

NASA Technical Memorandum 101400

High-Strength Silicon Carbides by Hot Isostatic Pressing

(NASA-TM-101400) HIGH-STRENGTH SILICON
CARBIDES BY HOT ISOSTATIC PRESSING (NASA)
19 p CSCL 11C

N89-13666

g3/27 Unclass
0183239

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Prepared for the
Third International Symposium on Ceramic Materials
and Components for Engines
sponsored by the American Ceramic Society
Las Vegas, Nevada, November 27-30, 1988

NASA

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higher temperatures (1900 to 2200 °C) for densification. Glass encapsulation softens at this high temperature and, because of its very low viscosity, tends to penetrate into the densifying specimens. Although work has been reported on glass and metal encapsulation for HIPing of silicon nitride preforms, very limited information exists about HIPing of silicon carbide with glass encapsulation.^{10,11} Furthermore, information on the HIPing of SiC encapsulated with a refractory metal such as tantalum is virtually nonexistent. This paper reports the properties obtained by the hot isostatic pressing (HIPing) of α - and β -SiC compacts encapsulated in tantalum cans, including characterization of strength, microstructure, and toughness.

EXPERIMENTAL PROCEDURES

Two types of α -SiC and two types of β -SiC powders were used in this study. Chemical analyses of these powders are shown in Table 1. The α -SiC(I) powder* had no premixed sintering aids such as boron and carbon added; however, it did contain 1.74 wt % excess carbon in the as-received condition. On the other hand, the α -SiC(II)* powder had premixed sintering aids (0.6 wt % boron and 7.31 wt % carbon). Both β -SiC(I)** and β -SiC(II)*** powders had no premixed sintering aids (boron and carbon) but contained small amounts of free carbon (Table 1). Both α - and β -SiC compositions were cold formed by a slurry pressing technique.^{12,13} The dried slurry-pressed compacts (4.67 cm in diameter by 0.6 cm thick) were wrapped with graphite foil, coated with boron nitride, and then placed in tantalum cans. The tantalum cans were outgassed for 6 to 8 hr at 1100 °C and then vacuum sealed. After a thorough leak check the cans were placed in a hot isostatic pressing furnace. An initial gas pressure of 14 MPa (2000 psi) was applied; then both temperature and pressure were increased simultaneously until the desired values were reached. The cans were HIPed at temperatures between 1850 and 2100 °C for 30 to 120 min under 138-MPa argon gas pressure. HIPed specimens were machined into test bars (2.54 by 0.64 by 0.32 cm), and the surfaces were ground with a 400-grit fine diamond wheel to a final surface finish of 8 rms. Density was measured by the water immersion method. Microstructural characterization was made by optical and electron microscopy. Flexural strength tests were conducted by four-point bending, with 0.95-cm inner and 1.87-cm outer spans. Testing was conducted at room temperature with a crosshead speed of 0.05 cm/min. Weibull modulus values were generated from the strength data, and the results were analyzed by using F- and t-tests. Fracture surfaces of selected test bars were examined by scanning electron microscopy to identify the strength-controlling critical flaws. Fracture toughness was determined by the Vicker's hardness indentation technique with a 2500-g load.

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RESULTS AND DISCUSSION

Hot-Isostatic-Pressed α -SiC

The densification behaviors of HIPed α -SiC(I) and α -SiC(II) are shown in Fig. 1. The α -SiC(I) exhibited sluggish densification behavior, and this resulted in lower final densities at all temperatures from 1800 to 2100 °C.

The maximum density for this material (97 percent of theoretical) was achieved at 2100 °C (Fig. 1). By contrast, only 1900 °C was required to sinter α -SiC(II) powder (containing boron and carbon sintering aids) to a final density of 98 percent of theoretical. No further increase in final density was observed with further increases in temperature from 1900 to 2100 °C. The results suggest that, although it is difficult, α -SiC(I) powder can be HIPed to high final density without any sintering aids. The results are in good agreement with Homma et al.¹⁰ and Larker et al.¹¹, who obtained high final density (96 percent of theoretical) in HIPed α -SiC without any sintering aids.

Microstructural characterization was made on polished and etched samples. Figure 2 shows photomicrographs of polished and etched α -SiC(I) HIPed at 1900 and 2000 °C for 30, 60, and 120 min. The microstructure essentially consisted of single-phase α -SiC matrix with residual porosities (determined from density measurements) varying from 14 percent for the 1900 °C, 30-min-HIPed specimens, to 5 percent for the 2000 °C, 120-min-HIPed specimens. The α -SiC grains were difficult to etch, and therefore the overall microstructure could not be resolved by optical microscopy. By contrast, clear microstructures with distinct grain boundaries were achieved in α -SiC(II) specimens (Fig. 3). Very little grain growth was observed in α -SiC materials HIPed between 1900 °C for 30 min and 2000 °C for 120 min (Figs. 2 and 3). The high-density α -SiC produced by HIPing exhibited an ultra-fine-grained microstructure with grain size varying from 0.2 to 5.0 μ m. Grain morphology was equiaxed in both α -SiC materials.

The room-temperature flexural strengths σ_f of α -SiC(I) HIPed at 1900 and 2000 °C for various times are shown in Fig. 4. Average strengths ± 1 standard deviation (12 test bars) were 368 ± 51 MPa, 444 ± 51 MPa, and 488 ± 40 MPa for specimens HIPed at 1900 °C for 30, 60, and 120 min, respectively. Final densities were 86, 88, and 91.5 percent of theoretical, respectively. Statistical analysis by the F-test (comparison of variances) indicated no significant differences among the variances. Student's t-test indicated that at the 95 percent confidence level there was no statistically significant difference in the room-temperature strength. At the higher HIPing temperature (2000 °C), average strengths were 551 ± 98 MPa, 574 ± 73 MPa, and 619 ± 63 MPa for specimens having densities of 91.6, 92.7, and 95 percent of theoretical and HIPed for 30, 60, and 120 min, respectively. The F-test indicated no significant difference among the variances. Student's t-test at the 95 percent confidence level indicated no significant strength difference between specimens sintered for 30 min versus 60 min and for 60 min versus 120 min, but a significant difference in strength between specimens sintered for 30 min versus 120 min. Student's t-test also indicated that the room-temperature strengths of materials sintered at 2000 °C were statistically higher than 1900 °C strength at the 95 percent confidence level. The highest strength,

619 MPa, obtained in HIPed α -SiC(I) was higher than the strength (540 MPa) reported by Larker, et al.¹¹ for HIPed α -SiC material. The results suggest that α -SiC(I) containing no sintering aids (boron and carbon) can exhibit very high flexural strength when HIPed to high final density.

A density effect on strength or in other words a porosity effect on strength was observed in HIPed α -SiC(I) specimens. The strength of porous ceramics decreased nearly exponentially with increases in porosity. Various specific analytical relationships have been suggested for the effect of porosity on strength. Ryshkewitch¹⁴ has shown that porosity and strength are generally empirically related by

$$\sigma = \sigma_0 \exp(-nP) \quad (1)$$

where

σ strength of porous specimen

σ_0 strength of fully dense specimen

n ranges from 4 to 7

P volume fraction pores

From this relation the volume fraction pores as a function of strength in HIPed α -SiC(I) was determined as shown in Fig. 5. Regression analysis yielded the following equation:

$$\sigma = 845 \exp(-5.64P) \quad (2)$$

Note that the constant in the exponent ($n = 5.64$) falls at the approximate midpoint between 4 and 7 and is in good agreement with Ryshkewitch's relation and that at zero porosity the strength would be 845 MPa.

The flexural strength data of α -SiC(II) (containing boron and carbon) are shown in Fig. 6. The average strengths (12 test bars) were 596 ± 59 , 614 ± 61 , and 625 ± 65 MPa at 1900 °C and 650 ± 58 , 620 ± 79 , and 564 ± 78 MPa at 2000 °C for sintering times of 30, 60, and 120 min, respectively. Statistical analyses such as the F-test and Student's t-test indicated no significant differences in strengths and variances. These strengths were statistically higher than the strengths found in α -SiC(I) specimens HIPed at 1900 and 2000 °C. However, at a final density level of 95 percent of theoretical for the α -SiC(I), the strength (619 MPa) of α -SiC(I) was statistically comparable with the strengths of α -SiC(II). The results suggest that α -SiC(I) (containing no sintering aids) can exhibit high flexural strength comparable to α -SiC(II) material when HIPed to the same high final density.

The data clearly indicated no systematic correlation between strength and Weibull modulus. Although a significant improvement in strength was achieved in hot isostatically pressed α -SiC over the sintered α -SiC strength of 428 MPa,¹⁵ no improvement in Weibull modulus was obtained. For materials HIPed at 1900 to 2000 °C, the Weibull modulus was estimated to be 8 to 14 for α -SiC(I) and 8 to 13 for α -SiC(II). The Weibull modulus for pressureless sintered α -SiC ranges from 8 to 12.^{15,16} Process-related

critical flaws, such as large isolated pores and shrinkage cracks, typical of sintered materials⁵ were not observed in HIPed materials. Typical fracture initiation sites were found to be surface related in HIPed α -SiC. These are shown in Fig. 7.

Hot-Isostatic-Pressed β -SiC

As-received β -SiC(I) and β -SiC(II) powders were used. The chemical analysis of these powders are shown in Table 1. Neither β -SiC(I) nor β -SiC(II) powders had premixed sintering aids. The β -SiC(I) powder was slurry pressed with 3 and 4 wt % carbon and 0.6 wt % boron; the β -SiC(II) powder was slurry pressed only with 3 wt % carbon and 0.6 wt % boron. On the basis of a preliminary HIPing study, a temperature of 1900 °C for 60 min was chosen for HIPing β -SiC compositions to near-theoretical density (97 percent). Typical microstructures of HIPed α -SiC(II) and β -SiC(I) are shown in Fig. 8. The submicrometer β -SiC grain size ($<1.00 \mu\text{m}$) was much finer than the typical microstructure of HIPed α -SiC (Fig. 3). Furthermore, the β -to- α phase transformation typical of high-temperature sintered β -SiC microstructure (Fig. 9) was not observed in the "low"-temperature-HIPed β -SiC (Fig. 8). The grain morphology was completely equiaxed and uniform.

The room-temperature flexural strengths of β -SiC(I) and β -SiC(II) HIPed at 1900 °C for 60 min are shown in Fig. 10. Average strengths (22 test bars) were 659 ± 79 and 673 ± 66 MPa for β -SiC(I) specimens with 3 and 4 wt % carbon, respectively. The strength of β -SiC(II) specimens with 3 wt % carbon was 645 ± 100 MPa. Statistical analyses by the F-test (comparison of variances) and Student's t-test indicated no significant differences in variance or in room-temperature strength between β -SiC(I) and β -SiC(II) specimens. The results indicated that both commercially available β -SiC powders, when HIPed to high final density, yield material similar in strength to HIPed α -SiC. The strength data for both β -SiC(I) and β -SiC(II) were compared with those for HIPed β -SiC(II) specimens (Fig. 11). In general, no statistically significant difference in strength was found. The Weibull modulus was estimated to be 9.5 to 11 for β -SiC(I) and 7 for β -SiC(II) compositions, about the same as for α -SiC compositions. The strength-controlling flaws in HIPed β -SiC were generally surface related and similar to those observed in HIPed α -SiC. These are shown in Fig. 12. The highest-strength samples broke into numerous pieces, and specific failure origins could not be identified.

Fracture toughness K_{1C} was determined by direct crack measurements of Vicker's indentations in both α - and β -SiC HIPed to a final density of 97 percent of theoretical. Fracture toughness K_{1C} was estimated by using the relation derived by Evans,¹⁷ which is as follows:

$$\frac{\log K_{1C}}{H_a^{1/2}} \frac{H^{2/5}}{E} = 16.32X^5 - 24.97X^4 + 11.23X^3 - 2.02X^2 - 0.34 - 1.59$$

where

K_{1C} fracture toughness

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- H hardness
- a indentation radius
- E Young's modulus
- X $\log(c/a)$
- c crack length

Fracture toughness values determined by this relationship gave results closest to existing K_{IC} data¹⁶ determined by chevron notch and controlled-surface flaw techniques. The K_{IC} values ranged from 3.6 to 4.0 MPa m^{1/2} for HIPed α -SiC and from 3.7 to 4.1 MPa m^{1/2} for HIPed β -SiC. Statistical analyses such as the F-test indicated no significant difference in variances, and Student's t-test indicated no significant difference in K_{IC} between HIPed α - and β -SiC specimens. However, K_{IC} values for HIPed α - and β -SiC were higher than those observed in pressureless sintered α -SiC (3 to 3.6 MPa m^{1/2}). For HIPed α - and β -SiC and pressureless sintered α -SiC, the F-test indicated no significant differences among the variances, but Student's t-test indicated statistically significant differences in K_{IC} values. Although statistically significant differences were observed, the improvement was not of sufficient magnitude to be of practical significance. Higher K_{IC} values were related to smaller grain size in HIPed α - and β -SiC materials.^{18,19}

CONCLUDING REMARKS

In this study both α - and β -SiC compositions, with and without sintering aids (boron and carbon), were HIPed to near-theoretical density (97 to 98 percent of theoretical). The HIPed materials had ultrafine grain size (0.3 to 5 μ m for α -SiC and <1.00 μ m for β -SiC compositions). Both materials exhibited high average flexural strength. Furthermore, α -SiC bodies without any sintering aids, when HIPed to high final density, exhibited very high strength. Also, higher fracture toughness was obtained in HIPed α - and β -SiC than in pressureless sintered α -SiC from an earlier study. The strength-controlling flaws, such as large voids and shrinkage cracks, typical of sintered SiC materials, were not observed in HIPed SiC. However, no improvement in Weibull modulus was observed over sintered SiC materials. Thus it is clear that although significant improvement has been made in strength via improved microstructure and minimization of processing flaws by HIPing, improvements in reliability and practically significant improvements in fracture toughness were not achieved. Thus other approaches such as compositing are needed to advance significantly beyond the current state of the art.

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Table 1. - Analysis of As-received α -SiC and β -SiC Powders

Element	α -SiC(I)	α -SiC(II)	β -SiC(I) ^a	β -SiC(II) ^b
	Content, ppm			
Al	50	140	300	100
Cr	70	40	Trace	20
Fe	10	10	700	200
Ti	20	30	-----	-----
V	20	20	-----	-----
B	-----	^c 0.60	-----	-----
Free C	^c 1.74	^c 7.31	^c 0.65	^c <2.0
Surface area (BET), m ² /g	31.47	22	19.8	10

^aIbiden Co. Ltd., Ogaki, Japan.

^bSuperior Graphite Co. Ltd., Chicago, Illinois.

^cUnit is weight percent.

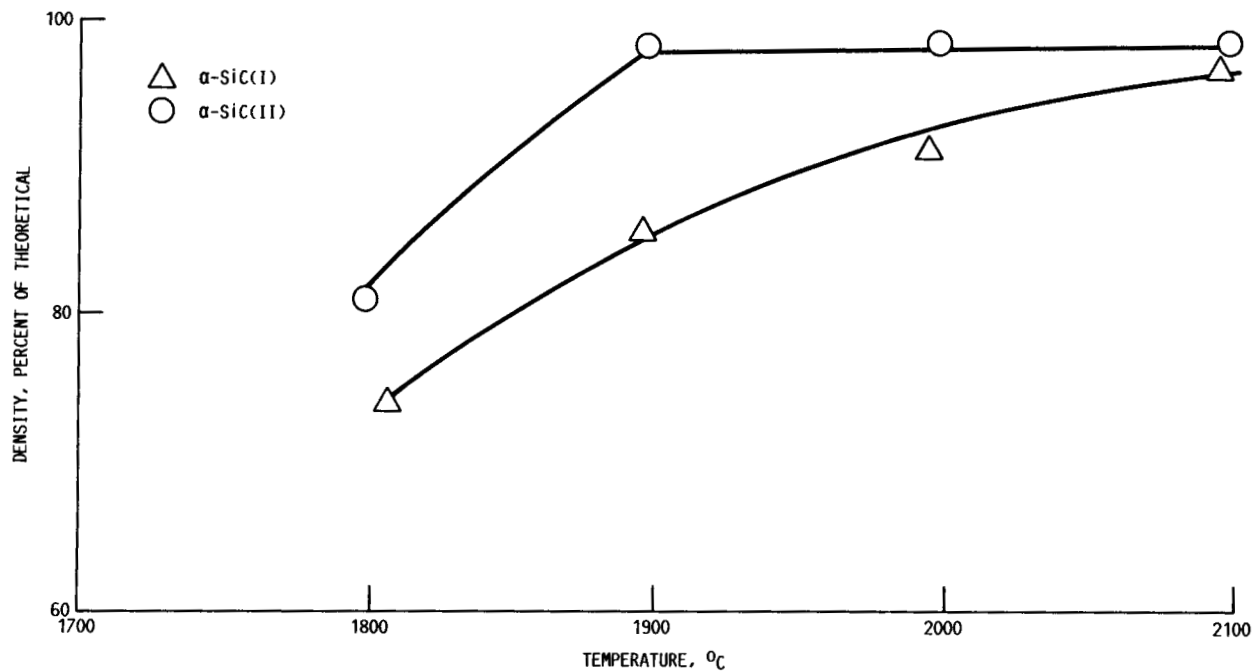
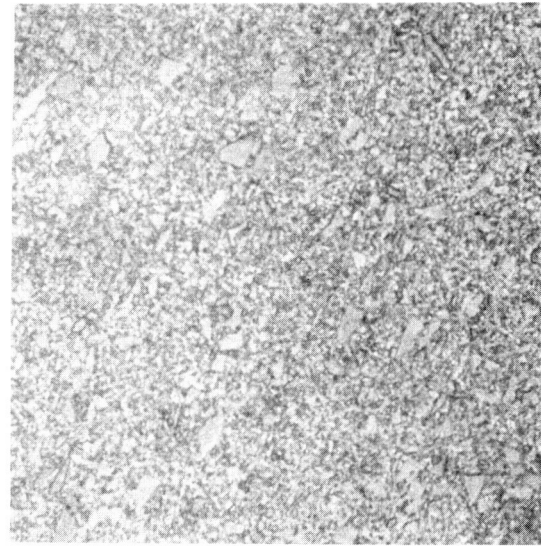
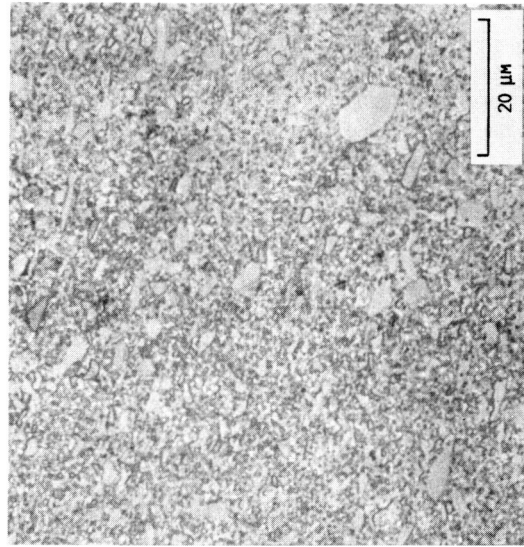


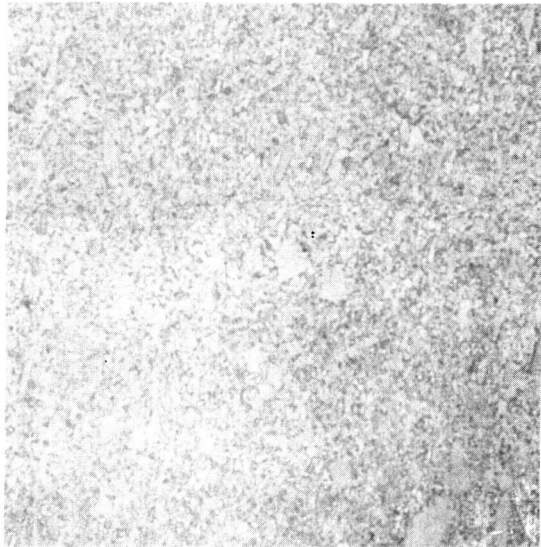
FIGURE 1. - RELATIVE DENSITY OF α -SiC POWDERS HIPED FOR 30 MIN AT DIFFERENT TEMPERATURES.



(c) 1900 °C; 120 MIN.



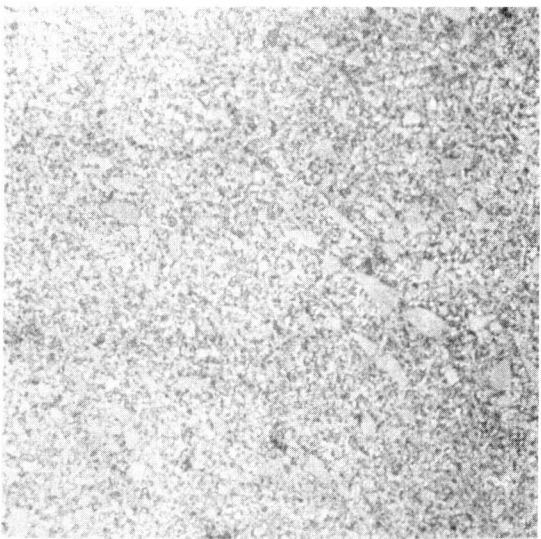
(f) 2000 °C; 120 MIN.



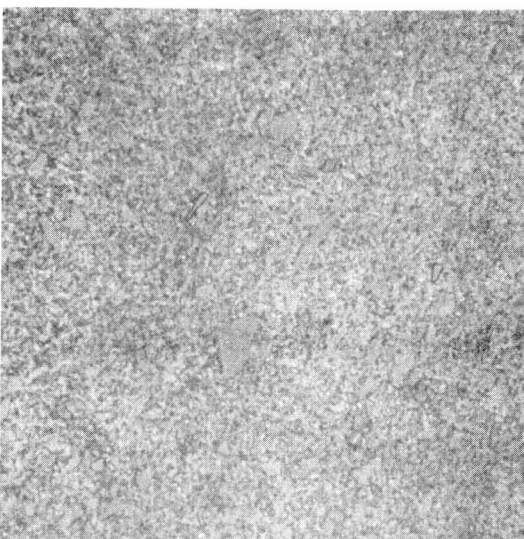
(b) 1900 °C; 60 MIN.



(e) 2000 °C; 60 MIN.

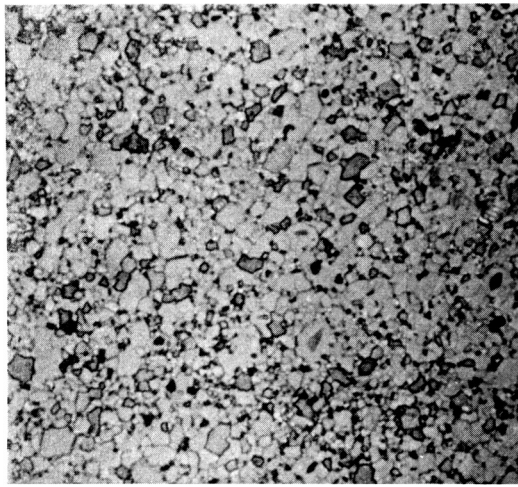


(a) 1900 °C; 30 MIN.

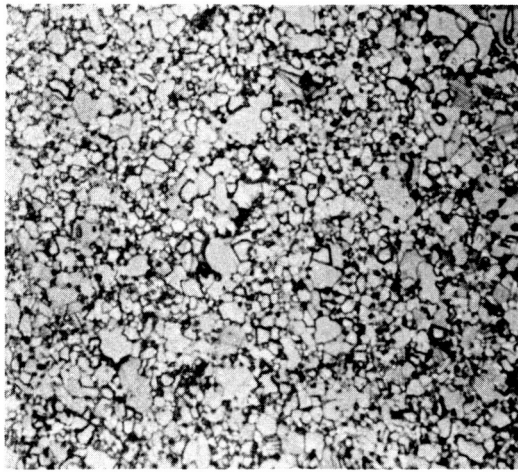


(d) 2000 °C; 30 MIN.

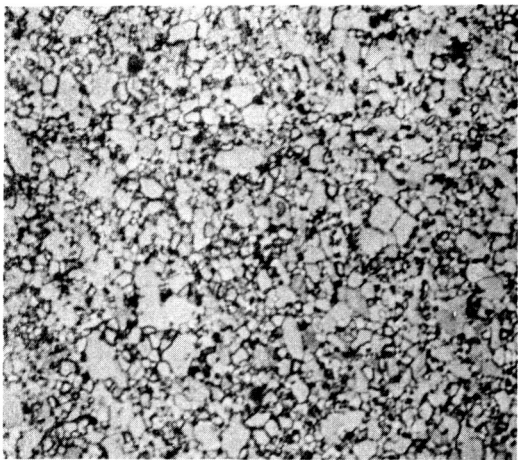
FIGURE 2. - MICROSTRUCTURES OF HOT-ISOSTATIC-PRESSED α -SiC(1).



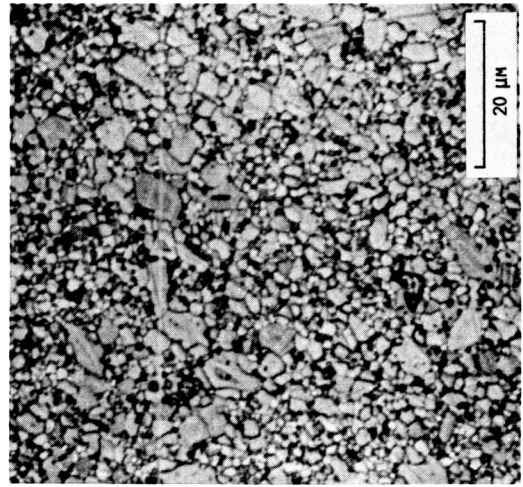
(c) 1900 °C; 120 MIN.



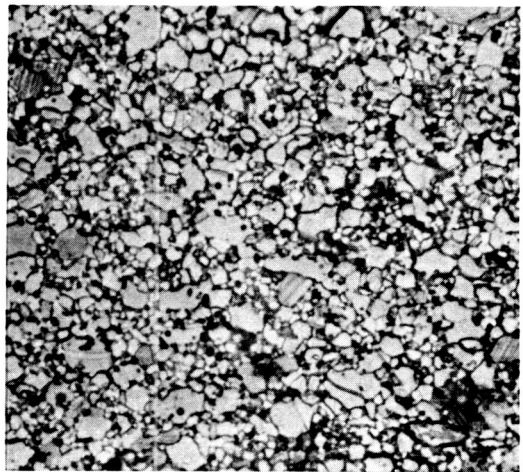
(b) 1900 °C; 60 MIN.



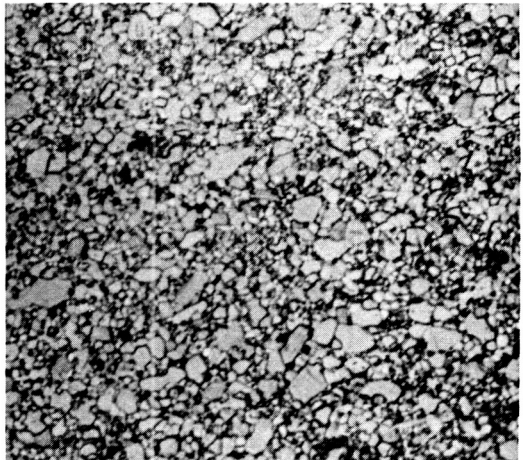
(a) 1900 °C; 30 MIN.



(f) 2000 °C; 120 MIN.



(e) 2000 °C; 60 MIN.



(d) 2000 °C; 30 MIN.

FIGURE 3. - MICROSTRUCTURES OF HOT-ISOSTATIC-PRESSED α -SiC(11).

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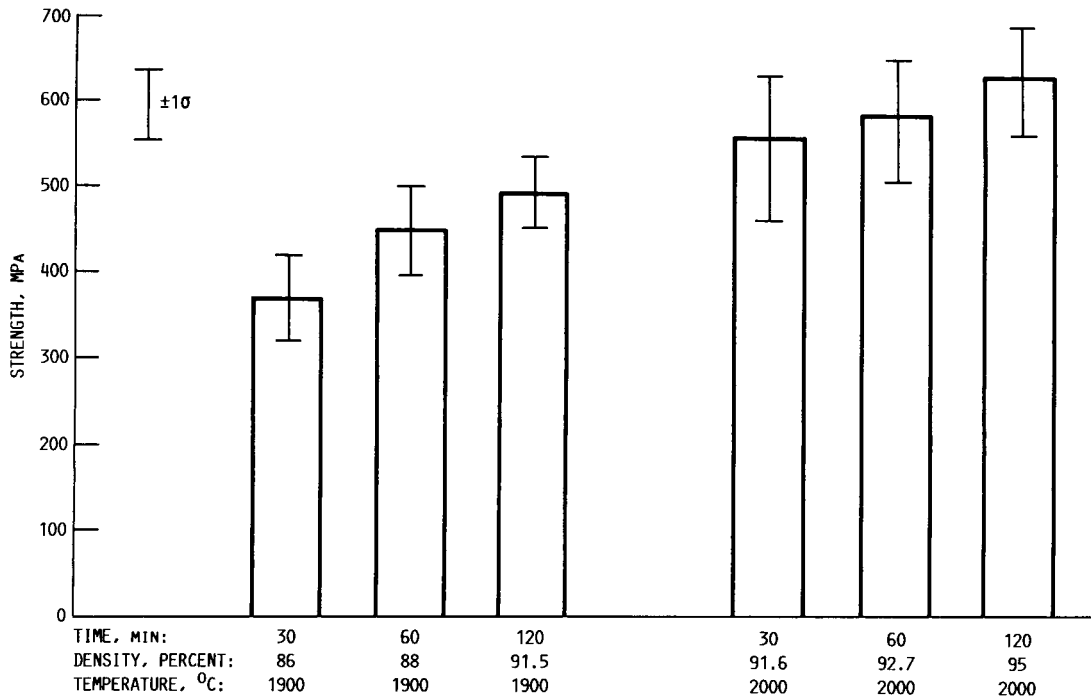


FIGURE 4. - ROOM-TEMPERATURE FLEXURAL STRENGTH OF α -SiC(I) HIPed AT DIFFERENT TEMPERATURES AND TIMES.

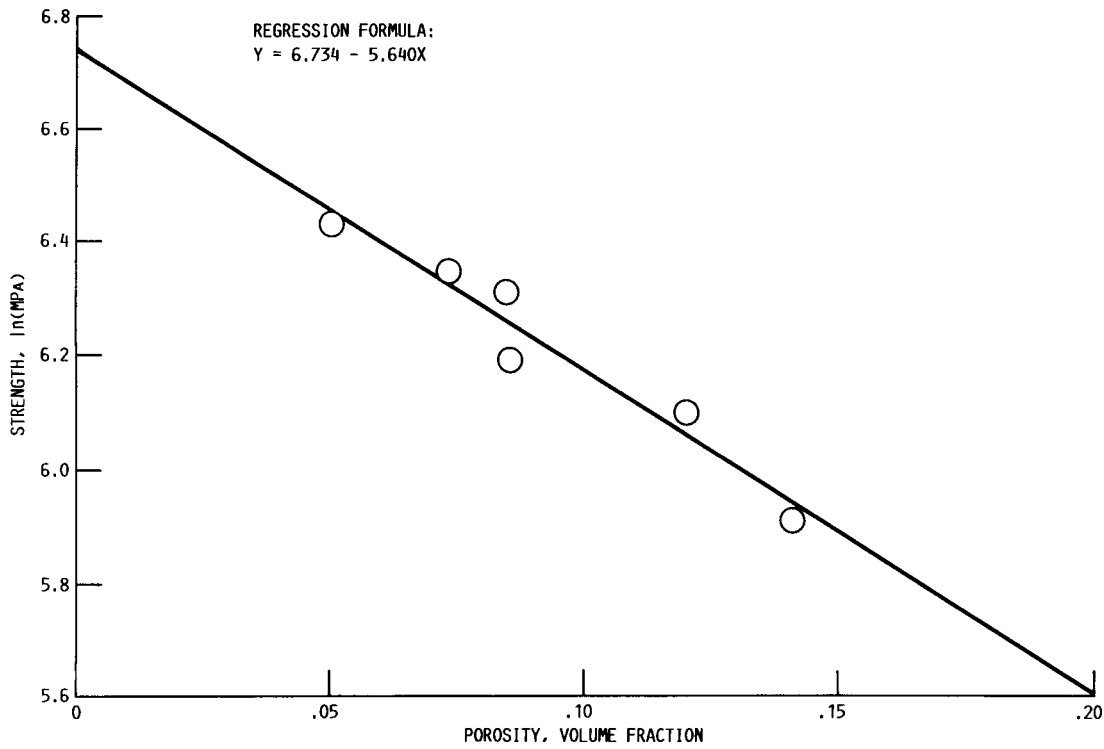


FIGURE 5. - ROOM-TEMPERATURE FLEXURAL STRENGTH OF HIPed α -SiC(I) AS FUNCTION OF POROSITY.

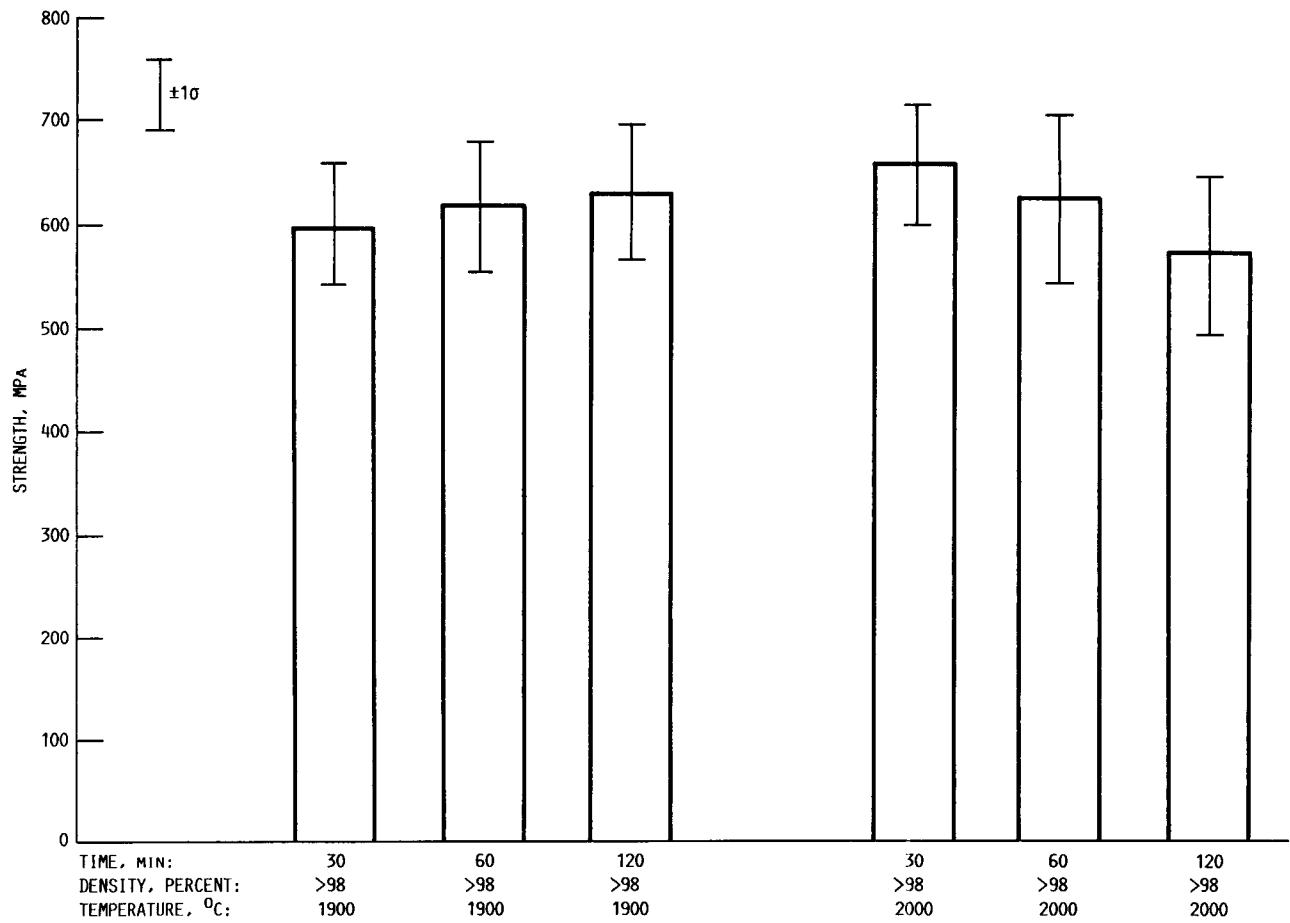
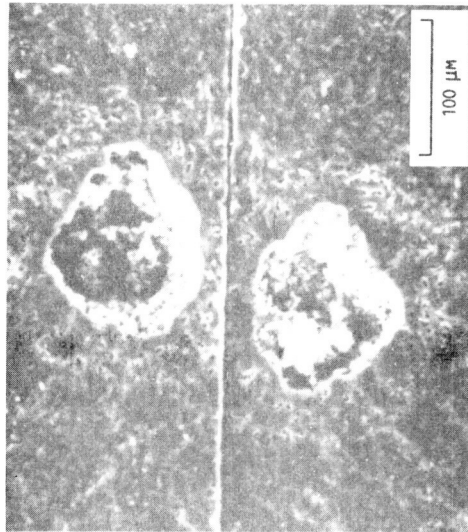


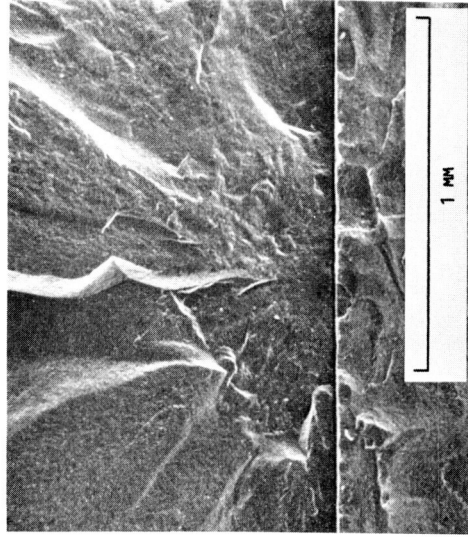
FIGURE 6. - ROOM-TEMPERATURE FLEXURAL STRENGTH OF α -SiC(II) HIPed AT DIFFERENT TEMPERATURES AND TIMES.



(a) α -SiC(II); $\sigma_f = 227$ MPa; SINTERED AT 2150 °C FOR 30 MIN.



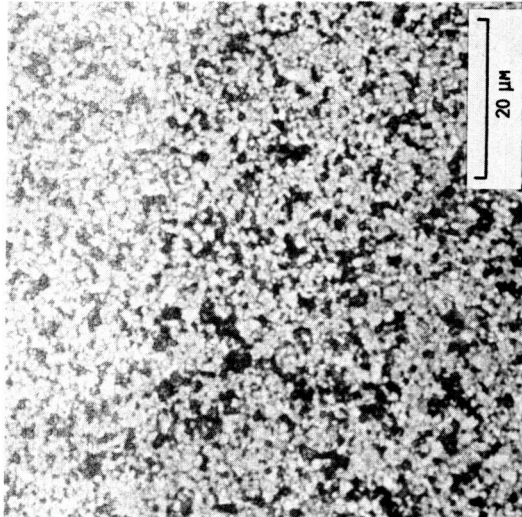
(b) α -SiC(I); $\sigma_f = 492$ MPa; HIPED AT 2000 °C FOR 120 MIN.



(c) α -SiC(II); $\sigma_f = 480$ MPa; HIPED AT 1900 °C FOR 30 MIN.

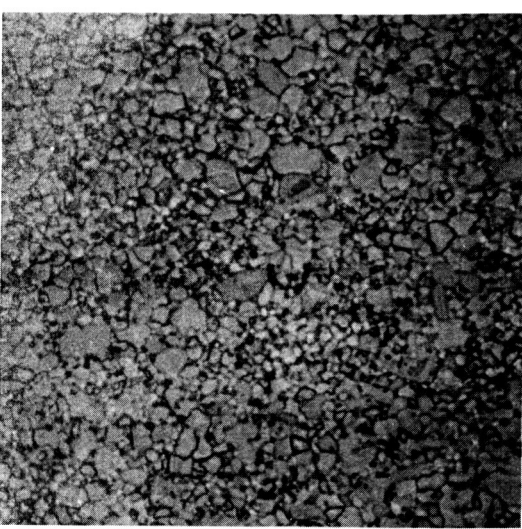
FIGURE 7. - FRACTURE INITIATION SITES IN SINTERED AND HIPED α -SiC BARS.

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(a) α -SiC(II).

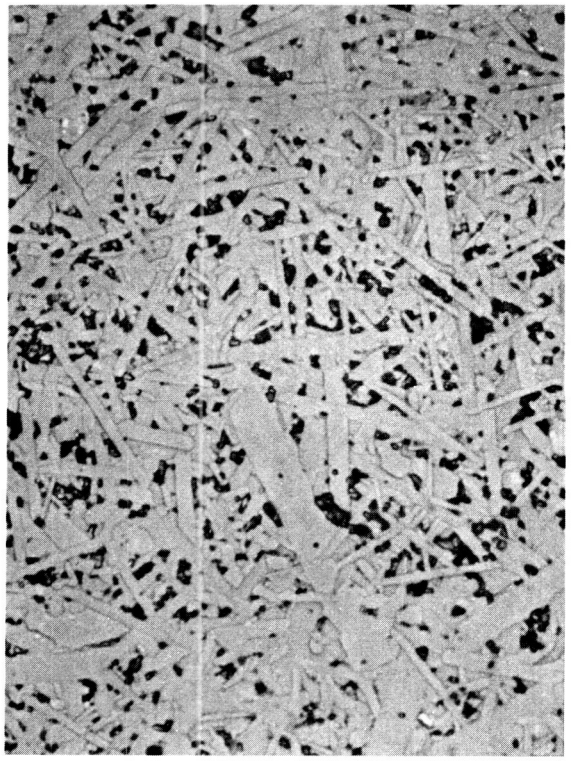
FIGURE 8. - MICROSTRUCTURES OF α -SiC AND β -SiC HIPED AT 1900 °C FOR 60 MIN.



(b) β -SiC(I).



(a) 3 WT% C (DENSITY, 92 PERCENT).



(b) 4 WT% C (DENSITY, 94 PERCENT).

FIGURE 9. - MICROSTRUCTURES OF β -SiC(I) SINTERED AT 2150 °C FOR 60 MIN.

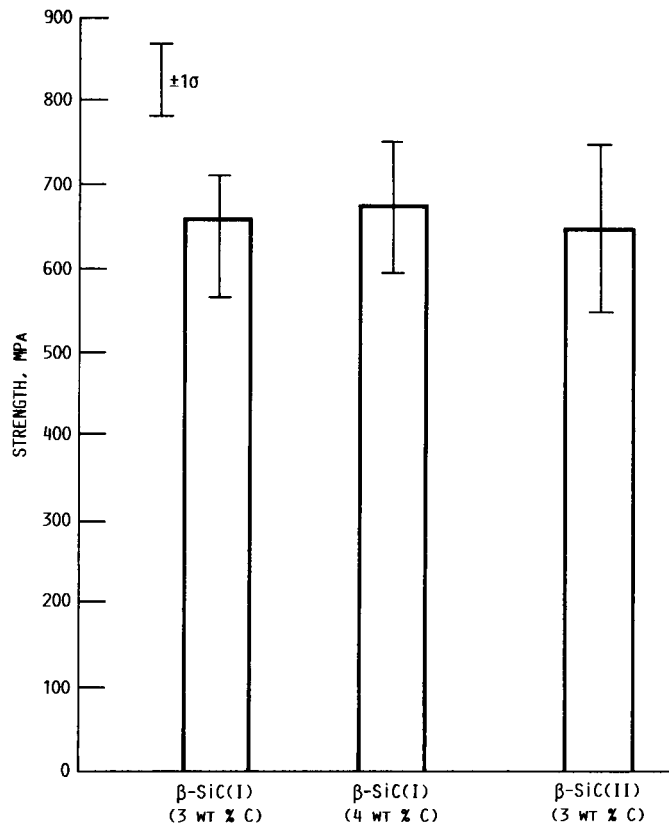


FIGURE 10. - ROOM-TEMPERATURE FLEXURAL STRENGTH OF β -SiC HIPED AT 1900 °C FOR 60 MIN.

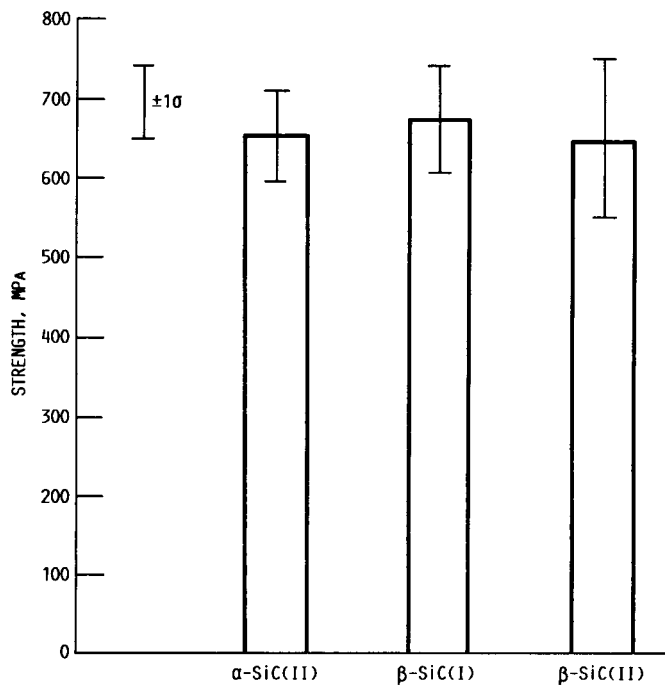
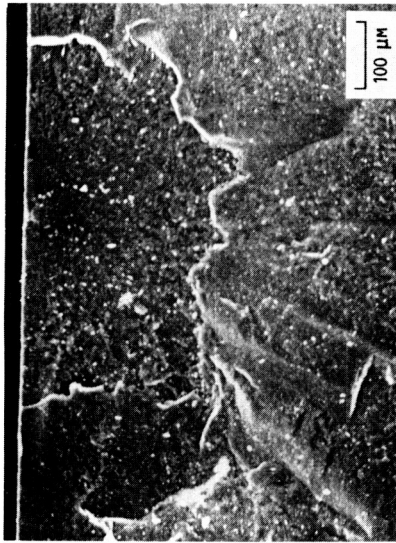


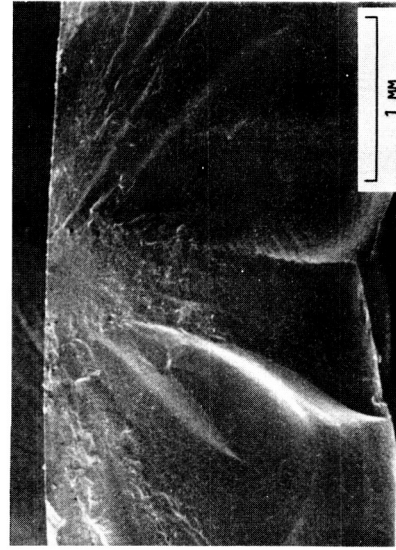
FIGURE 11. - ROOM-TEMPERATURE FLEXURAL STRENGTH OF α -SiC AND β -SiC HIPED AT 1900 °C FOR 60 MIN.



(a) β -SiC(1): $\sigma_f = 700$ MPA; 3 WT% C.



(b) β -SiC(1): $\sigma_f = 745$ MPA; 4 WT% C.



(c) β -SiC(1): $\sigma_f = 506$ MPA; 3 WT% C.

FIGURE 12. - SURFACE FRACTURE INITIATION SITES IN β -SiC BARS HIPED AT 1900 °C FOR 60 MIN.

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1. Report No. NASA TM-101400		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle High-Strength Silicon Carbides by Hot Isostatic Pressing				5. Report Date	
				6. Performing Organization Code	
7. Author(s) Sunil Dutta				8. Performing Organization Report No. E-4484	
				10. Work Unit No. 505-63-1A	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135-3191				11. Contract or Grant No.	
				13. Type of Report and Period Covered Technical Memorandum	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546-0001				14. Sponsoring Agency Code	
15. Supplementary Notes Prepared for the Third International Symposium on Ceramic Materials and Components for Engines sponsored by the American Ceramic Society, Las Vegas, Nevada, November 27-30, 1988.					
16. Abstract Silicon carbide has strong potential for heat engine hardware and other high-temperature applications because of its low density, good strength, high oxidation resistance, and good high-temperature creep resistance. Hot isostatic pressing (HIP) was used for producing α and β silicon carbide (SiC) bodies with near-theoretical density, ultrafine grain size, and high strength at processing temperatures of 1900 to 2000 °C. The HIPed materials exhibited ultrafine grain size (0.3 to 5 μm for α -SiC and $<1.00 \mu\text{m}$ for β -SiC). Furthermore, no phase transformation from β to α was observed in HIPed β -SiC. Both materials exhibited very high average flexural strength; as high as 650 MPa for α -SiC and 673 MPa for β -SiC. It was also shown that α -SiC bodies without any sintering aids (such as boron and carbon), when HIPed to high final density, can exhibit very high strength. Fracture toughness K_{IC} values were determined to be 3.6 to 4.0 MPa $\text{m}^{1/2}$ for HIPed α -SiC and 3.7 to 4.1 MPa $\text{m}^{1/2}$ for HIPed β -SiC. In the HIPed specimens strength-controlling flaws were typically surface related. In spite of improvements in material properties such as strength and fracture toughness by elimination of the larger strength-limiting flaws and by grain size refinement, HIPing had no effect on Weibull modulus.					
17. Key Words (Suggested by Author(s)) Processing; Hot isostatic pressing; Strength; Microstructure; Fracture			18. Distribution Statement Unclassified - Unlimited Subject Category 27		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No of pages 18	22. Price* A03