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BENDING STRENGTH STUDIES ON HOT-PRESSED SILICON CARBIDE

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Translation of "Biegefestigkeitsuntersuchungen an heissgepresstem Siliciumcarbid," Berichte der Deutschen Keramischen Gesellschaft, Vol. 55, No. 8, 1978, pp. 391-394.

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16. Abstract

The 4-point bending strength of 4 grades of hot-pressed SiC was determined at different temperatures. With a transgranular mode of fracture the values for bending strength are retained up to high temperatures. For intergranular fracture the decrease of strength is governed by subcritical crack growth. The intergranular fracture is caused by a high content of silicate glassy phase at the grain boundaries of hot-pressed SiC.

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BENDING STRENGTH STUDIES ON HOT-PRESSED SILICON CARBIDE **

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SUMMARY The 4-point bending strength of four different grades of hot-pressed SiC was measured at different temperatures. Using a scanning electron microscope it was possible to demonstrate that with a transgranular mode of fracture the values for bending strength are retained up to high temperatures. In the case of intergranular fracture the decrease in strength is governed by subcritical crack growth. The reason for intergranular fracture is assumed to be a high content of silicate glassy phase at the grain boundaries of hot-pressed SiC.

1 INTRODUCTION

To date, mechanical engineering has used almost exclusively metallic materials. Because of their deficient thermal resistance, in most cases the use of metals and alloys under oxidizing conditions is limited to temperatures below 1000°C. For this reason, ceramic construction materials are usually /391*

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considered at high temperatures. Among the possible candidates, the ceramic materials most suitable for high temperature mechanical engineering are silicon nitride (Si_3N_4) and silicon carbide (SiC), because in addition to their high hardness they exhibit excellent corrosion resistance and unusually good temperature change resistance, in comparison to other ceramics.

In this paper we shall examine the bending strength of hot-pressed silicon carbide. The high temperature hardness characteristics will be discussed jointly with the fracture behavior and the structure arrangement. We shall show that for this material high room temperature hardness does not necessarily entail an elevated high-temperature hardness.

2 SAMPLE PREPARATION

Four SiC powders of 3 µm grain size or finer, which differed in the kind and quantity of sintering-promoting additive used, were pressure-sintered into discs in a Degussa hot press VSPI 25/15 at 2100°C and a pressure of 400bar. The discs' diameter was 72 mm and their height 6 mm. Using diamond tools, 4-point bending strength specimens were cut from these formed pieces, with the dimensions 3.15 mm x 4.1 mm x 35 mm, and smoothed longitudinally. The average density of the little rods was 3.19 mg/cm^3 (>99% of the theoretical density of SiC).

3 TEST DESIGN AND TEST METHOD

The bending specimens were tested on edge, such that the

hot-pressing direction coincided with the direction in which the pressure is applied.

The tests were performed using a 4-point bending apparatus of our own manufacture. The load is transmitted according to the lever principle and applied through an overflowing water container. The apparatus' core - the bending device - is located in the heating zone of a silicon carbide tube furnace. The individual parts of the bending device are made of hot-pressed (HP) silicon carbide.

The pressure transmission to the bending device and the specimen occurs in the furnace via two corundum tubes that are water-cooled at the upper and lower ends. The temperature is measured with a PtRh/Pt thermoelement whose sensor is directly under the specimen. Another thermoelement, next to the specimen, is used for controlling the furnace. The furnace itself can be displaced upwards or downwards by means of a chain drive, to insert the specimens. The furnace's heating rate is 100 K/min. The bending strain's stressing rate is $3MN/m^2s$.

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4 TEST RESULTS

The test results for the four kinds of HP-SiC examined are shown graphically in Figure 1, below.

Of the four kinds examined, HP-SiC No. 1 had the highest strength at room temperature, but it decreased most rapidly with temperature, such that at 1500°C it was less than 1/5 of the value attained at room temperature. While in specimen No. 2 the strength also decreased with increasing temperature, it fell much less rapidly than for No. 1: at 1500°C the material had

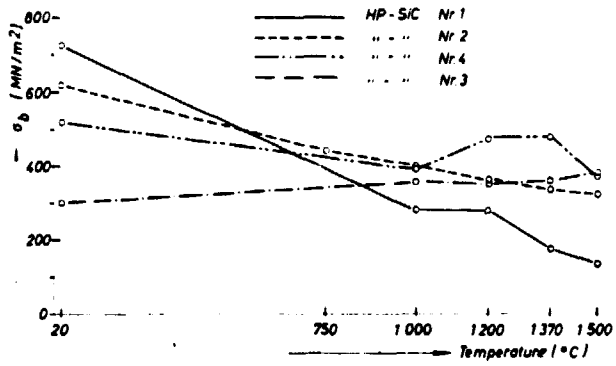


Figure 1. Hot bending strength of HP-SiC as a function of temperature

lost only approximately half its strength. Specimen No. 3 had the lowest strength of the four, at room temperature, but its strength proved to be substantially independent of temperature. In fact, it seems to improve with temperature, such that at 1500° C it had the highest strength of any of these four materials. While at room temperature the strength of No. 4 is below that of No. 1 and No. 2, it is substantially higher than that of No. 3. It decreases slightly near 1000°C, but at 1200°C rises again, nearly to the original value, to decrease again only at 1500°C.

5 CERAMOGRAPHIC EXAMINATION

The characterization of the four types of SiC was performed on

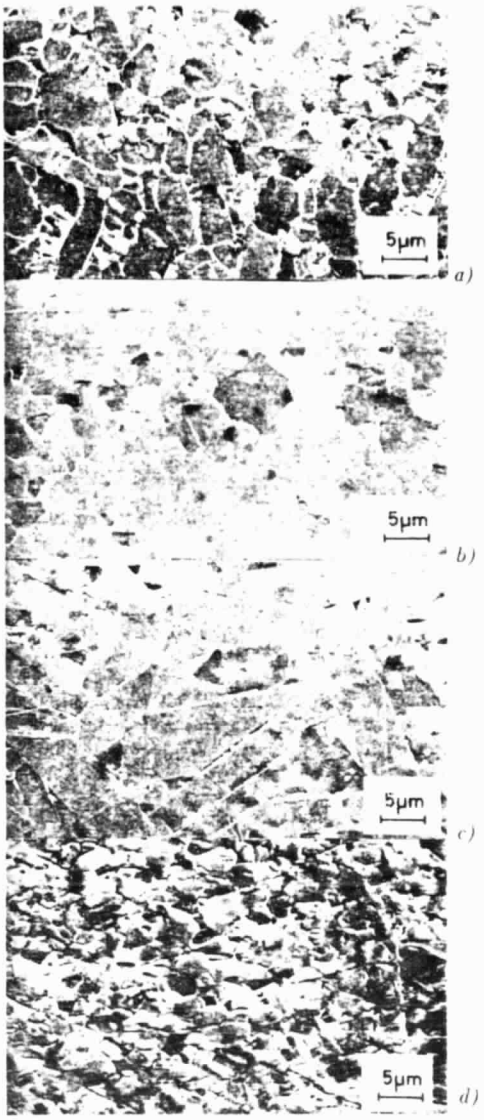
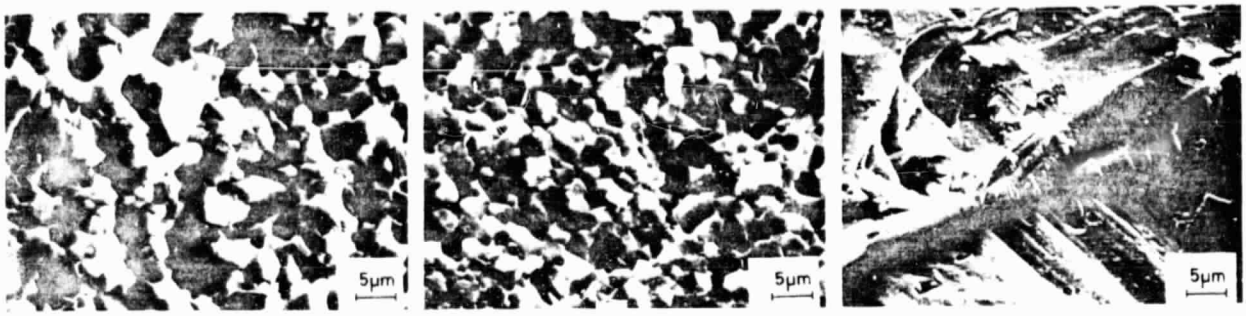


Figure 2. SEM image of etched, ground and polished surfaces. a) HP-SiC No. 1, b) HP-SiC No. 2, c) HP-SiC No. 3, d) HP-SiC No. 4

the etched and polished structural images of specimens, obtained with a type Super II International Scientific Instruments scanning electron microscope (SEM). The etching agent used was boiling Murakami solution (alkaline ferricyanide solution); the etching time was approximately 3 minutes. With this method it is



a) b) c)

Figure 3. SEM images of fracture planes, broken at room temperature. a) HP-SiC No. 1, b) HP-SiC No. 2, c) HP-SiC No. 3

possible to attain grain surface etching on the polished SiC specimen.

The polished surface images in Figure 2, page 5, clearly show that the grains in HP-SiC Nos. 1, 2 and 4 are equiaxial, while that of No. 3 represents long, radial crystals. In No. 3, grain growth occurred during sinterization under pressure.

Figures 3 (above) and 4 (page 7) show the SEM images of the fracture planes. For types No. 1, 2 and 3 there are no significant differences in the fracture images at different test temperatures. Hence the representation at room temperature (Figure 3) is adequate to characterize the fracture behavior. But HP-SiC No. 4 exhibits different fracture behaviors at the different temperatures and hence Figure 4 shows a fracture plane image for each temperature, for this type. The high temperature fracture planes were etched with HF, because the original fracture surfaces became covered with an oxide film after the high temperature strength test.

Types Nos. 1 and 2 exhibit intergranular fracture. The fracture

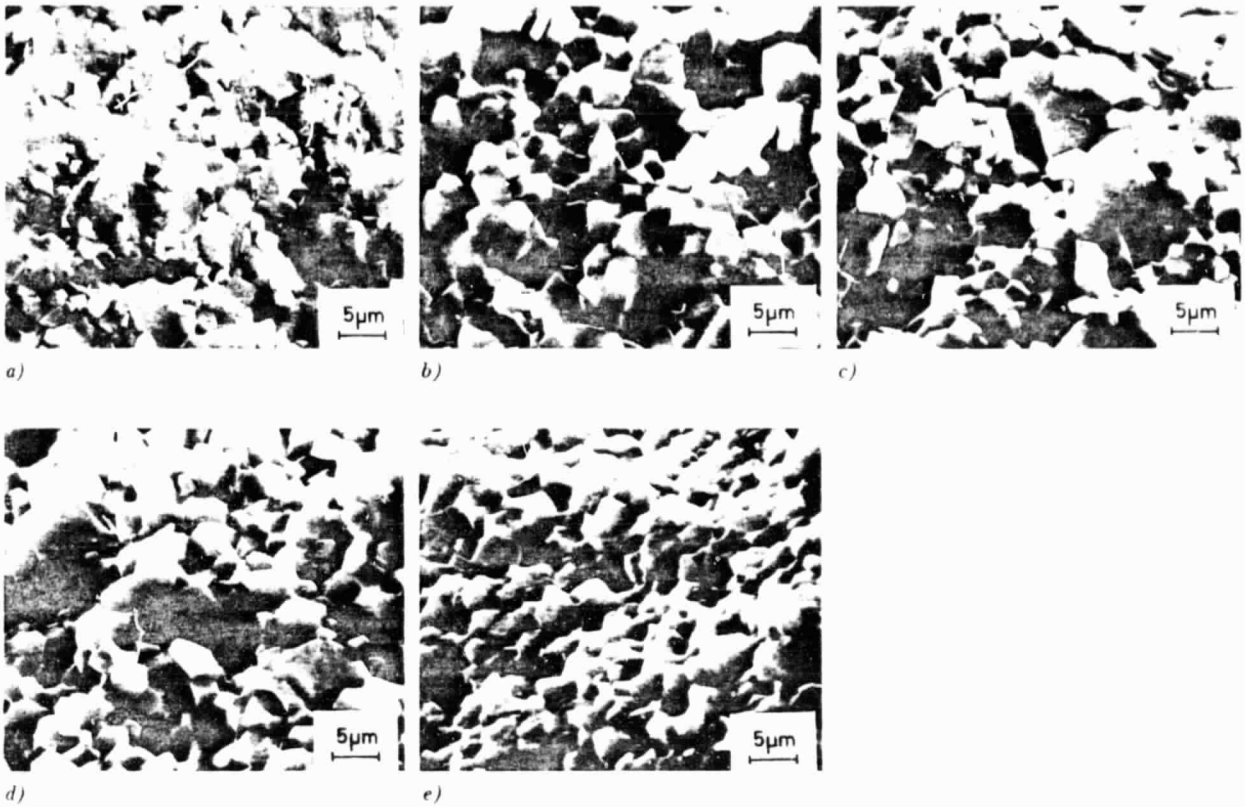


Figure 4. SEM images of HP-SiC No. 4 fracture planes, broken at a) Room temperature, b) 1000°C, c) 1200°C, d) 1370°C, e) 1500°C; b) - e) etched with concentrated HF

in No. 3 is transgranular throughout the temperature range tested. While No. 4 exibits transgranular fracture at room temperature, at 1000°C the fracture is intergranular; at 1200°C the transgranular portion increases again, even though it is not comparable to the fracture image at room temperature.

If the images of the fracture surfaces are compared to the course of the bending strengths, it becomes apparent that an intergranular fracture is associated with a decrease in strength at increasing temperature (HP-SiC Nos. 1 and 2). In the case of

transgranular fractures, the strength remains constant as the temperature is increased (HP-SiC No. 3), provided the transgranular mode of fracture is retained over the entire temperature range.

In HP-SiC No. 4, the transition to intergranular fracture becomes apparent, at 1000°C, by the decrease in strength. The transgranular portion increases in this case, at higher temperatures, so that an improvement in strength is noted also here. Beyond 1370°C both the strength and the fraction of transgranular fractures decrease again.

6 DISCUSSION

In many materials the crack length hardly changes, when a strain is imposed on the specimen at low temperatures; at high temperatures, in contrast, most ceramic materials will experience "subcritical crack propagation" [2]. In this case the material's strength becomes dependent on the stressing rate: the higher it is, the higher becomes the mean measured strength. Charles [3,4] has found an analytical expression for this phenomenon, which he confirmed for glasses. Lange and Iskoe [5], as well as Evans and Lange [6] performed strength measurements as a function of stressing rate with SiC. They showed that at room temperature the subcritical crack propagation is low, while it becomes quite evident at high temperatures. Hence subcritical crack propagation can be considered the cause for the decrease in strength, with increasing temperature, at higher temperatures. Two models were suggested for the slow crack propagation in ceramic materials [2,7]. In materials with high

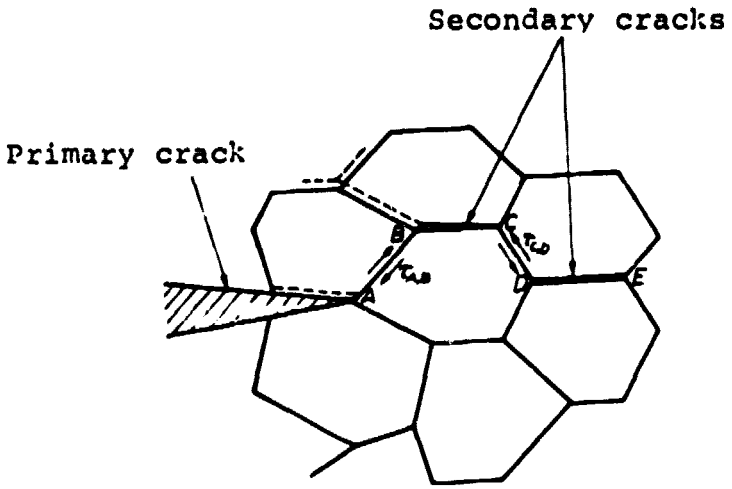


Figure 5· Model of subcritical crack propagation by the grain boundary slippage mechanism, from [7]

dislocation mobility a dislocation mechanism is assumed; in cases of low dislocation mobility - which are found among covalent solids- grain boundary slippage is assumed (Figure 5, above). The model for this was described in detail by Evans [7].

According to the grain boundary slippage model, subcritical crack propagation is only possible for intercrystalline fracture. If trans-crystalline fracture occurs, either another model is valid or slow crack growth barely takes place. For transcrystalline fracture we found either no strength decrease or a slow strength decrease with temperature in the materials investigated. Apparently the influence of subcritical crack propagation is small in these materials which show transcrystalline fracture.

We can ask which material properties have a decisive effect on determining which fracture mode occurs (trans- or intercrystalline). HP-SiC no. 1 and 2 were doped with aluminium, just like the HP-SiC material of Lange [5.6]. No. 1 had about twice as much aluminium as no. 2. Perhaps alumosilicate glass was formed at the grain boundary, which would be present at a higher concentration on the grain boundary for a larger aluminium content. This would explain the clear strength decrease of no. 1 with temperature. HP-SiC No. 4 had a much lower aluminium

content, so that there is hardly any glass phase. Some transcrystalline fracture is present in this material. HP-SiC no. 3 was doped with boron. We can assume that the grain boundary phase is B_4C. A protective layer is formed by oxidation, which leads to crack healing on the surface.

This explains the slight strength increase as a function of temperature. In a less compressed, boron-containing HP-SiC series, the author [1] found a particularly clear improvement of the strength as a function of temperature, which could also be attributed to this curing process.

7 CONCLUSIONS

Hot-pressing of SiC powders makes it possible to manufacture materials in which subcritical crack propagation barely takes place, during stressing at high temperatures. Such materials could be used as high temperature construction materials wherever long stress periods are required, since they are especially resistant to creeping processes.

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