

much lower peak intensity compared to the first peak [$f(Q_1) \gg f(Q_2)$]. The intensity of Q_2 is, however, higher than Q_1 . Therefore, a random oxygen distribution can be excluded in the presence of microtraces of iron. The large amount of iron with the neighboring tellurium partially substituted by oxygen shows the existence of an association between interstitial iron and the substituted oxygen.

The spectrum of $\text{SnTe}^{(57}\text{Fe)}$ oxidized for the same time is split as shown in Fig. 1a. The shift of the isomer shift of Q_2 and thus the variation of $|\psi(0)|^2$ has the same δ value as oxidized $\text{SnS}^{(57}\text{Fe)}$ ($\delta \approx 0.23 \text{ mm sec}^{-1} \approx 1.15 \times 10^{-7} \text{ eV}$). Annealing in an inert atmosphere also leads to an increase in the intensity of Q_2 , which is comparable to that occurring in $\text{SnS}^{(57}\text{Fe)}$. In all events, the appearance of more complex clusters is dependent upon a higher oxygen concentration in these materials.

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VICENZO FANO
IDA ORTALLI
MASPEC, Laboratory of CNR,
Physics Institute,
University of Parma,
Parma, Italy

Dynamic K_{IC} and dynamic flexural strength in HS-130 Si_3N_4

The mode I fracture-toughness value, K_{IC} , of hot-pressed Si_3N_4 has been shown to increase significantly with increasing temperature above 1000°C [1, 2]. However, at normal testing speeds, this increase in K_{IC} is not associated with an increase in fracture strength, σ_f ; rather σ_f decreases rapidly with increasing temperature above 1000°C [3, 4]. The increase in K_{IC} and the decrease in σ_f at high temperatures have been attributed to the presence of a glassy grain-boundary phase whose viscosity decreases with increasing temperature. At high temperatures fast fracture is preceded by considerable sub-critical crack growth due to grain-boundary sliding [4] which decreases σ_f . On the other hand, the low-viscosity glassy phase absorbs energy during fast fracture, causing an increase in K_{IC} . Under impact loading conditions (i.e. high loading rates), the dynamic K_{IC} and σ_f have not been determined. Since rapid loading conditions should minimize or eliminate slow crack growth, it was of interest to determine $K_{IC(\text{dynamic})}$ and to examine whether fracture strength is reduced at high temperatures. The $K_{IC(\text{dynamic})}$ was measured by employing a controlled surface-flaw technique [2] in conjunction with an instrumented drop-weight test apparatus [5].

A controlled, Knoop indentation flaw [2] was placed on the tensile side of each of six HS-130 (Norton Co, Worcester, Mass), hot-pressed Si_3N_4 bend bars, $6.35 \text{ mm} \times 3.17 \text{ mm} \times 50 \text{ mm}$ long. An indentation load of 2.6 kg was applied, producing a nearly semicircular, sharp microcrack having $\sim 0.07 \text{ mm}$ depth and 0.170 mm surface length. The bend bars were then annealed at 1300°C for 1 h in air to relieve the residual stress around the indent [2, 6]. Two bars were broken in four-point bending at room temperature at a cross-head speed of $0.005 \text{ cm min}^{-1}$, and $K_{IC(\text{static})}$ was determined from the fracture stress and flaw dimensions. The remaining bars were broken in three-point bending in an instrumented drop-weight test apparatus used for impact testing [5]. In this apparatus the bar deflects into a laser beam, and the change in beam intensity is recorded photographically as a function of time for measurement of the deflection at fracture. Standard elastic analysis of the bending of bars [7] was then used to compute the fracture strength from the measured values of deflection. A 1.27 cm diameter steel ball was used as the impactor, and a deflection rate of $1.3 \times 10^4 \text{ cm min}^{-1}$ was measured at fracture. Two bars were fractured at room temperature and two at 1300°C . The flaw dimensions were measured from the fracture surfaces and, using fracture-mechanics analysis, $K_{IC(\text{dynamic})}$ was calculated from values of σ_f

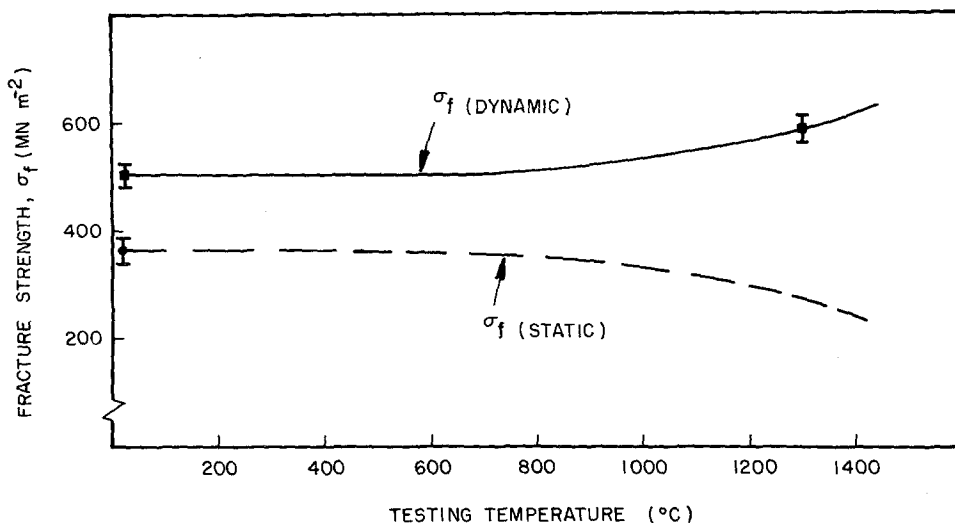


Figure 1 Temperature dependence of dynamic and static fracture strengths of indented HS-130 Si₃N₄ bend bars.

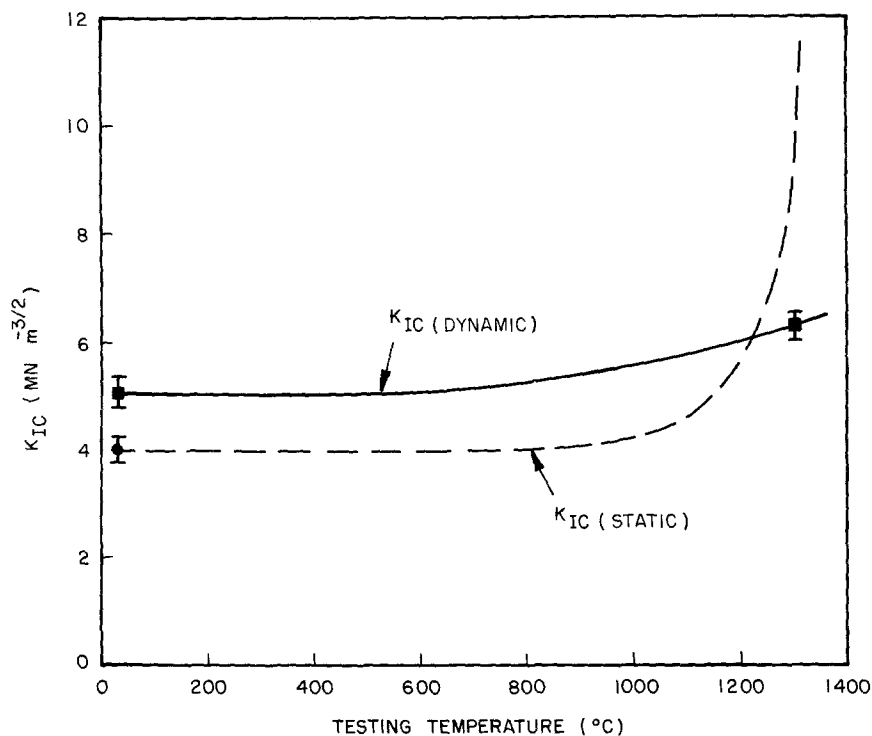


Figure 2 Variation of dynamic and static K_{IC} with temperature for HS-130 Si₃N₄.

and flaw dimensions [2].

The flaw dimensions measured on specimens subjected to dynamic loading at room temperature and at 1300°C were found to be very nearly equal and very close to those obtained on specimens

fractured at a low loading rate at room temperature. These observations indicate that no sub-critical crack growth occurred during dynamic loading at 1300°C. It is concluded that because of the high loading rate (1.3×10^4 cm min⁻¹) and

consequent short duration of the test, grain-boundary sliding and consequent microcracking could not occur.

The values of static and dynamic flexural strength for indented specimens and the value of static and dynamic K_{IC} as a function of testing temperature are shown in Figs. 1 and 2. A slight increase in the value of $\sigma_{f(\text{dynamic})}$ was observed at 1300°C as compared to the room-temperature value; however, this increase is not considered to be significant. The important result is that no decrease in $\sigma_{f(\text{dynamic})}$ occurred as a function of temperature. This behaviour should be compared to that exhibited by $\sigma_{f(\text{static})}$ (dashed line, Fig. 1)—showing a drop in $\sigma_{f(\text{static})}$ at 1300°C. The room-temperature difference between $\sigma_{f(\text{dynamic})}$ and $\sigma_{f(\text{static})}$ may be due to several factors including the difference in three-point and four-point loading, uncertainties in the measurement of the dynamic deflection, and scatter in the data. The $K_{IC(\text{dynamic})}$ is about 25% higher than $K_{IC(\text{static})}$ at room temperature and increases by about 25% at 1300°C, while $K_{IC(\text{static})}$ increases about 125% at 1300°C. These results indicate that under rapid loading conditions, $K_{IC(\text{dynamic})}$ does not increase markedly at high temperatures. These observations are consistent with an explanation that the viscous grain-boundary mechanism responsible for the increase in K_{IC} and for the occurrence of sub-critical crack growth at low strain-rates does not operate under rapid loading conditions.

The fact that $K_{IC(\text{dynamic})}$ can be obtained conveniently through the use of controlled flaws indicates that the $K_{IC(\text{dynamic})}$ value could be used as a measure of impact resistance. In the case of flexural failure, the absorbed energy is essentially the stored elastic energy at fracture, and the impact resistance is usually represented by the parameter

σ_f^2/E . Since, in the absence of controlled surface flaws, σ_f is always subject to the type and distribution of natural flaws, the use of the parameter $[K_{IC(\text{dynamic})}]^2/E = 2\gamma_f(\text{dynamic})$, where γ_f is the fracture surface energy, should be a better approach to the determination of the inherent impact resistance of a ceramic material.

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M. G. MENDIRATTA
Systems Research Laboratories, Inc
2800 Indian Ripple Road,
Dayton, Ohio USA

J. WIMMER
I. BRANSKY*
Air Force Materials Laboratory,
Wright-Patterson Air Force Base,
Ohio, USA

*Present address: State of Israel, Ministry of Defence, P.O. Box 7063, Tel Aviv, Israel.