP silicon nitride; these elastic constants were measured using ultrasonics.

(2) Dynamic Fatigue Tests

Figures 4(a) and (b) show typical stress-axial strain and stress-transverse strain curves for ABP and CBP silicon nitrides, respectively. Since the total strain does not exceed 1%, the engineering stress and strain are essentially the same as the true stress and strain. Both materials exhibit linear stress-strain curves with no appreciable global inelasticity. The values of the elastic constants measured from the stress-strain curves are in good accord with the elastic constants measured using ultrasonics (Table I). The area between the loading and unloading path for the stress-axial strain curve for both materials is nearly the same, suggesting that the same amount of energy is spent, per cycle, in generating damage such as dislocations and microcracks. For both materials, extensive dislocation activity was seen at the microstructural level. However, this dislocation activity was not sufficient to produce any measurable permanent strains in the specimens.

Silicon nitride samples from both materials were repeatedly tested at 3.2 GPa peak stress and at a strain rate of approxi-
approximately 400/s. Two dynamic fatigue tests were conducted to evaluate the fatigue life of each material. CBP silicon nitride samples failed in 33 and 44 cycles, whereas ABP samples failed in 91 and 104 cycles. Fatigue life data of ceramics are generally more scattered than the fatigue life data of metals. Hence, to completely characterize the fatigue life of a ceramic, a statistical analysis is necessary that requires data from numerous fatigue tests. The purpose of this research was to study damage evolution in silicon nitride and to correlate the damage evolution with the microstructure, and therefore, statistical analysis to determine the fatigue life of silicon nitride was not pursued.

Figure 5 shows degradation of the longitudinal speed under dynamic fatigue tests along the axial and two orthogonal trans-

![Fig. 3. TEM micrographs showing the microstructures of (a) ABP silicon nitride and (b) CBP silicon nitride.](image)

![Fig. 4. Typical stress–axial strain and stress–transverse strain curves obtained during fatigue of (a) ABP silicon nitride at a strain rate of 400/s, and (b) CBP silicon nitride at a strain rate of 400/s. The loading and unloading paths are indicated by arrowheads.](image)

![Fig. 5. Degradation of normalized longitudinal speed versus number of fatigue cycles for ABP and CBP silicon nitrides at 400/s.](image)
verse directions for ABP and CBP silicon nitrides. The fatigue tests were stopped at an intermediate number of cycles to measure longitudinal wave speed as a function of the number of loading cycles. The longitudinal wave speed did not degrade significantly along the axial direction for either material, suggesting that cracks in both materials were axial in nature. The degradation of the longitudinal wave speed along the axial direction in ABP silicon nitride was somewhat greater than that for CBP silicon nitride, likely due to a greater density of microcracks and/or their greater deviation from the axial direction.

Under dynamic tests, the longitudinal wave speed degrades more in one transverse direction than the other for CBP silicon nitride, indicating that greater damage is taking place along a plane whose normal is in the direction along which the wave speed degrades more; the samples failed by axial splitting along this plane. For ABP silicon nitride, the degradation in the longitudinal wave speed was nearly the same along both transverse directions, suggesting that nearly the same amount of damage had occurred along two orthogonal transverse directions. Thus, ABP silicon nitride behaves like a transversely isotropic material, with the plane of isotropy normal to the loading direction. This behavior favored multiple cracking, and the samples fragmented into numerous pieces at failure. The critical density of cracks (density of cracks at the time of failure) was much greater in ABP silicon nitride than in CBP silicon nitride.

(3) Microstructural Observations of Dynamically Tested Hot-Pressed Silicon Nitride Samples

To understand the damage mechanisms which lead to failure in both materials, samples were repeatedly tested as described, and the test was stopped at intermediate numbers of cycles. A few tests were conducted at a higher peak stress of 3.45 GPa and at a slightly higher strain rate than 400/s. Scanning and transmission electron microscopy was then conducted on these samples to study damage evolution and to explain the observed differences in the fatigue lives of the ABP and CBP silicon nitrides.

(4) Crystalline-Boundary-Phase Silicon Nitride

TEM observations of repeatedly loaded and unfractured specimens showed incremental increases in the extent of dislocation activity as a function of the number of stress cycles (see Fig. 6). Dislocation activity was greatest in the largest grains of silicon nitride. The dislocations in large grains of silicon nitride usually originated from the contact region where a smaller grain impinges on a large grain (Fig. 6). During the applied load, the stresses in the contact region become very large, resulting in the plastic deformation of the larger grain, which may lead to its transgranular failure. In this process, very few dislocations were generated in the small grain by comparison to the large grain. Thus, the place where a small grain impinges on a large grain is a potential site for fatigue crack nucleation within the material. Extensive dislocation activity was seen in large silicon nitride grains close to failure.

Close to the cycles-to-failure, the crystalline grain-boundary phase was often observed to be absent or lost in the TEM samples (Fig. 7); this sample was subjected to 40 cycles at 3.2 GPa. The voids evident in this figure were not produced during the operation of ion milling, as other specimens subjected to fewer cycles and prepared in the same manner did not show such voids. These voids most likely resulted from intense dislocation activity within the boundary region, eventually leading to boundary-phase fragmentation.

The nature and composition of the grain-boundary phase are critical factors in determining the properties of silicon nitride. For example, a crystalline grain-boundary phase usually results in an increase in high-temperature properties and creep resistance. However, the crystalline grain-boundary phase may be susceptible to dislocation activity and subsequent cracking. As a result, the crystalline boundary phase may be less effective in blocking dislocations or cracks, and consequently the dominant crack may grow axially. Dislocations in the crystalline boundary phase are shown in Fig. 8 (40 cycles at 3.2 GPa).

The fatigue failure mode in CBP silicon nitride is shown in Fig. 9. The larger grains cleaved, and cracks followed an intergranular path around the small grains. In this process, the grain-boundary phase also failed by cracking. Relatively few cracks were seen under both SEM and TEM because of the low density of cracks in these samples. Figure 10 shows a lower-magnification view of the fatigue fracture surface in the CBP material. The presence of striations on the fracture surface is indicative of fatigue, each striation representing the successive position of an advancing crack front with each cycle. From the pattern of fatigue striations, it appears that the fatigue failure originated from surface flaws.

Fig. 6. TEM micrograph showing dislocations originating from the contact region (shown by arrows) between two grains in CBP silicon nitride (1 cycle at 3.2 GPa).

Fig. 7. TEM micrograph revealing crystalline grain-boundary phase failure in silicon nitride, resulting in the formation of voids between the grains of silicon nitride (40 cycles at 3.2 GPa). Arrows indicate the location of voids.
In CBP silicon nitride, the dominant surface flaws grow, at the expense of less favorably oriented cracks. At the same time, fatigue cracks are being nucleated at certain contact regions between small and large grains. When the dominant flaw traverses the length of the sample, the sample fails by axial splitting. The spacing between two consecutive striations is the amount by which the crack had advanced in one cycle. The number of striations matched well with the number of loading cycles, confirming the validity of the modified Hopkinson bar technique in the study of dynamic compression fatigue of hard ceramics.

(5) Amorphous-Boundary-Phase Silicon Nitride

The average dislocation density in ABP silicon nitride was found to increase with the number of stress pulses. Similar to CBP silicon nitride, the maximum dislocation activity was found in large grains which eventually leads to their transgranular fracture. Force is transmitted from one grain to another through the contact region (Fig. 11(a)); this sample was subjected to 40 cycles at a peak stress of 3.45 GPa. The intense dislocation activity in the large grain, originating from the contact region, has resulted in the shearing of the large grain, thereby nucleating a fatigue microcrack. The small grain had very few dislocations, even though the contact stresses were essentially the same in both grains. Finally, close to the cycles-to-failure, heavily dislocated grains of silicon nitride were found (Fig. 11(b)); this sample was subjected to 100 cycles at 3.2GPa.

In contrast to CBP silicon nitride, the amorphous nature of the grain-boundary phase precludes dislocations from passing through it. As a result, dislocations are blocked at the grain boundaries (Fig. 12); this sample was subjected to 40 cycles at 3.45 GPa. Depending upon the strength of the amorphous phase, the cracks may be deflected or may even get blocked at the amorphous intergranular phase, thereby increasing the toughness of the material. Deflection of cracks by the amorphous boundary phase forces them to follow a tortuous path which leads to a higher crack density and more deviation of the cracks from the axial direction. This is in accordance with the ultrasonic results of the ABP material (see Fig. 5) for which the longitudinal wave speed degrades more along both axial and transverse directions than in the CBP silicon nitride.

Another mode of deformation found in ABP silicon nitride was twinning. The formation of twins is favored at high strain rates and low temperatures. Figure 13 shows twinning in a grain of silicon nitride prior to cracking. By comparison to the number of heavily dislocated grains, very few twinned grains of silicon nitride were found. The most extensive mode of damage was microcracking.

The mechanism of crack propagation in ABP silicon nitride was somewhat different from that in CBP silicon nitride. Cracks in the ABP material preferred to grow along the interface between silicon nitride grains and the amorphous grain-boundary phase (Fig. 14). When a crack reached one of the corners of a grain, it had two alternatives: (a) it could change its direction and still continue along the interface, or (b) it could go through the amorphous grain-boundary phase. The actual path taken by the crack depends on the local microstructure around the crack tip, local stress and strain fields, the angle through which the crack had to change its direction, the size of the grain-boundary phase, and possibly other factors. In other words, the path that maximizes the energy release rate for any incremental crack extension is chosen. At the place where a relatively large grain contacted a small grain, transgranular fracture of the large grain occurred. In this case, the crack, after cleaving the large grain, usually propagated along the interface between the amorphous grain-boundary phase and the small grain. Fracture micromorphology consisted of both intergranular and transgranular fracture modes in the ABP material, as shown in Fig. 15(a). There is a greater propensity for intergranular fracture, in contrast to CBP silicon nitride.
Fatigue in ABP silicon nitride also starts from the flaws present on the surface. The most favorable surface flaws grow during each cycle, causing fatigue striations. Also, during each fatigue cycle, microcracks nucleate from the contact regions between small and large grains. Because of the amorphous nature of the grain-boundary phase, these flaws and microcracks deviate locally from the axial path. This results in a decrease in the stress intensity factor at the crack tip, allowing other (less favorable) cracks to grow. As a result of the higher density of cracks and their greater propensity to deviate from the axial direction, failure in the ABP material occurred by fragmentation. By comparison, the CBP material generally failed by axial splitting.

![TEM micrograph showing (a) dislocations originating in a grain from the contact region (shown by arrows) between two grains of silicon nitride (40 cycles at 3.45 GPa), and (b) a heavily dislocated grain of ABP silicon nitride (100 cycles at 3.2 GPa).](image)

Fig. 11. TEM micrograph showing (a) dislocations originating in a grain from the contact region (shown by arrows) between two grains of silicon nitride (40 cycles at 3.45 GPa), and (b) a heavily dislocated grain of ABP silicon nitride (100 cycles at 3.2 GPa).

![Dislocations in ABP silicon nitride blocked at the amorphous grain-boundary phase (40 cycles at 3.45 GPa). B indicates the grain-boundary phase.](image)

Fig. 12. Dislocations in ABP silicon nitride blocked at the amorphous grain-boundary phase (40 cycles at 3.45 GPa). B indicates the grain-boundary phase.

(6) Estimation of Degradation of Elastic Constants as a Function of Number of Fatigue Cycles

The data for the degradation of the longitudinal wave speed as a function of the number of fatigue cycles (Fig. 5) can be used to find the changes in the elastic constants as a function of the number of fatigue cycles. The normalized longitudinal speed shown in Fig. 5 is given through the following relation:

$$\frac{C_L}{C_L^0} = \sqrt{\frac{E(1-\nu)(1+\nu)(1-2\nu)}{E(1-\nu)(1+\nu)(1-2\nu)}}$$

where $E$ is the Young modulus, $\rho$ is the density, and $\nu$ is the Poisson ratio of the pristine material, and $E^0$ is the Young modulus, $\rho^0$ is the density, and $\nu^0$ is the Poisson ratio of the material tested along the direction of interest. Since the elastic strains are small, the density of the tested material can be assumed to be equal to the density of the untested material.

Micromechanics can be used to find the degradation of the elastic constants as a function of the number of fatigue cycles. In micromechanics, the elastic properties at each continuum point are found by applying suitable boundary conditions to a material volume which is statistically representative of the infinitesimal material neighborhood (representative volume element) of the continuum point. Consider a linearly elastic representative volume element containing penny-shaped microcracks parallel to the $x_3$ direction. The normals to the microcracks lie in the $x_1x_2$ plane. If the distribution of crack sizes is independent of their orientation and the interaction between microcracks can be neglected (a dilute distribution of cracks), then the elastic constants of a transversely isotropic body are related to the elastic constants of an isotropic body through the following relations:23

$$\frac{E}{E^0} = \left\{1 + f \frac{2(1-\nu^0)^2(8\nu^0-3\nu^0)}{3(2-\nu^0)}\right\}^{-1}$$

$$\frac{\nu}{\nu^0} = \left\{1 + f \frac{2(1-\nu^0)}{3(2-\nu^0)} \left\{1 + f \frac{2(1-\nu^0)(8\nu^0-3\nu^0)}{3(2-\nu^0)} \right\}^{-1}$$

$$\frac{\mu}{\mu^0} = \left\{1 + f \frac{4(1-\nu^0)(4-\nu^0)}{3(2-\nu^0)} \right\}^{-1}$$

where $E$, $\nu$, and $\mu$ are the overall Young modulus, Poisson ratio, and shear modulus, respectively, in the plane of isotropy (the $x_1x_2$ plane). $f$ is the crack density parameter, given by,

$$f = \sum_{p=1}^{n} N_{p} a_{p}^{3}$$

where $a_{p}$ is the radius of the $p$th penny-shaped crack.

The crack density parameter as a function of the number of compression fatigue cycles can be found by substituting Eqs. (5), (6), and (7) into Eq. (4). The above equations can be used for ABP silicon nitride, as the material behaves like a transversely isotropic material (Fig. 5). Figure 16(a) shows the variation of the crack density parameter as a function of the number of compression fatigue cycles for ABP silicon nitride at a strain rate of 400/s. The corresponding curve for CBP silicon nitride is not shown in Fig. 16(a), as it does not exhibit a plane of isotropy.

Once the functional relationship between the crack density parameter and the number of fatigue cycles is known, Eqs. (5) to (7) can be used to find the degradation of the elastic constants as a function of the number of fatigue cycles. Figure 16(b) shows the degradation of the Young and shear moduli, in the plane of isotropy, for the ABP silicon nitride at a strain rate of 400/s. From Fig. 16(b) the failure of ABP samples appears to occur when the Young modulus, in the plane of isotropy, degrades by approximately 2.25%.

A typical dynamic test, at a peak stress of 3.2 GPa and at a strain rate of 400/s, takes approximately 60 ns to complete. There is a long gap (of the order of minutes) between two successive cycles, during which the Hopkinson bar apparatus is reset manually. Higher crack growth rates in air compared to tests conducted under vacuum were observed under four-point bending fatigue tests of silicon nitride by Okazaki et al.\(^4\) It was concluded that enhanced crack growth rates in air are caused by stress corrosion cracking. In view of the results of Okazaki et al.,\(^4\) the effect of the long delay between two successive dynamic cycles on the fatigue life of silicon nitride was evaluated. Quasi-static tests were performed on CBP silicon nitride at a strain rate of 0.01/s with a gap of approximately 4 min between two cycles. No change in the material response occurred after 300 cycles. The longest fatigue life of the CBP silicon nitride tested dynamically was approximately 44 cycles. Therefore, 300 cycles represent approximately a sevenfold increase in the life, with the same time gap, for stress corrosion cracking to have occurred. It is concluded that the delay involved between two successive fatigue cycles did not affect the fatigue life of silicon nitride in the present study.

**Fig. 13.** TEM micrograph showing deformation twins in ABP silicon nitride, prior to cracking (100 cycles, 3.2 GPa).

**Fig. 14.** TEM micrograph revealing a crack path in ABP silicon nitride. Cracks prefer to propagate along the interface between silicon nitride grains and the amorphous grain-boundary phase. The sample was subjected to 100 cycles at 3.2 GPa and the crack path is shown by arrows.
Because of the higher strength of the glassy boundary phase compared to the crystalline boundary phase, the room-temperature dynamic compression fatigue life of ABP silicon nitride was observed to be somewhat higher than the corresponding fatigue life of CBP silicon nitride. The differences in the thermomechanical properties may also lead to different internal stresses within the materials. The internal stresses are caused by the cooling of the material to room temperature and by the volume changes upon crystallization. Even though the pristine materials (Figs. 3 and 4) have very few dislocations and no microcracks, small differences in the internal stress can affect the crack growth rate and the fatigue life of silicon nitride significantly. While it is possible to evaluate the effect of internal stresses on the fatigue life of CBP silicon nitride by performing the test after heat-treating the material, this was not pursued in the present investigation.

At elevated temperatures, CBP silicon nitride may be expected to have better fatigue properties compared to ABP silicon nitride due to its superior high-temperature strength. As such, caution should be used in extending these room-temperature dynamic results to high-temperature regimes.

References