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OAK RIDGE NATIONAL LABORATORY

MARTIN MARIETTA

HIGH TEMPERATURE HEXOLOY™ SX SILICON CARBIDE

FINAL REPORT

G. V. Srinivasan
S. K. Lau
R. S. Storm

CERAMIC TECHNOLOGY PROJECT

MANAGED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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ABSTRACT

HEXOLOY®[1] SX-SiC, fabricated with Y and Al containing compounds as sintering aids, has been shown to possess significantly improved strength and toughness over HEXOLOY® SA-SiC. This study was undertaken to establish and benchmark the complete mechanical property database of a first generation material, followed by a process optimization task to further improve the properties. Detailed mechanical characterizations were then conducted on the optimized material.

Mechanical characterization on the first generation material indicated that silicon-rich pools, presumably formed as a reaction product during sintering, controlled the strength from room temperature to 1232°C. At 1370°C in air, the material was failing due to a glass-phase formation at the surface. This glass-phase formation was attributed to the reaction of yttrium aluminates, which exist as a second phase in the material, with the ambient. This process was determined to be a time-dependent one that leads to slow crack growth. Fatigue experiments clearly indicated that the slow crack growth driven by the reaction occurred only at temperatures >1300°C, above the melting point of the glass phase. Experimental results also revealed that this material possesses excellent creep resistance, though the creep mechanism is not yet well understood.

Process optimization tasks conducted included the selection of the best SiC powder source, studies on mixing/milling conditions for SiC powder with the sintering aids, and a designed experiment involving a range of sintering and post-treatment conditions. During the course of the mixing/milling study it was found that the turbomilling process provided the best room-temperature strengths ever achieved (>1 GPa) when conducted on a small batch of powders. It was, however, found that more development was needed to effectively translate the milling conditions to a larger batch.

The optimization study conducted on the densification variables indicated that lower sintering temperatures and higher post-treatment pressures reduce the Si-rich pool formation, thereby improving the room-temperature strength. In addition, it was also determined that furnacing configuration and atmosphere were critical in controlling the Si-rich formation.

[1]HEXOLOY is a trademark of The Carborundum Company registered in the U.S. Patent & Trademark Office.

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INTRODUCTION

HEXOLOY®[1] SA Silicon Carbide, a pressureless sintered α-SiC, has shown excellent oxidation, corrosion, erosion, and wear resistance. Its toughness and strength retention at elevated temperatures up to 1500°C in air have also been demonstrated. Powder availability, price competitiveness, and net-shape-forming capability make this SiC material an attractive candidate for many structural applications. However, relatively low strength (primarily due to its low toughness) still represents limitations for some applications involving higher stresses such as rotating components in advanced gas turbine engines. Various approaches have been considered to enhance its fracture toughness and strength. Among them the key ones are based on the modification of microstructure and fracture characteristics of the material by the incorporation of appropriate sintering additives and variation of processing conditions. Using these approaches, Carborundum recently developed an improved SiC material, namely HEXOLOY® SX SiC, which is sintered with the addition of yttrium and aluminum compounds.

HEXOLOY® SX SiC had been demonstrated to possess a higher toughness and strength than HEXOLOY® SA SiC[1]. Its toughness was about 50% to 100% higher than that of SA and its typical room- temperature MOR value ranged between 620-915 MPa (90-133 ksi). However, the available database was preliminary in nature. A detailed characterization was necessary to completely establish the mechanical property database for benchmarking and, more importantly, to understand the failure mechanisms and establish microstructure-property correlations. Using the structure-property correlation, it was believed that the mechanical properties could be further improved via proper optimization of composition and processing conditions.

SCOPE AND OBJECTIVE

The approach taken for the current work was first to establish a complete mechanical property database and conduct detailed microstructural characterization on the first generation SX material. Then a process optimization study was carried out to further enhance the high-temperature mechanical properties. Simultaneously, a Carborundum in-house sponsored program with the objective of identifying a second generation additive composition with improved high-

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Research sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies, as part of the Ceramic Technology Project of the Propulsion System Materials Program, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

temperature properties was conducted. Then a second set of designed experiments was to be conducted to optimize the properties of this second generation SX material. Finally, the complete property database was to be established for the second generation composition.

The three major objectives for this program were as follows: (1) establish a property database and conduct detailed characterization for the best SX material, (2) improve the processing conditions of that material via a designed experimental method, and (3) develop a second generation SX material with superior high-temperature properties. To achieve these objectives the work was split into six tasks:

Task	Objective	Duration
1	Complete Characterization of First Generation SX	April '91-Dec. '91
2	Selection of SiC Powder Source	Aug. '91-Dec. '91
3	Develop Improved Dispersion Technique	Aug. '91-Dec. '91
4	Property Optimization of First Generation SX	Jan. '92-April '92
5a	Property Optimization of Second Generation SX	May '92-Aug. '92
5b	Complete Characterization of Second Generation SX	Sept. '92-Dec. '92

From previous internal work at Carborundum, a particular composition of SX, designated SX-G1, with 2 wt % total additive, had shown excellent MOR strengths. Preliminary evaluation of its creep and dynamic fatigue resistance was also encouraging. SX-G1 was, therefore, chosen as the composition for the work to be conducted in Tasks 1, 2, 3, and 4 of this contract.

Task 1: Complete Characterization of First Generation SX-SiC Material.

In this task, a complete mechanical and microstructural characterization was conducted. Mechanical characterization included MOR, tensile strength, and K_{Ic} determinations at room and elevated temperatures; stress rupture, dynamic fatigue, and creep measurements at elevated temperatures. Microstructural characterization included: X-ray diffraction; quantitative image analysis; optical, scanning electron, and Auger microscopies.

Experimental Procedure

Sample Preparation: Two 20-lb batches of a commercial SiC powder from source A were mixed with the appropriate amounts of yttrium and aluminum compound sintering additives and spray dried into soft, flowable agglomerates. The powder was compacted into 63.5-mm-square plates and subsequently isostatically pressed to 117 MPa. The green plates were pressureless sintered and post-treated at elevated temperatures and pressures to >99% theoretical density. The plates were then machined into flexural bars of 3 x 4 x 48 mm. In fabricating the tensile specimens, green rods of 216 mm in length and 21 mm in diameter were isostatically pressed to 117 MPa. They were then pressureless sintered to about 96% theoretical density, which was next enhanced to >99% by a post-treatment process. A higher sintering temperature was required to achieve this density as compared to that used for the plates. The densified rods were then machined into ORNL buttonhead-type tensile specimens.

Mechanical Properties Evaluation: A detailed microstructural and mechanical property characterization was conducted. The mechanical properties evaluated included fracture toughness, flexural strength, tensile strength, dynamic fatigue, stress rupture, and creep.

The fracture toughness (K_{Ic}) was determined by the Chevron-notch technique. Chevron notches were cut in the bars (3 x 4 x 48 mm) which were subsequently fractured at a cross-head speed of 0.5 mm/min in four-point bending. K_{Ic} was evaluated using the fracture load from five samples each at room temperature, 1000, 1232, and 1370°C.

Flexural strength was evaluated using MIL-STD-1942 on bars (3 x 4 x 48 mm) in four-point bending at a cross-head speed of 0.5 mm/min with 20- and 40-mm inner and outer spans. Flexural strength was evaluated from 20 bars each at room temperature, 1000, 1232, and 1370°C.

The strength of the SX-G1 samples was also determined in uniaxial tension. The specimens were machined according to the ORNL buttonhead tensile specimen specification. The specimens were then tested at a stressing rate of 11 MPa/s using self-aligning Instron Super Grip™ hydraulic

couples in the load train to minimize specimen bending. The uniaxial tensile strengths were determined at room temperature, 1000, 1232, and 1370°C. These tension tests were carried out at the ORNL/HTML User Facility.

Dynamic fatigue tests were conducted at 1232 and 1370°C in four-point bending using MIL-STD-1942 flexure bars. About six specimens were tested at each of three loading rates covering three orders of magnitude. Stress-rupture experiments were also conducted at 1232 and 1370°C in four-point bending. The load-point deflection of each specimen was monitored during the testing.

Creep tests were conducted in uniaxial tension at 1260, 1370, and 1450°C. Strain rates were determined using extensometers at several temperatures ranging from 1260 to 1450°C and at several stress levels ranging from 50 to 250 MPa.

Extensive microstructural and fractographic analyses were conducted using both optical and scanning electron microscopy (SEM). When needed, scanning auger microscopy (SAM) was also used to identify the chemistry of the fracture origin(s).

Results

Microstructural Characterization: The typical microstructure for a polished surface of a SX-G1 sample is shown in Figure 1. The brighter regions in the backscattered SEM image are deduced by elemental mapping to be YAG. XRD also revealed that the major second phase in the SX-G1 is YAG. An etched microstructure is shown in Figure 2. The SiC grains are equiaxed with a narrow grain-size distribution. The average grain size is estimated to be 1.5 μm.

Toughness and Strength: The fracture toughness (K_{Ic}) determined as a function of temperature is shown in Figure 3. The K_{Ic} of SX-G1 decreases from 4.05 MPa·m$^{1/2}$ at room temperature to 2.6 MPa·m$^{1/2}$ at 1370°C. The K_{Ic} for single-phase α-SiC is also shown for comparison[2]. Note that the K_{Ic} for α-SiC remains unchanged at all temperatures, while K_{Ic} for SX-G1 decreases at elevated temperatures.

It has been proposed that the increase in toughness of SX relative to SA is related to a microcracking mechanism that resulted from residual stresses developed by the coefficient of thermal expansion (CTE) mismatch between SiC and YAG[3]. At elevated temperatures the residual stresses are reduced and a decrease in fracture toughness is expected. The observation that toughness decreases with increasing temperatures is consistent with the proposed toughening mechanism in SX-SiC materials.

The strength of SX-G1 measured from four-point bending and uniaxial tension at various temperatures is shown in Figure 4. Note that the variation in uniaxial tensile strength with temperature is similar to that of the flexural strength. However, at a given temperature the tensile strengths are consistently lower due to the larger effective volume of the buttonhead specimen. The strength decrease with an increase in temperature is consistent with the K_{Ic} reduction at high temperature.

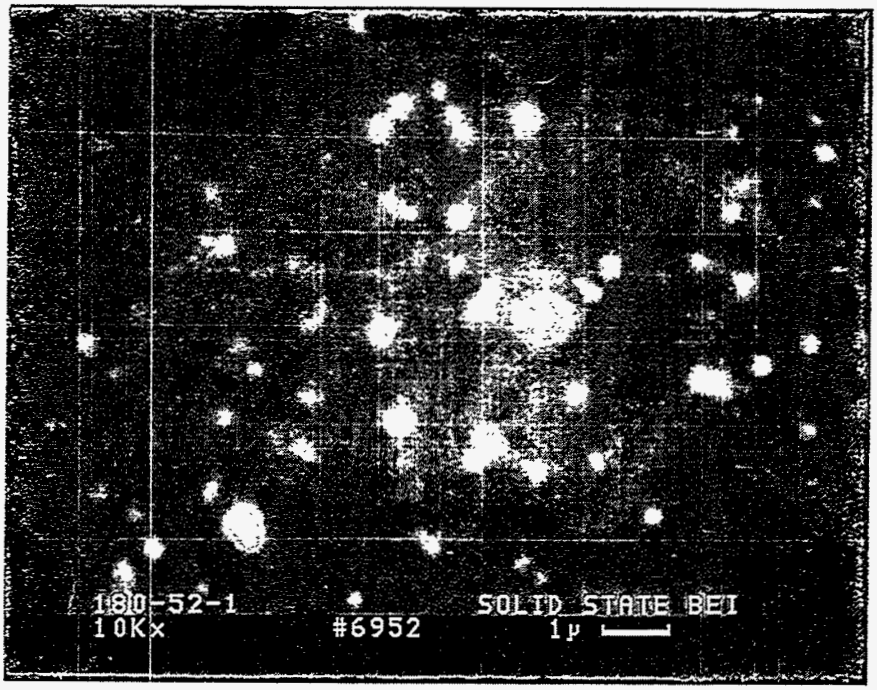

Figure 1. Microstructure of SX-Gl from a Polished Surface.

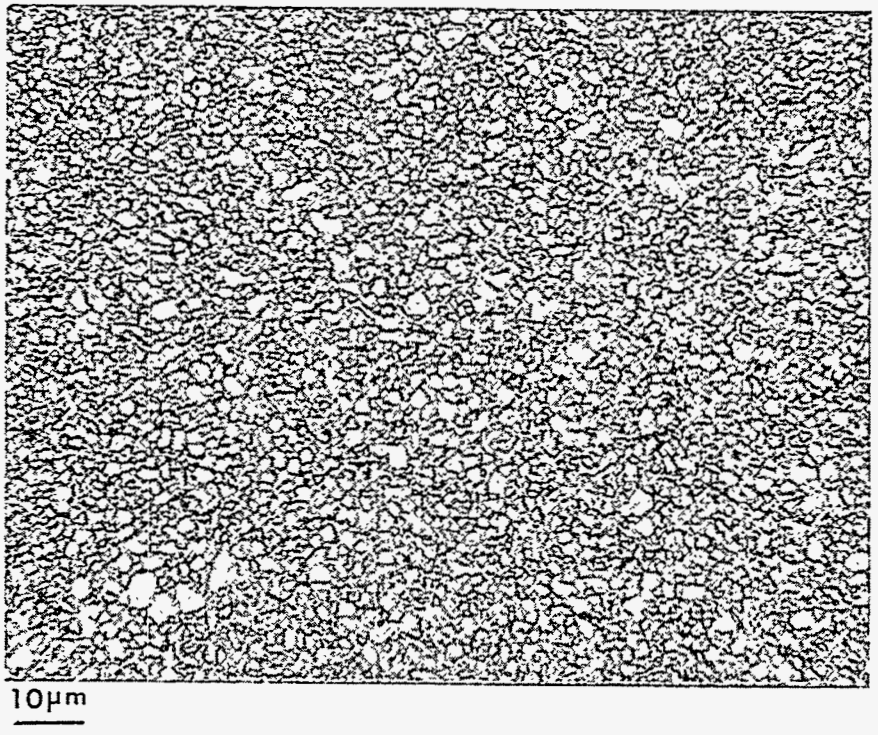

Figure 2. Microstructure from an Etched Surface.

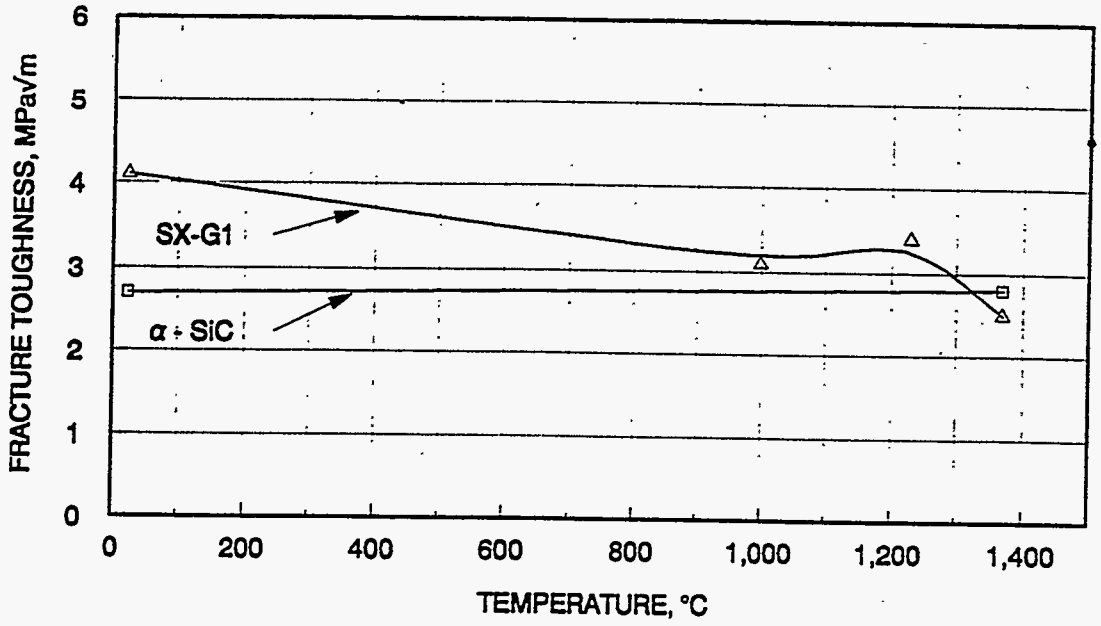

Figure 3. Fracture Toughness at Various Temperatures.

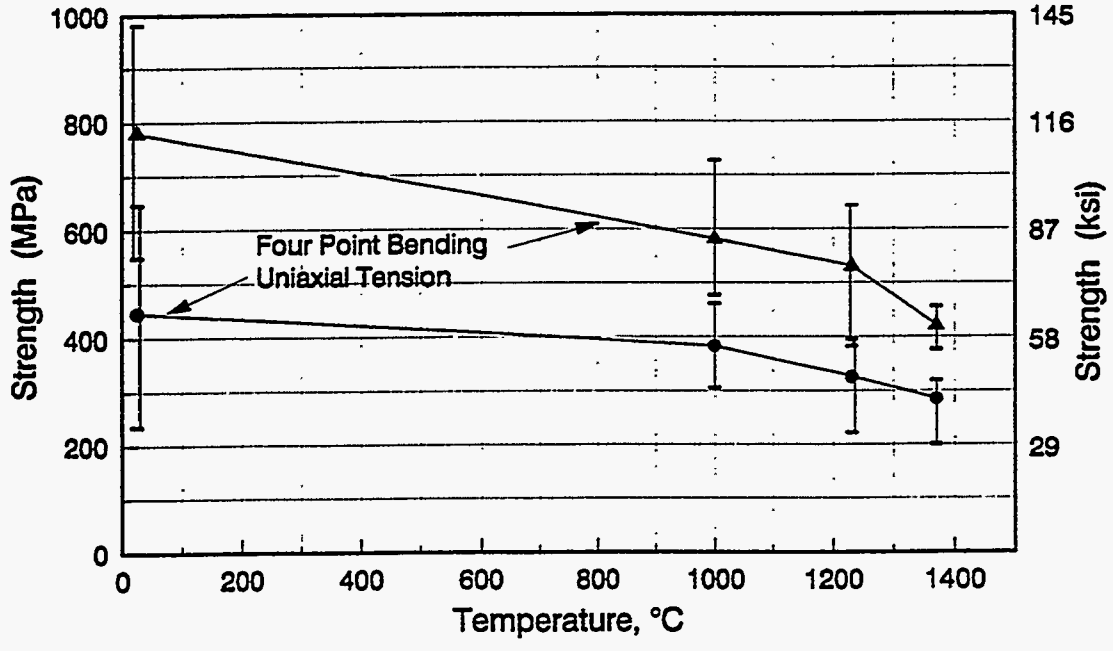

Figure 4. Flexural and Uniaxial Tensile Strength at Various Temperatures.

Strength-Limiting Defects: Optical fractography was performed on all of the fractured specimens. Selected specimens were analyzed using SEM and SAM. Figure 5 shows a fracture surface and failure origin from a typical flexure bar tested at room temperature. The same sample was examined in the SAM. Elemental mapping obtained from the SAM suggests that the fracture origin consisted of elemental silicon and Y-Al-O phases as seen in Figure 6.

Similar volume flaws were identified as the strength-limiting defects in all the tensile specimens tested at room temperature, 1000, and 1232°C. Figures 7 and 8 show typical fracture origins which are very similar to those observed in the flexural specimens.

Although the strength-limiting flaws are associated with the formation of elemental silicon and an associated void area, the starting powder premix does not contain any significant amount of elemental Si to explain the presence of Si in all the specimens. One possibility is, therefore, that the Si forms as a product of some reaction between the Y-Al-O secondary phase and SiC matrix as given below:

$$YAlO_x + SiC \text{ -----> } YAlO_y + Si + CO$$

At higher temperatures, further reduction occurs to produce additional elemental Si:

$$YAlO_y + SiC \text{ -----> } Y_2O_3 + Al + Si + CO$$

The existence of these reaction products can be seen using SAM in samples sintered at higher temperatures as shown in Figure 9. Similar reactions have been reported in the system SiC-Al_2O_3-Y_2O_3 during sintering by Omori and Takei[4].

The fracture behavior at 1370°C was different from that noted above for lower temperatures. In the 1370°C flexural tests, all the specimens failed from the surface. Figure 10 shows a typical fracture surface and failure origin. Note the appearance of a "glassy phase" along the fracture origin. Elemental mapping of the "glassy phase" was conducted using SAM and is shown in Figure 11. The composition of the "glassy phase" contains Y, Al, Si, and O. This phase could be yttrium alumino silicate glass. When the tensile specimens were tested at 1370°C under a lower stress rate (0.11 MPa/s), the failure originated from the surface as shown in Figure 12. Such failure origins were very similar to those observed on the flexural specimens tested at 1370°C. Presumably the SiO_2 formed by the oxidation of SiC reacted with the Y-Al-O secondary phase to form a low-melting silicate that was responsible for the high-temperature failure origin. However, when the tensile specimens were tested at 1370°C and at higher stress rates (11 MPa/sec.), the failures were from a volume flaw as shown in Figure 13. This flaw is very similar to those shown in Figures 7 and 8 for low temperatures. It appears that the formation of this glass phase is a time-dependent phenomenon. The stress-strain plots for tension tests performed at 1370°C for

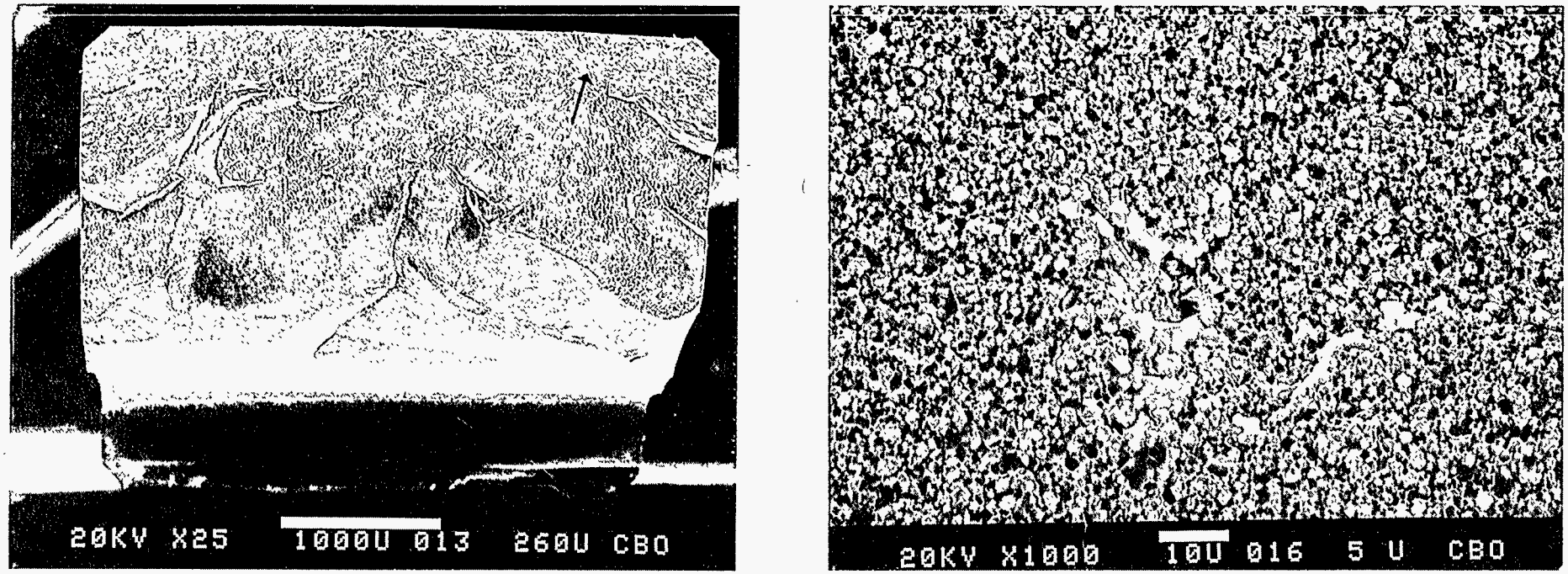

Figure 5. A Typical "Silicon-Rich Pool" as a Fracture Origin in a Flexural Bar Tested at Room Temperature. Flexural Strength = 875 MPa (127 ksi).

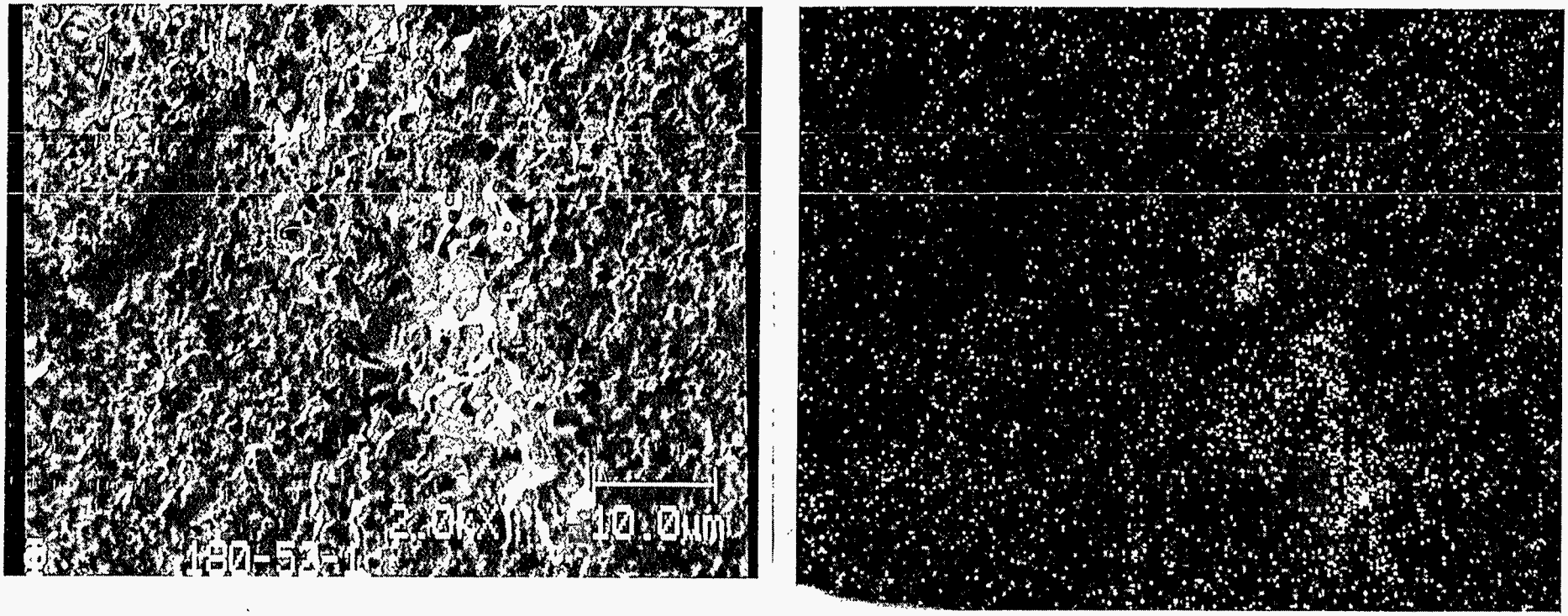

Figure 6. Elemental Mapping of a Strength-Limiting Pool from a Scanning Auger Microscope (SAM).

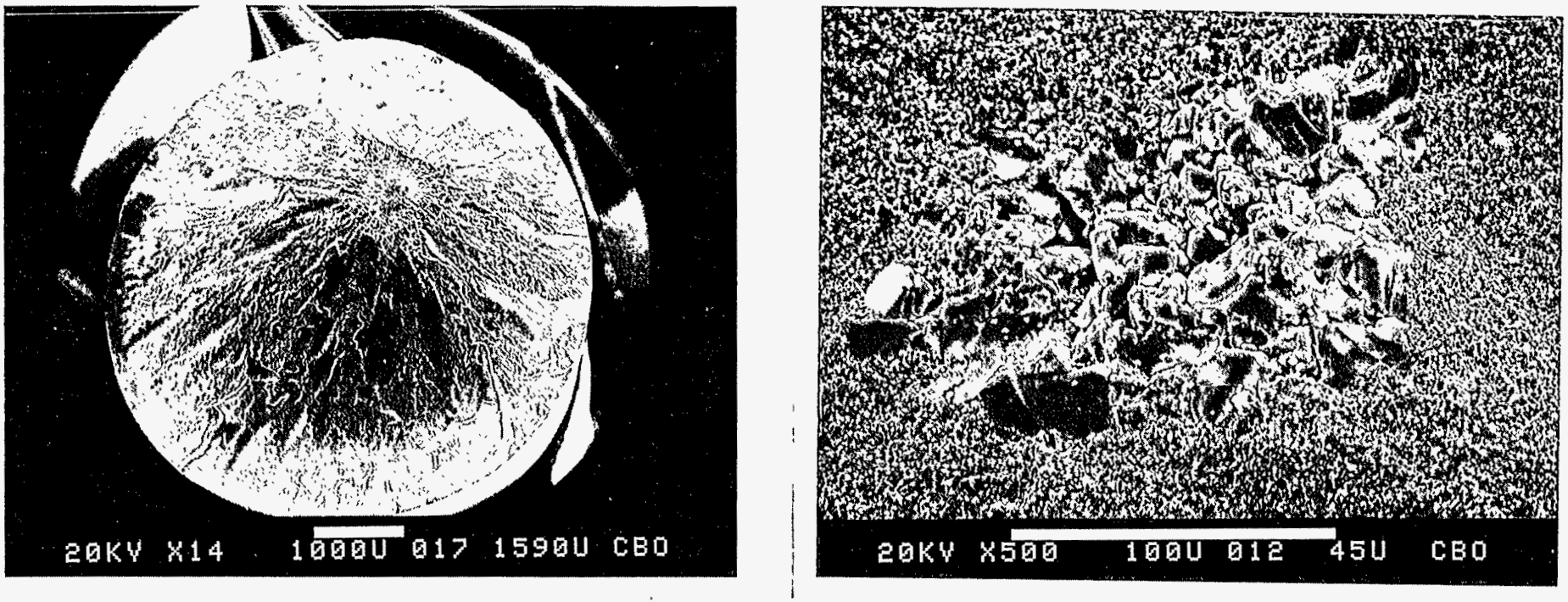

Figure 7. Similar "Silicon-Rich Pool" in a Tensile Sample Tested at Room Temperature.
Tensile Strength = 411 MPa (59.6 ksi).

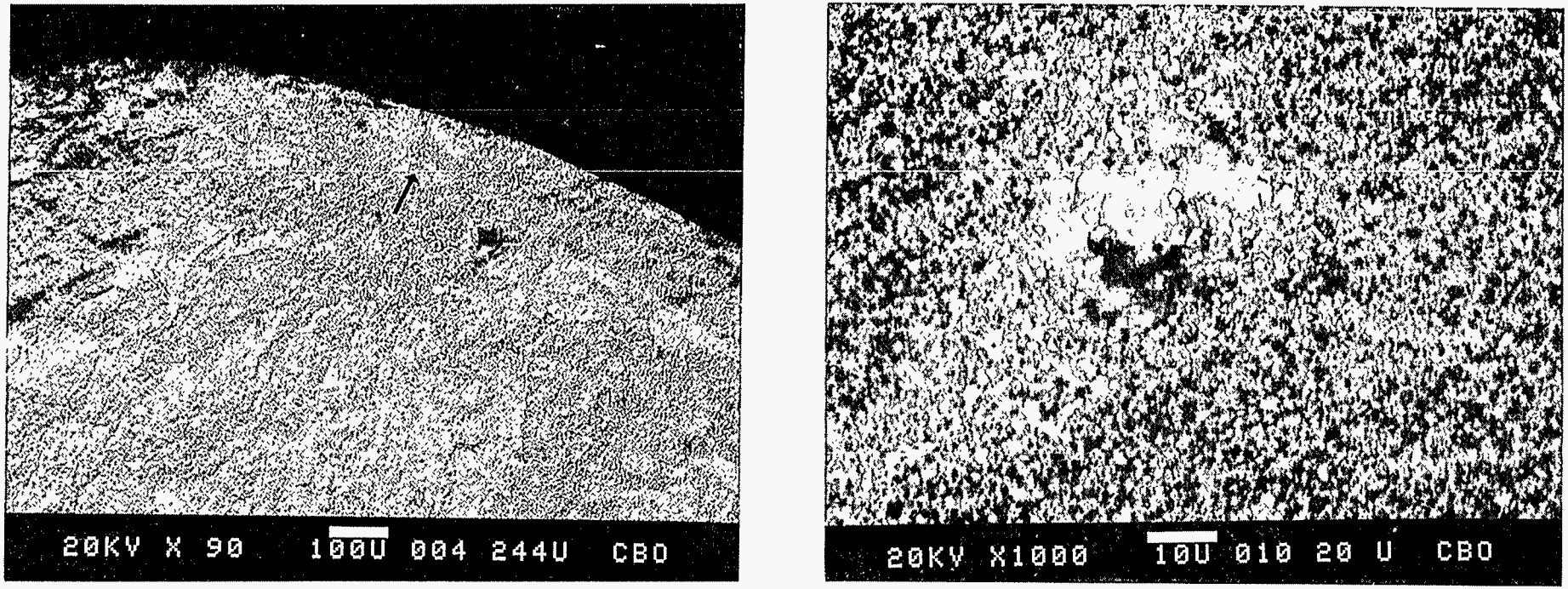

Figure 8. "Si-Rich Pool" in a High Strength Tensile Sample Tested at Room Temperature.
Tensile Strength = 567 MPa (82 ksi).

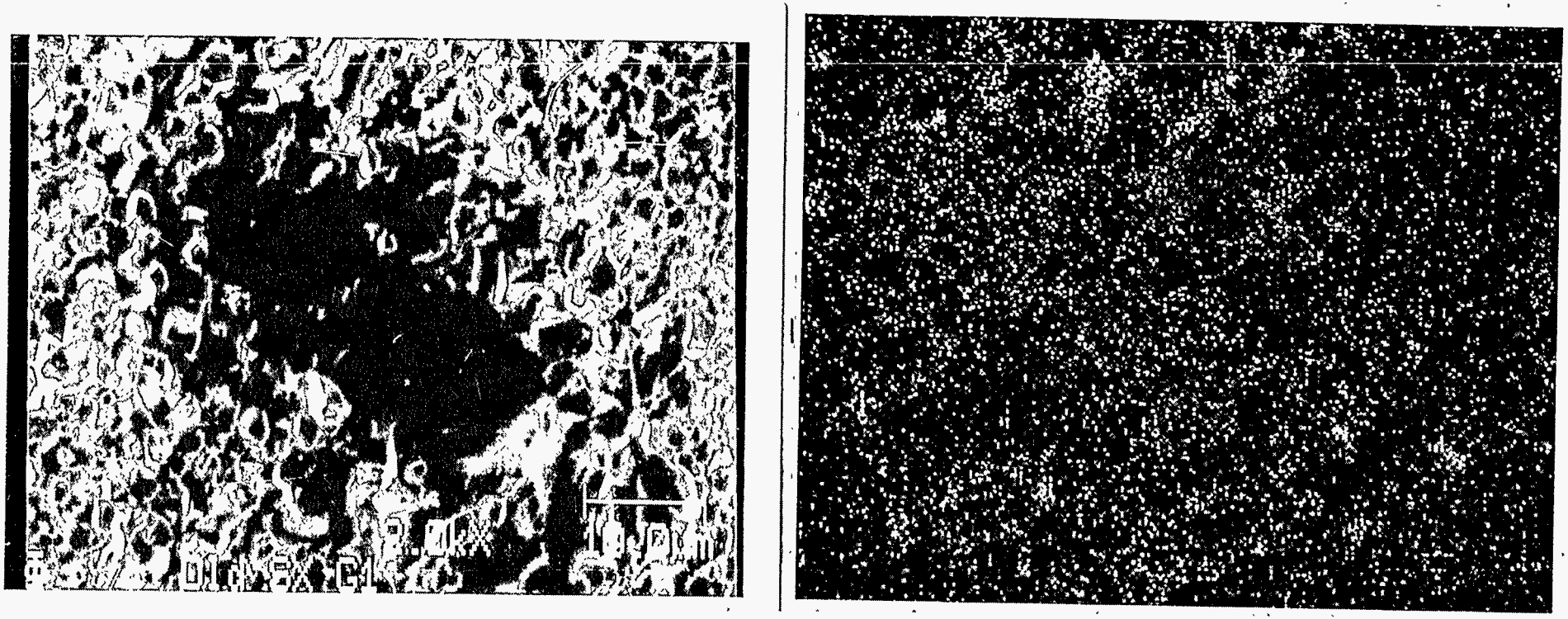

Figure 9. Elemental Mapping of a Strength-Limiting Pool from a Sample Sintered at a Higher Temperature.

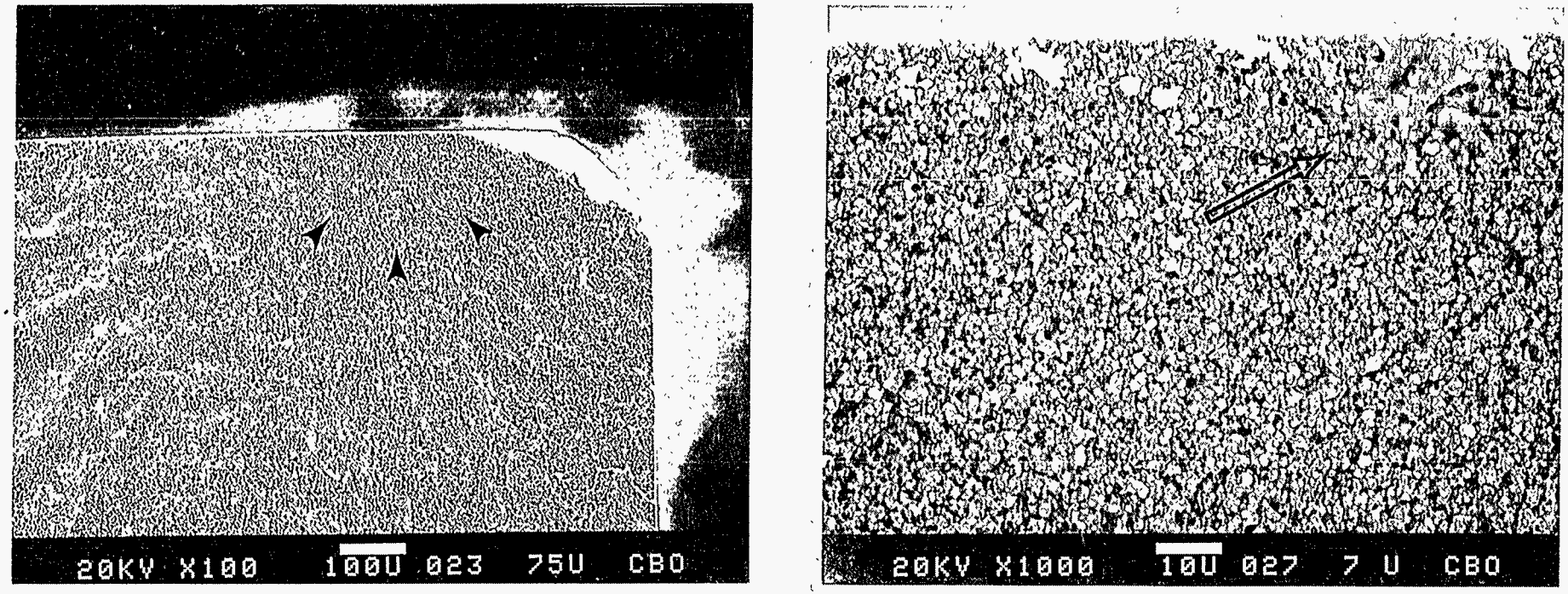

Figure 10. Fracture Origin from a Flexure Bar Tested at 1370°C.
MOR = 420 MPa (61 ksi).

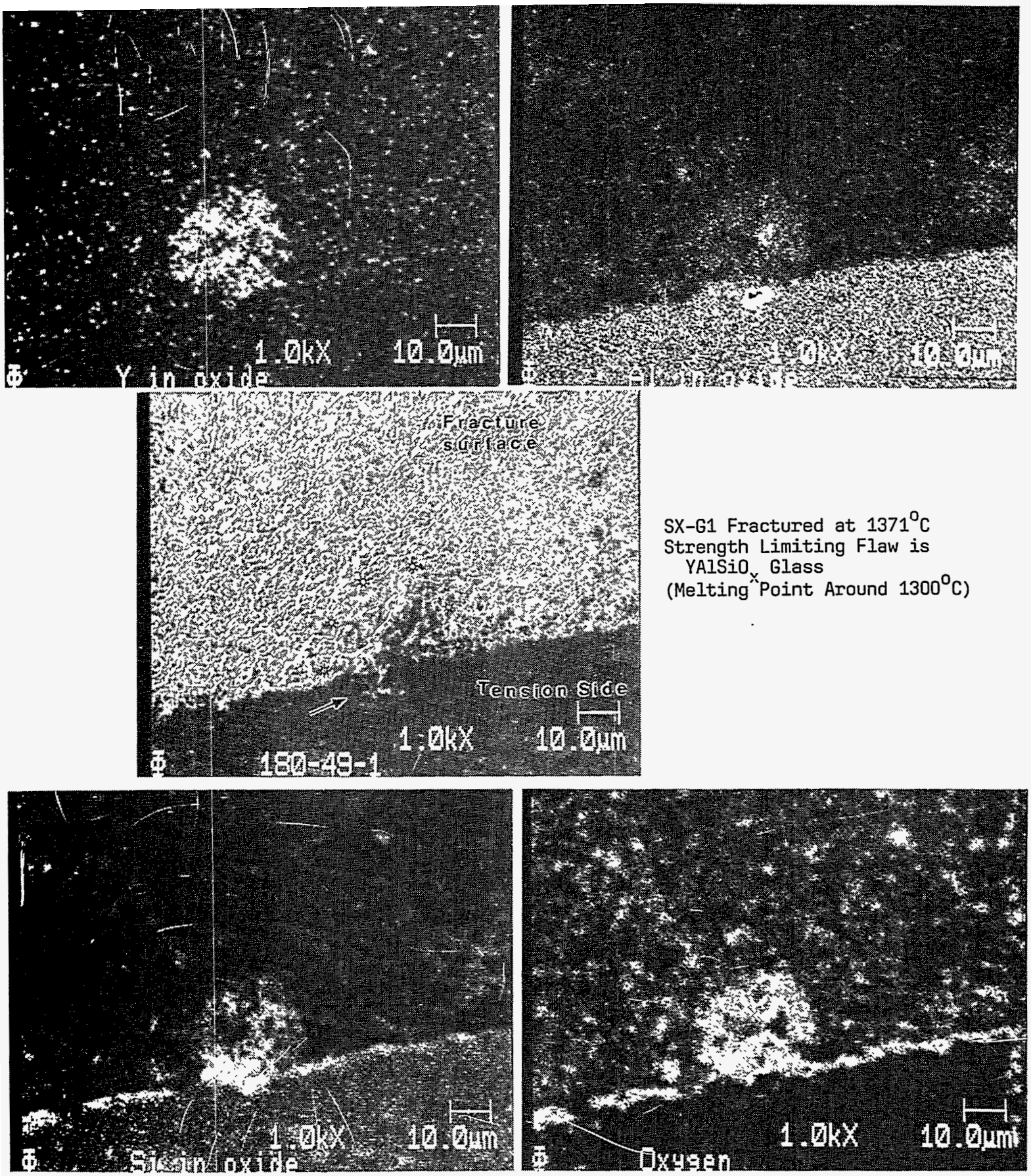

Figure 11. Elemental Mapping of the "Glassy Phase" as Observed in SAM.

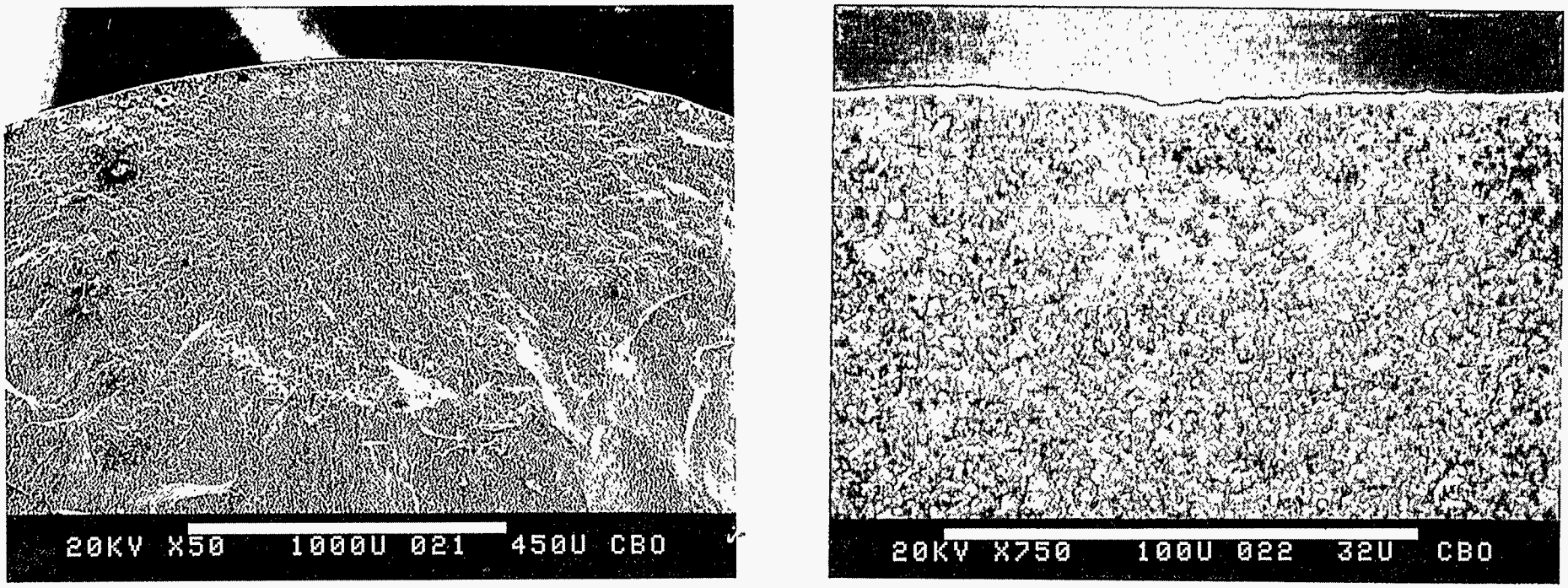

Figure 12. "Glassy Phase" as the Failure Origin in a Tensile Sample Tested at 1370°C and at a Stress Rate of 0.11 MPa/s. Tensile Strength = 279 MPa (40.4 ksi).

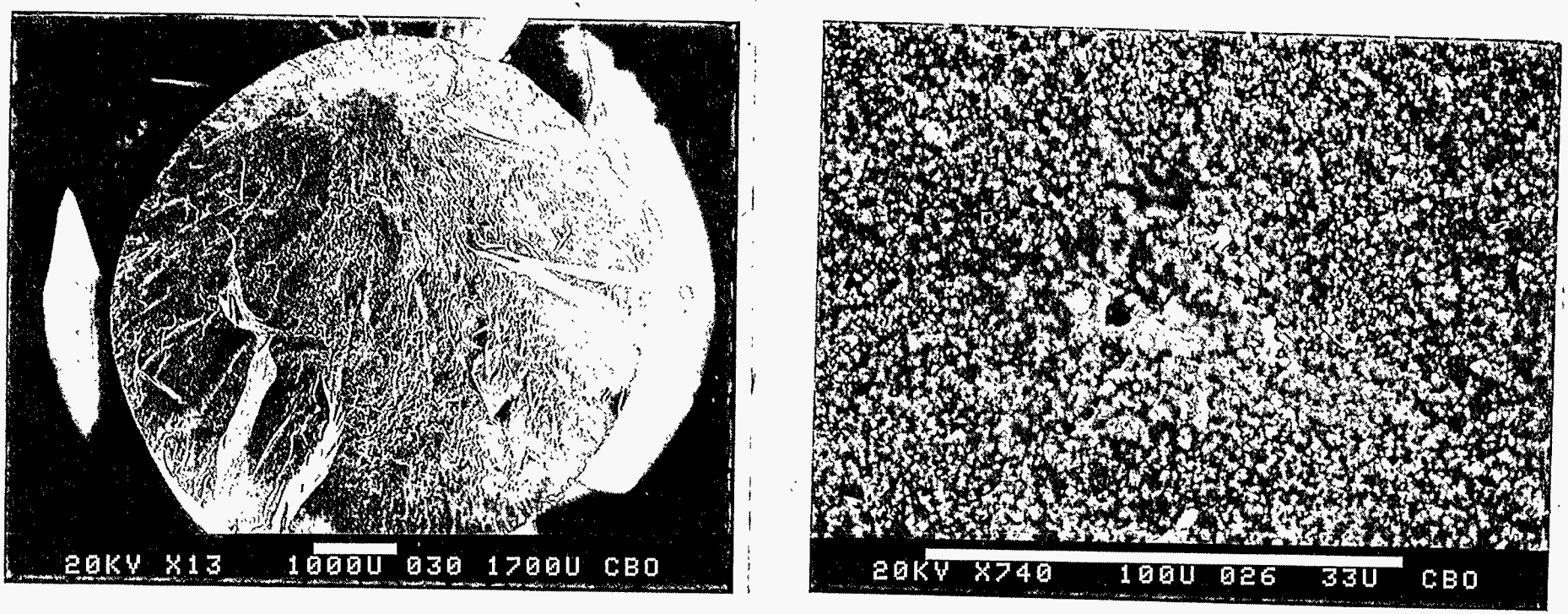

Figure 13. "Si Pool" as Strength-Limiting Defect in a Tensile Sample Tested at 1370°C and at a Stress Rate of 11 MPa/s. Tensile Strength = 309 MPa (44.8 ksi).

various stress rates are shown in Figure 14. The nonlinear behavior at lower stressing rates with the glass-phase formation at the surface suggests probable slow crack growth. The average strengths, Weibull modulus, and the defect type obtained from flexural tests and tension tests are summarized in Tables 1a and 1b.

Fatigue Results: Dynamic fatigue experiments were carried out in four-point flexure at 1232 and 1370°C. Constant stress rates were used, which varied from 0.633 MPa/s to 63.3 MPa/s. The average strengths measured at various stress rates are shown in Figure 15. The slow crack growth parameter is calculated using the relation:

$$\sigma_f = \dot{\sigma}^{1/(N+1)}$$

where σ_f is the fracture strength, $\dot{\sigma}$ is the stressing rate, and "N" is the slow crack growth parameter. The slow crack growth parameter "N" is calculated from the slope of the best fit regression line. At 1232°C there is very little reduction in strength at lower stress rates and hence a very high slow crack growth parameter of 51.6 was obtained. Also, the failures corresponded to volume defects which were very similar to the defects observed in flexural bars used for strength evaluation as shown in Figure 5. However, at 1370°C there was a definite strength reduction at lower stress rates—indicative of slow crack growth (N=14.5). At 1370°C all the specimens failed from surface flaws. The fracture origins were very similar to the one shown in Figure 10.

Stress rupture experiments were also conducted in four-point flexure at 1232 and 1370°C. The failure time for a given stress level was recorded and the applied stress vs failure time plot is shown in Figure 16. The slow crack growth parameter was estimated using the relation

$$t_f \propto \sigma_{app}^{N}$$

where t_f is the failure time, σ_{app} is the constant applied stress, and "N" is the slow crack growth parameter. The slow crack growth parameter was estimated as 75 at 1232°C. Again, a high value of "N" at 1232°C suggests very little or negligible slow crack growth or fatigue behavior. However, the fact that "N" decreased to 16.9 at 1370°C indicates that significant slow crack growth occurred at this temperature. These results are in good agreement with those obtained from the dynamic fatigue flexural results.

The slow crack growth here appears to be associated with the glass-phase formation at the surface. As discussed above, the glass phase, $YAlSiO_X$, might have formed from the reaction of

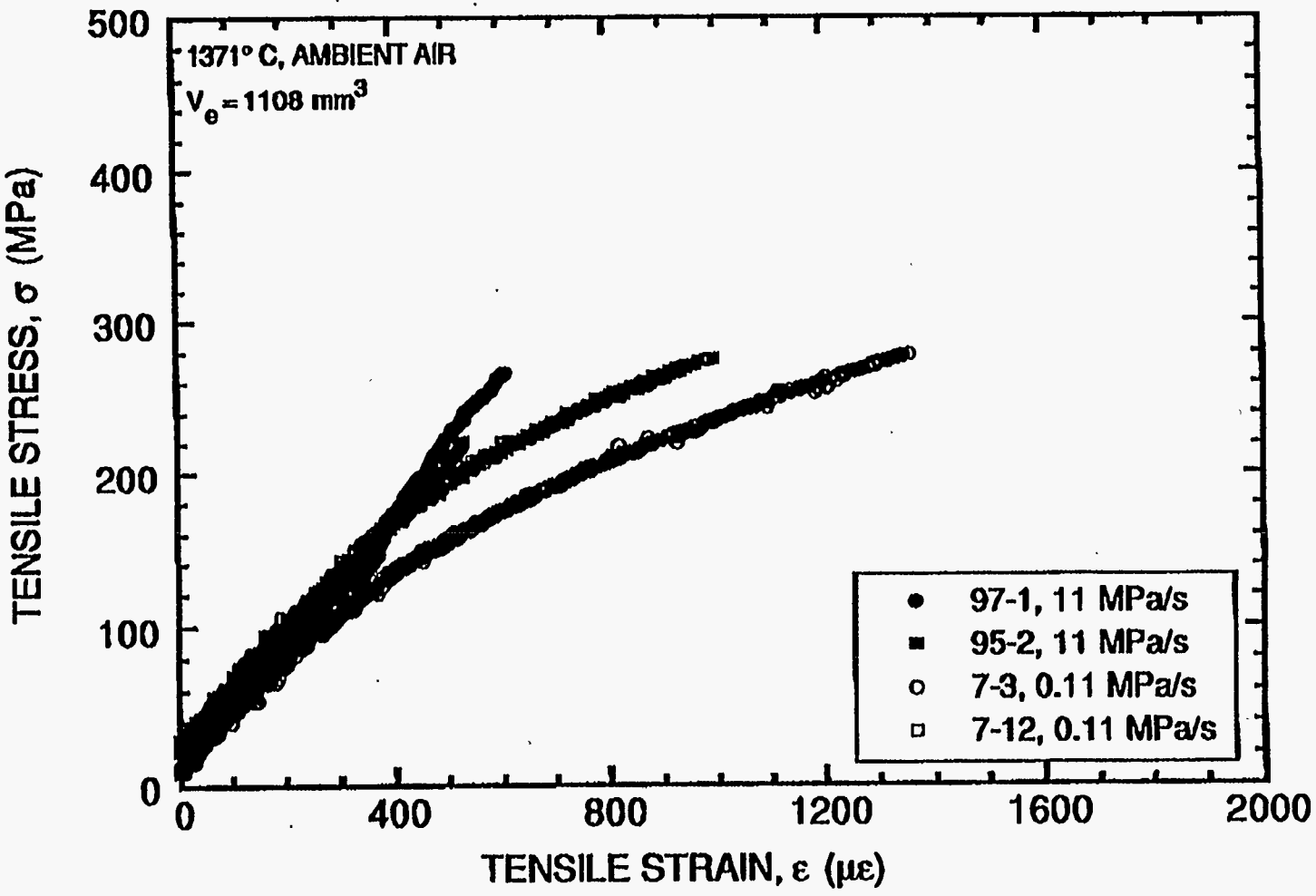

Figure 14. Stress-Strain Plot at 1370°C for Different Stress Rates

TABLE 1a

Flexure Test Data

Temp °C	MOR MPa (ksi)	Weibull Modulus	Flaw
RT	780 (113)	5.3	Volume defects, Si containing region
1000	580 (84)	8.2	"
1232	531 (77)	5.8	"
1370	421 (61)	10.3	Surface, glassy phase formation

TABLE 1b

Tension Test Data

Temp	# of Specimens	Stress Rate MPa/sec	Ultimate Tensile Strength MPa (ksi)	Weibull Modulus	Flaw
RT	15	11	446 (64.7)	3.0	Volume defects, Si containing region
1000	8	11	384 (55.7)	6.5	"
1232	8	11	325 (47.2)	4.3	"
1370	6	11	286 (41.5)	4.5	"
	2	0.11	281 (40.8)	-	Surface, glassy phase formation

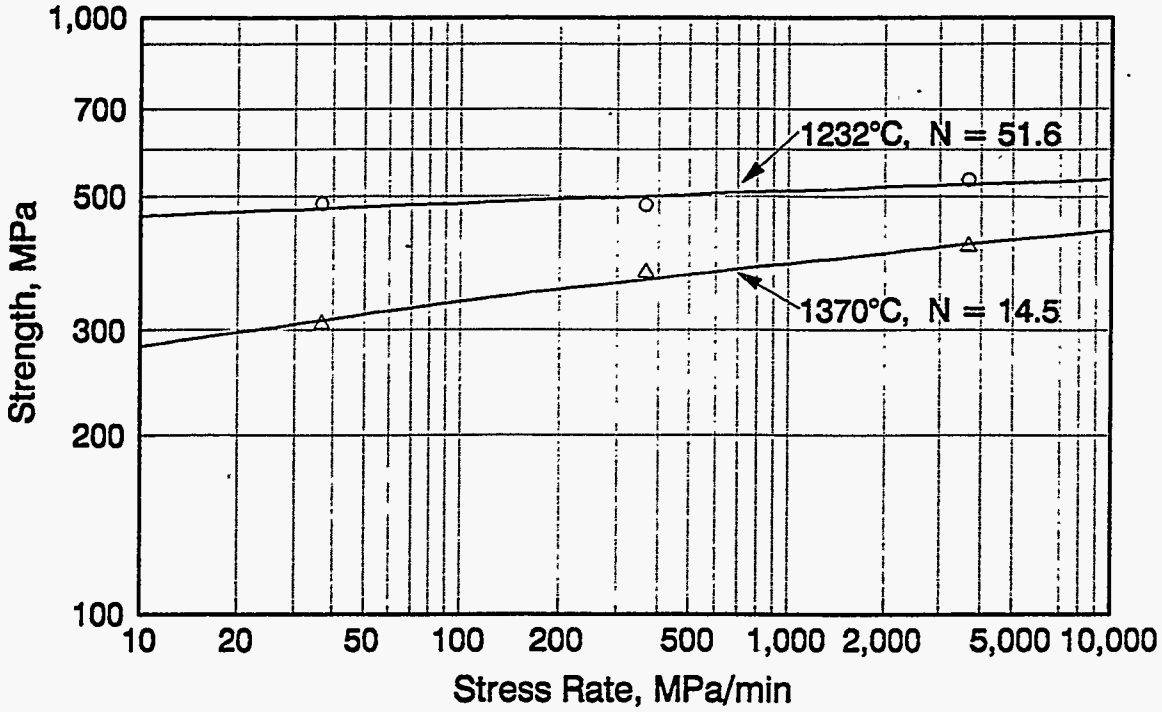

Figure 15. Dynamic Fatigue Response for SX-G1.

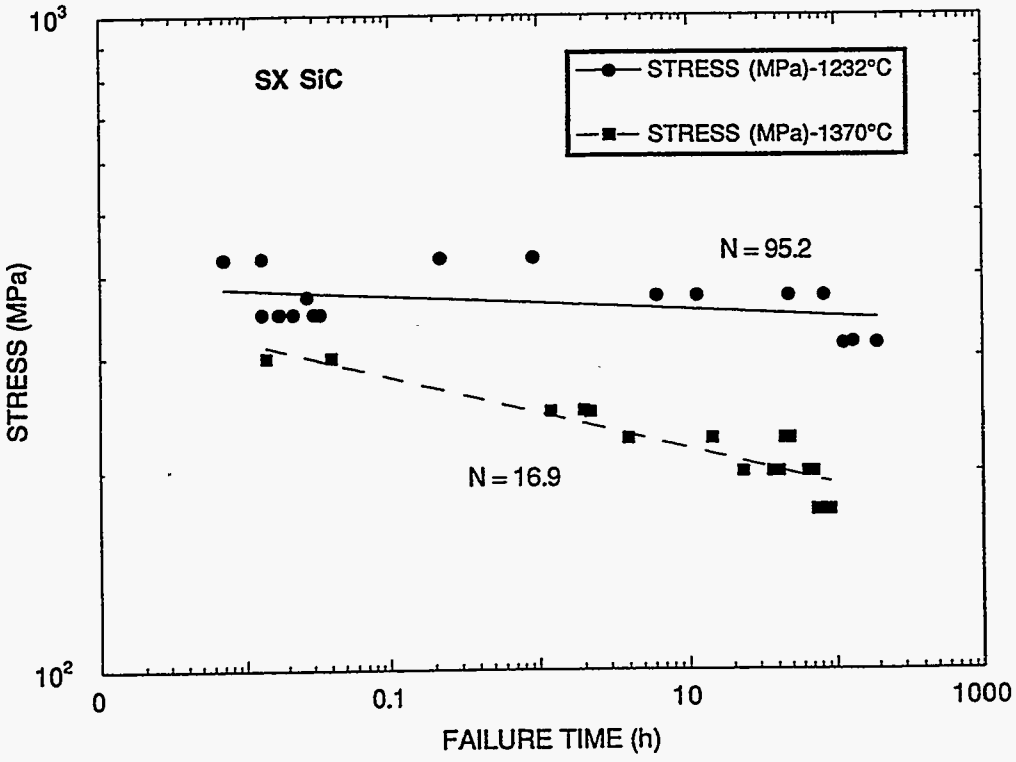

Figure 16. Stress Rupture Response for SX-G1.

yttrium aluminate with the SiO_2 layer which forms on SiC due to oxidation at elevated temperatures. The slow crack growth can occur as the reaction progresses from the tensile surface to the bulk of the sample. The failure origins for such samples always occur from the tensile surface when tested in air. Moreover, dynamic fatigue experiments conducted at 1400°C in argon indicated no glass phase formation and hence no slow crack growth was observed at 1400°C[5]. Therefore, the mechanism of slow crack growth can be classified as "Environmentally Induced Slow Crack Growth." Since a chemical reaction at the surface is involved which promotes slow crack growth, it can also be called "Stress Corrosion Cracking."

Creep: Creep tests were carried out in uniaxial tension at 1260, 1370, and 1450°C with stresses varying from 50 to 300 MPa. The creep strain vs time plots at these temperatures are shown in Figures 17-19. Very little or negligible deflection was recorded at 1260°C at stresses varying between 200 and 250 MPa. At 1370 and 1450°C measurable steady-state strain rates were obtained. However, samples failed in relatively shorter time periods at temperatures of 1370 and 1450°C. As seen from the creep plots, no tertiary creep was observed before failure. The fracture surface of the sample failed at 1370°C under a stress of 150 MPa is shown in Figure 20. A clear oxidation layer and a slow crack growth area were observed. The oxidation layer was large and the severity of slow crack growth was greater at 1450°C as seen in Figure 21. The oxidation layer and the fracture initiation sites appear "glassy" as seen in Figures 20 and 21.

A transmission electron microscopic (TEM) investigation was conducted on samples that failed in a creep test as well as on a pristine sintered sample. Samples were prepared from surfaces parallel and perpendicular to the tensile stress axis. Typical TEM micrographs from longitudinal and transverse sections of the sample subjected to creep are shown in Figures 22 and 23, respectively. A microstructure from a pristine sample is shown in Figure 24 for comparison. No cracking along grain boundaries and cavitation at triple junctions could be seen from the material subjected to creep. The microstructure appears to be unchanged compared to the pristine sample. It appears that microstructural damage due to creep has not occurred.

Appearance of an oxidation layer and slow crack growth regime associated with a glassy fracture origin from the surface of samples subjected to tensile creep suggest that the failure is attributed to slow crack growth due to glass phase formation by oxidation, rather than creep induced failure.

However, samples tested for creep at 1260°C did not show any observable creep strain at the various stress levels and did not fail even after a cumulative time of 160 hours. One of those samples was then loaded to failure. The sample failed because of a volume flaw as shown in Figure 25 at a stress of 379 MPa. This flaw was similar to those observed in tensile samples and

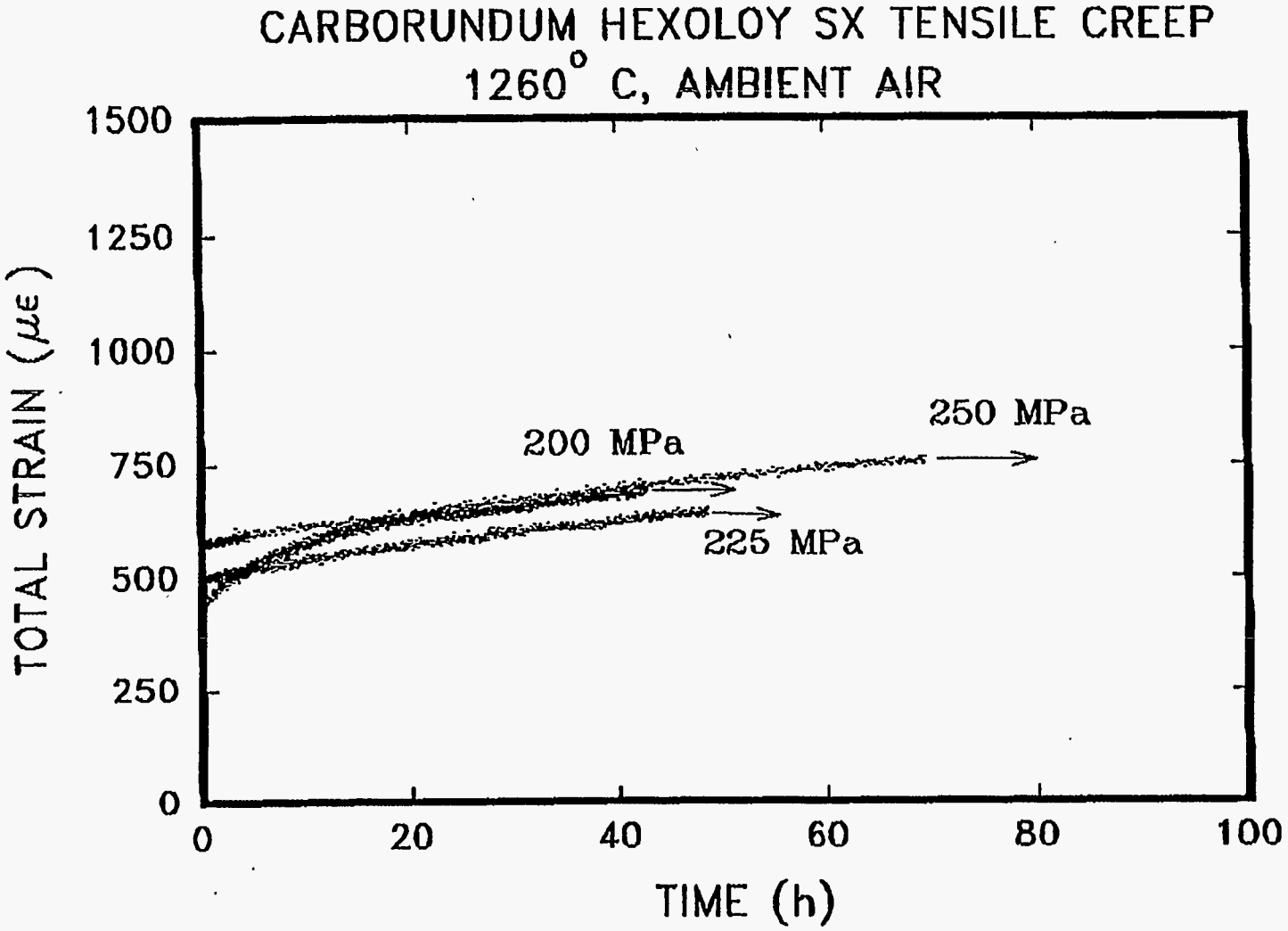

Figure 17. Creep Strain vs Time at 1260°C.

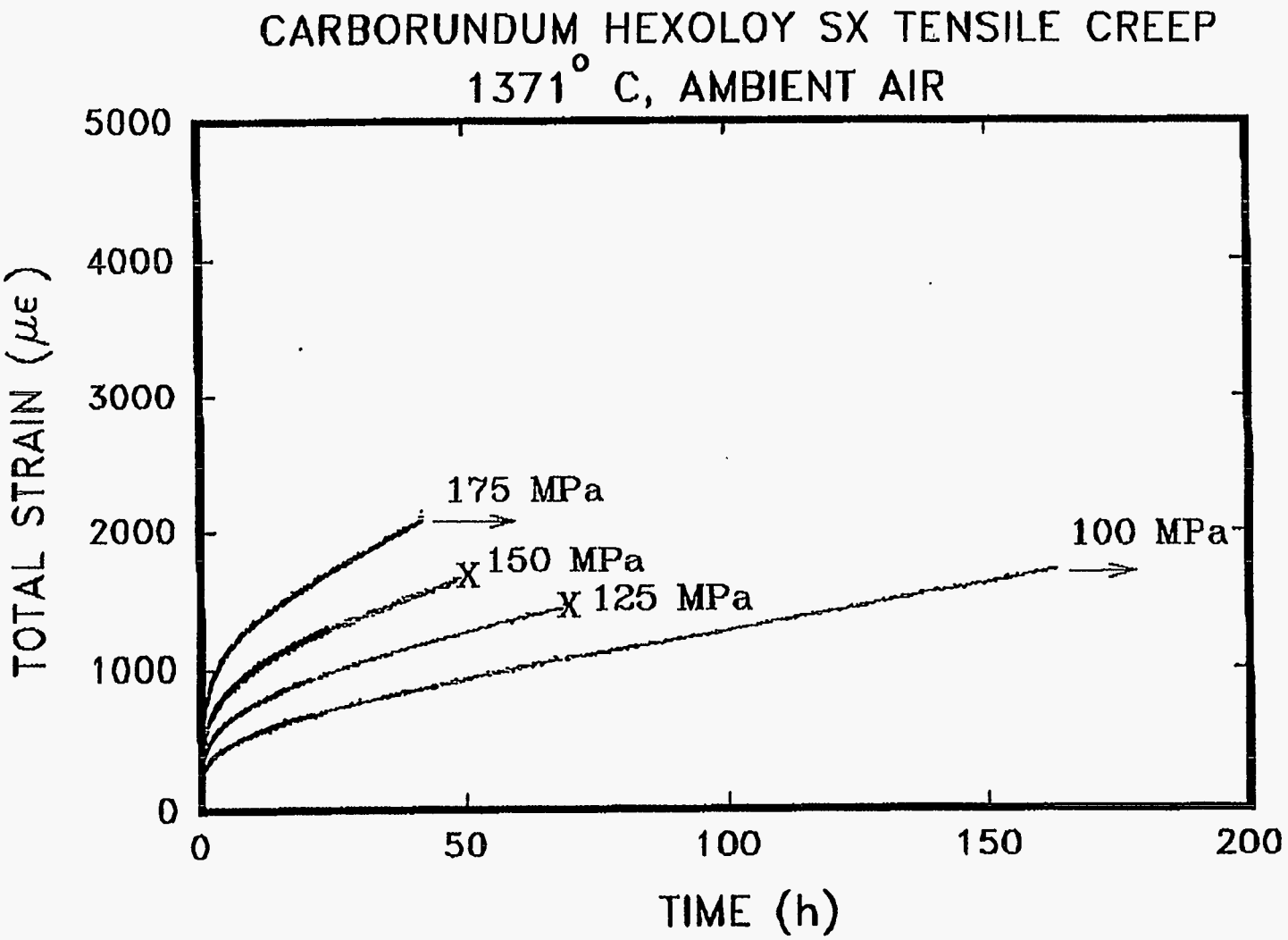

Figure 18. Creep Strain vs Time at 1370°C.

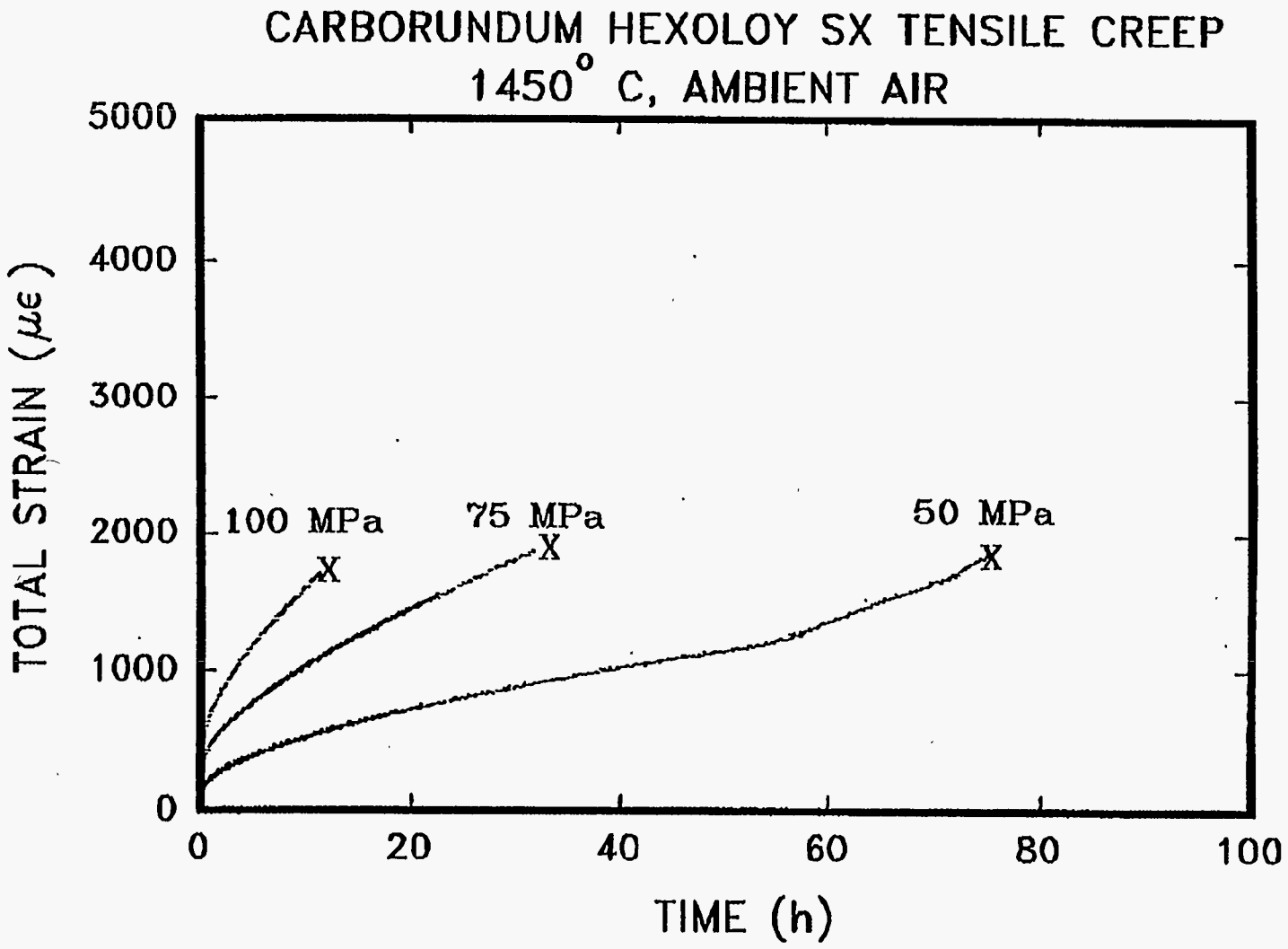

Figure 19. Creep Strain vs Time at 1450°C.

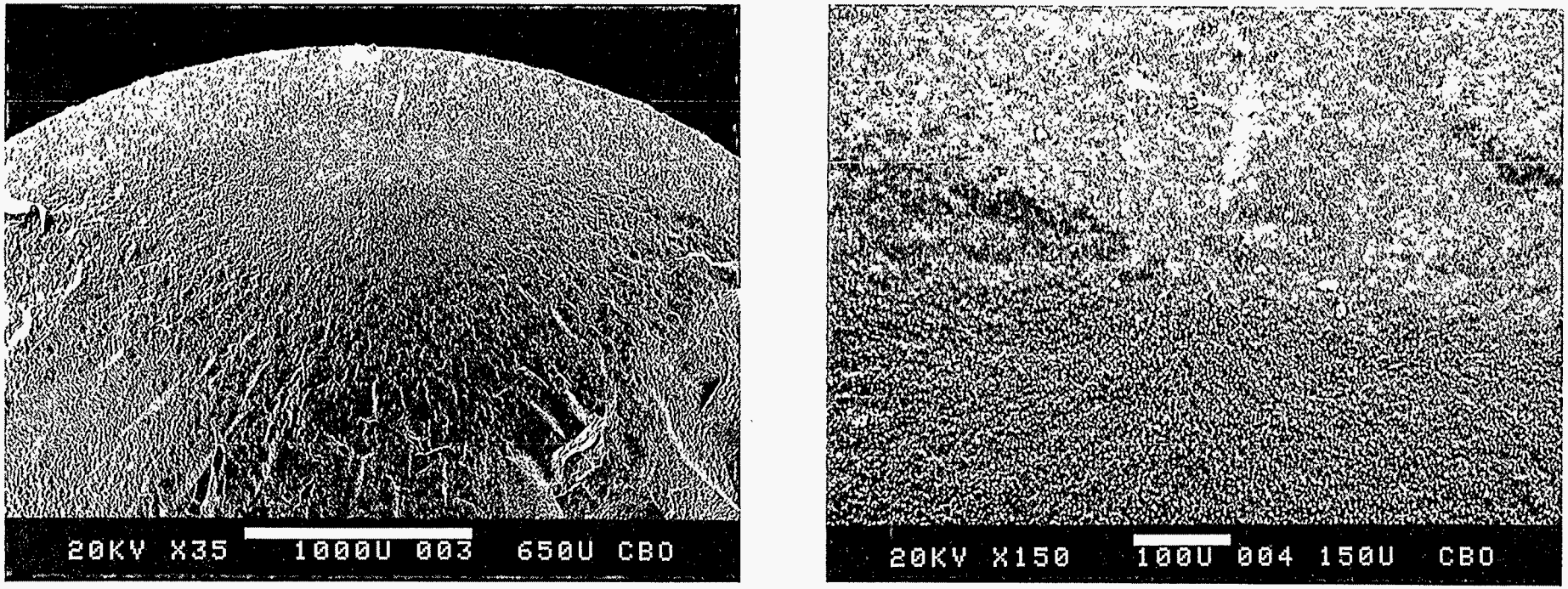

Figure 20. Fracture Origin in a Tensile Sample Failed at 1370°C at a Stress of 150 MPa for 49 Hours.

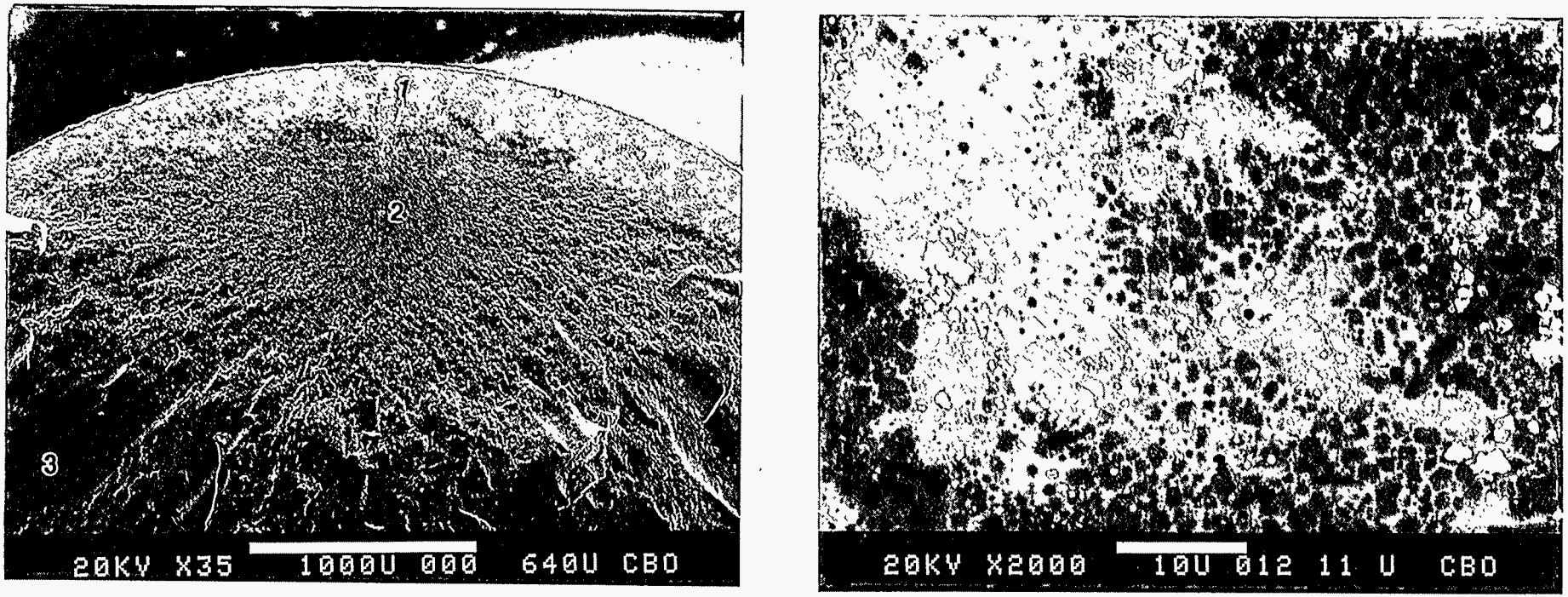

Figure 21. Fracture Origin in Tensile Sample Failed at 1450°C at a Stress of 100 MPa for 12 Hours.

Figure 22. Transmission Electron Micrograph from Longitudinal Section.

Figure 23. Transmission Electron Micrograph from Transverse Section.

Figure 24. Transmission Electron Micrograph from a Pristine Sample.

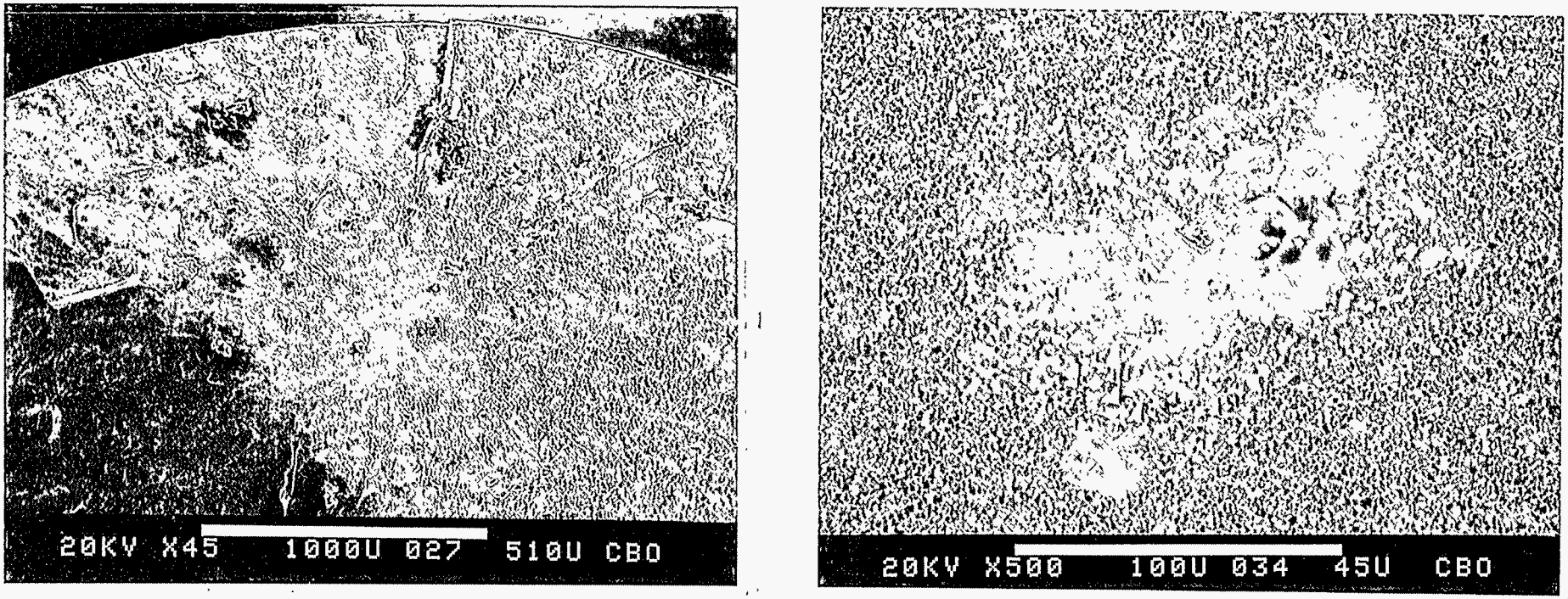

Figure 25. Fracture Surface of a Sample Loaded to Failure at 1260°C. Tensile Strength = 379 MPa. Sample was Stressed up to 250 MPa for >150 Hours at 1260°C before Loading to Failure.

MOR bars tested at 1232°C. The oxidation layer and the glass phase were not observed in this sample. This observation was another verification that slow crack growth due to glass phase formation does not occur up to a temperature of at least 1260°C. The slow crack growth becomes pronounced only at temperatures above 1300°C since the glass phase melting temperature is about 1300°C.

Figure 26 shows a plot of steady-state strain rate vs applied stress at 1370 and 1450°C. It can be observed that the steady-state strain rates were between 1 x 10^{-9} and 2 x 10^{-8}/sec. A multiple line regression analysis was used to determine the creep parameters from the generalized creep relation

$$\dot{\varepsilon} = A \sigma^{n} \exp(-Q/RT)$$

where $\dot{\varepsilon}$ is the steady-state strain rate, A is a constant, σ is the stress, "n" is the stress exponent, Q is the activation energy, R is the gas constant, and T is the absolute temperature. The values of "n" and "Q" were estimated as 2.4 and 720 kJ/mol, respectively. These creep parameters do not agree with the parameters determined for monolithic sintered SiC materials[6]. The mechanism of creep in the temperature and stress regime explored, if any, was not obvious in this material.

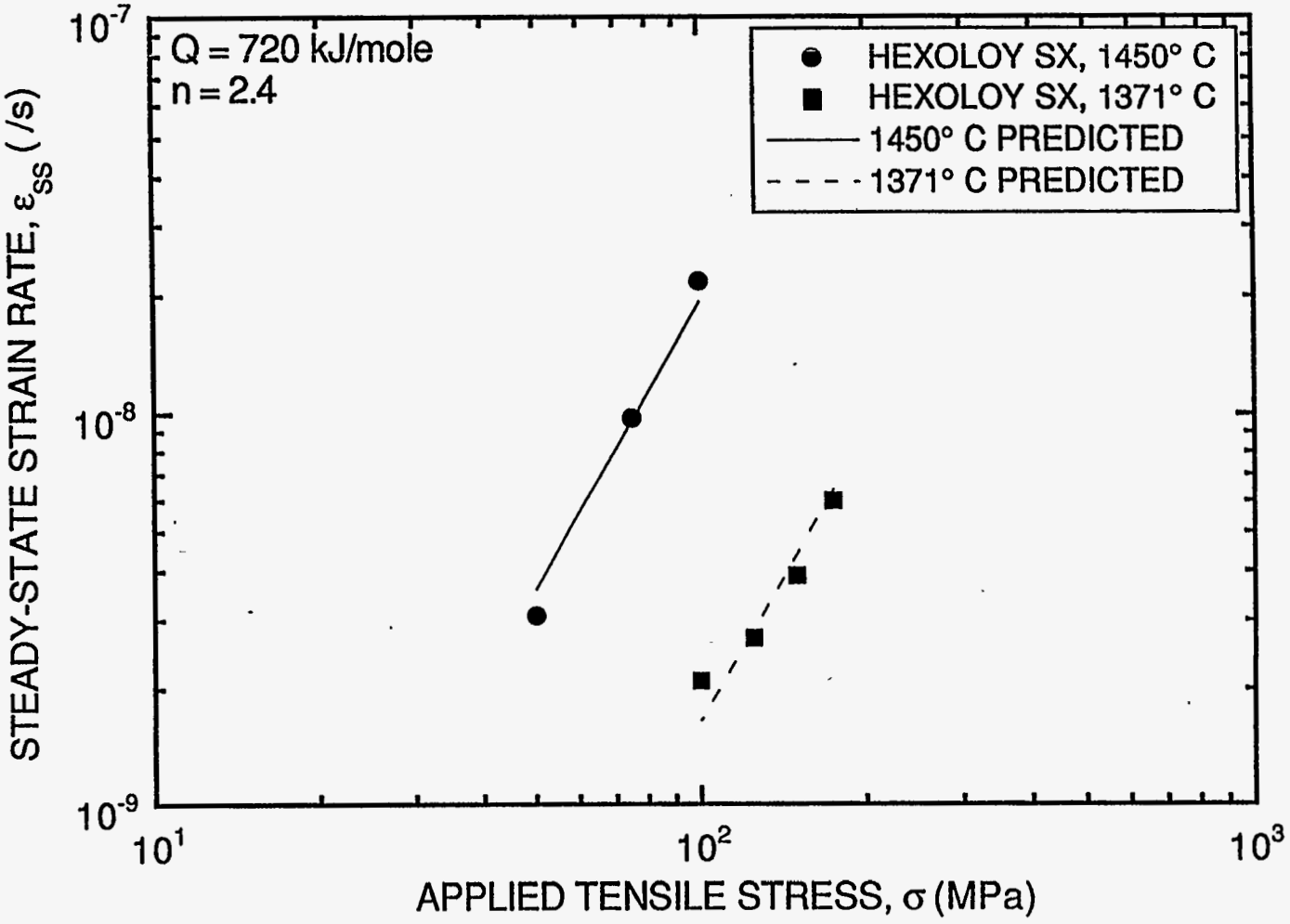

Figure 26. Steady-State Strain Rate vs Applied Stress at 1370 and 1450°C.

Task 2: SiC Powder Selection.

In this task the effect of starting SiC powder characteristics on densification and mechanical properties was explored. Powder availability and cost were also considered.

Experimental Procedure

Six different sources of commercially available SiC powder were chosen for this study. The greenforming process and densification conditions were the same for all powders. The powders were characterized for particle size distribution and surface area. The six different SiC powders were Sweco™ milled with sintering aid additives and spray dried into soft flowable agglomerates. The powders were isostatically pressed into 65-mm-square plates. The plates were pressureless sintered and later post-treated at a slightly higher temperature and pressure. Densities were measured by immersion techniques. The densified plate samples were machined into MIL-STD-1942 "B" (3 x 4 x 45 mm) MOR bars. Chevron notches were cut in the bars for fracture toughness (K_{Ic}) evaluation. Mechanical characterization included flexural strength and K_{Ic} at room temperature and dynamic fatigue at 1370°C. About 20 samples were used for the flexural strength evaluation and five Chevron-notched samples were used for the K_{Ic} evaluation of each powder. Six samples were tested per stress rate at three stress rates differing by an order of magnitude for the slow crack growth parameter evaluation (dynamic fatigue). Fractographic investigation was also conducted to establish the flaw population.

Results

The powder characteristics and the densities obtained after pressureless sintering and post-treating are listed in Table 2. The median particle size for all the SiC powders is approximately the same. However, noticeable differences are observed in the particle size distribution and surface area of the powders, leading to differences in densification. The as-sintered and post-treated densities as a function of cumulative percentage of particles <1 μm in the distribution are shown in Figure 27. The densities as a function of surface area are shown in Figure 28. It appears from Figures 27 and 28 that there is a sharp cut-off limit in the particle size distribution and surface area beyond which densification is facilitated.

The results of the mechanical property evaluation for different powder sources are listed in Table 3. Mechanical property evaluation from materials fabricated from SiC powder "E" was not attempted due to inadequate densification. The fracture toughness (K_{Ic}) evaluated from Chevron-notched bars fabricated from different SiC powder sources are not significantly different.

TABLE 2

Sintering Characteristics of Various SiC Powders

SiC Powder Source	Surface Area sq.m/gm	Median Particle Size μm	% Particles ≤1 μm	% Theoretical Density	
				Pressureless Sintered	Post Treatment
A	17.1	0.45	99.3	96.0	99.8
B	9.3	0.45	78.1	91.3	95.2
C	16.3	0.44	92.2	94.0	99.0
D	11.7	0.43	82.0	91.3	95.8
E	8.2	0.48	70.9	78.5	86.4
F	32.0	0.44	99.9	97.0	99.8

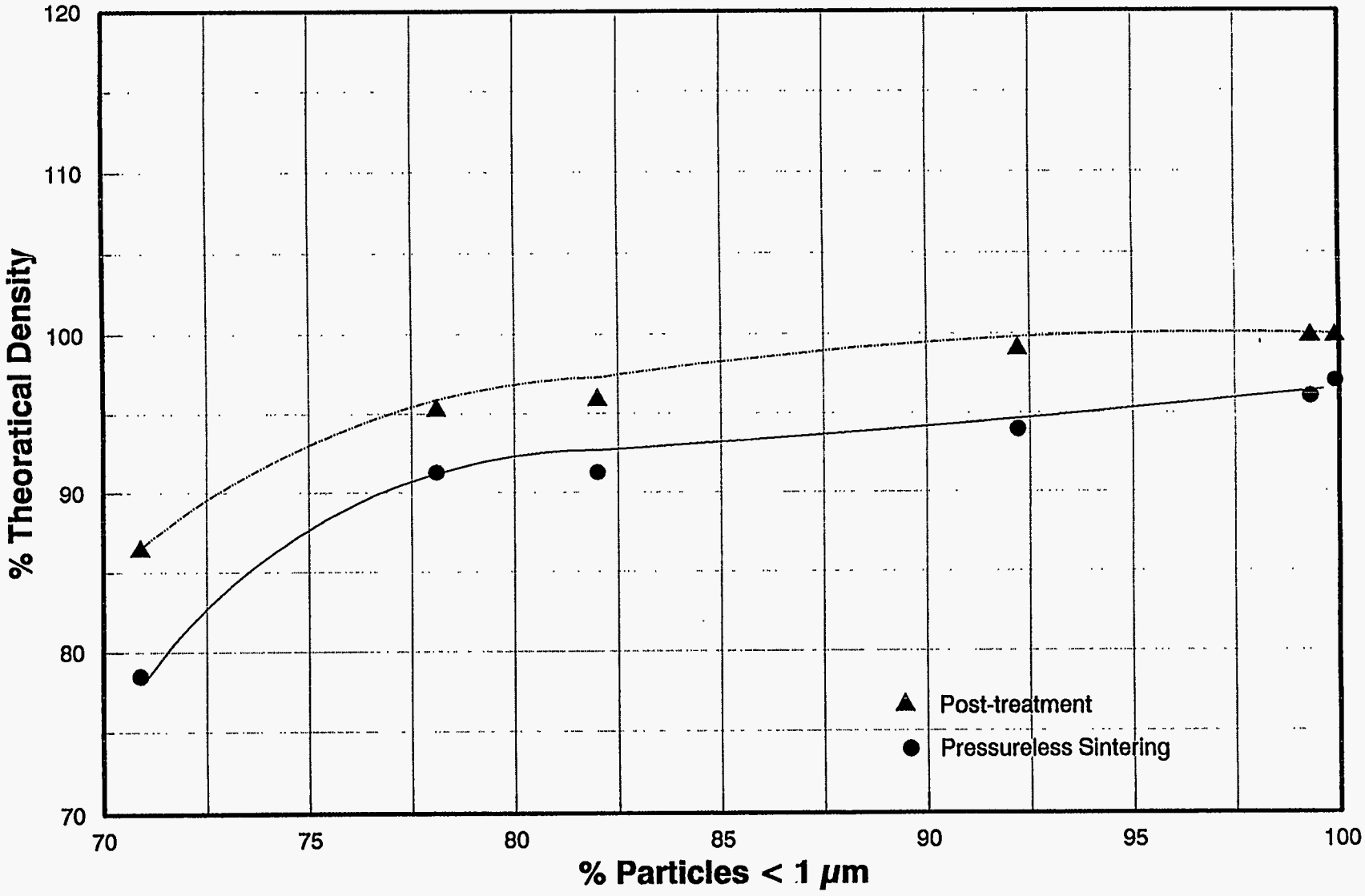

Figure 27. The As-Sintered and Post-Treated Densitites as a Function of % Particles <1 μm.

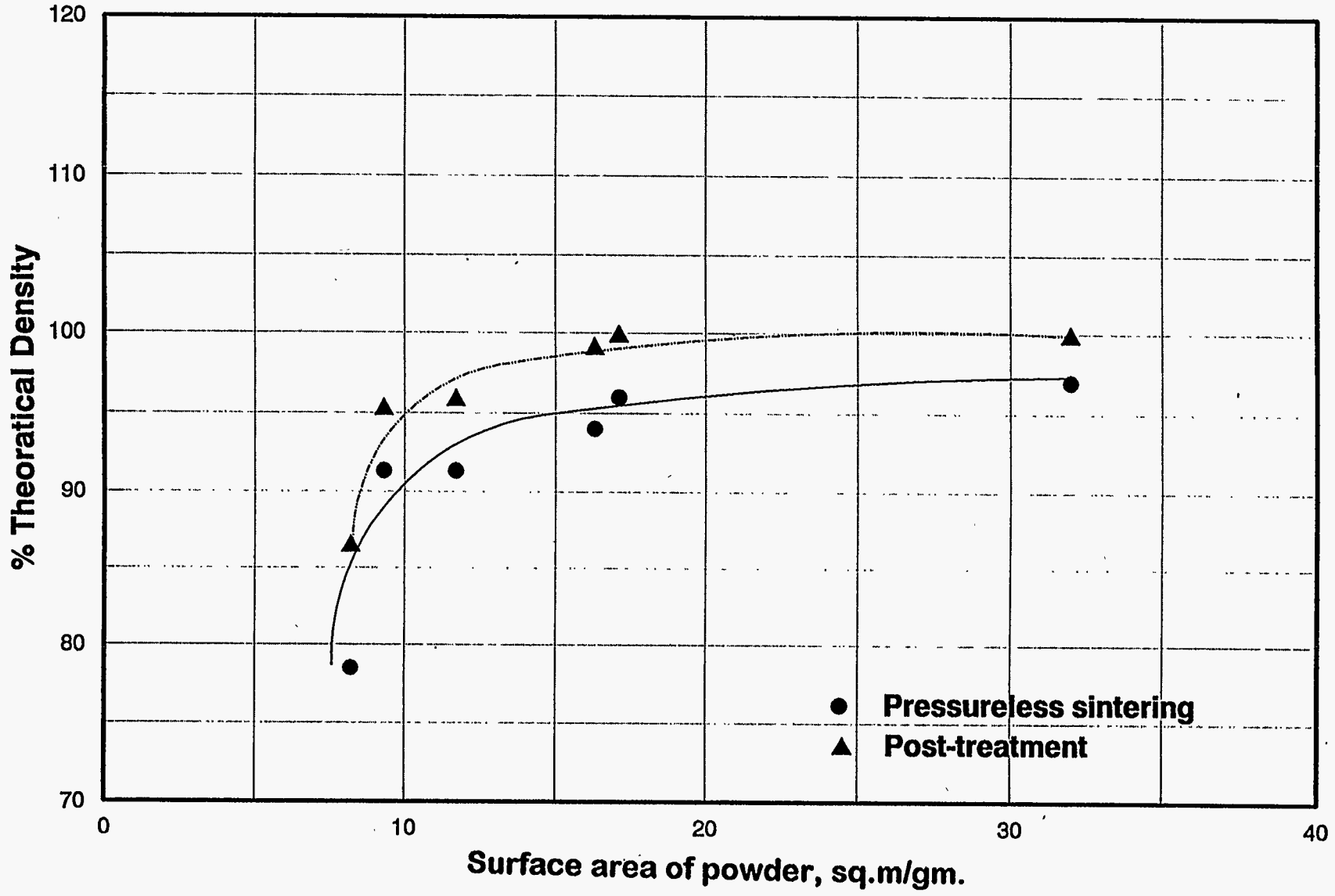

Figure 28. The As-Sintered and Post-Treated Densities as a Funcion of Surface Area of SiC Powders.

TABLE 3

Mechanical Properties of SX-G1 Material Fabricated with Various SiC Powders

	Room Temperature				1371°C			
Powder Source	Toughness $MPa.m^{1/2}$	MOR MPa	Weibull Modulus	Flaw	MOR MPa	Weibull Modulus	Flaw	SCG* Parameter
A	4.05	779	5.3	Volume, Pools	420	10.3	SCG	14.5
B	4.43	627	4.5	Volume, Pools	365	12.6	SCG	35.0
C	3.77	724	2.6	Volume, Agglm. Surface, Mach.	352	2.9	SCG	17.3
D	3.87	796	6.3	Volume, Voids	269	7.2	SCG	9.0
E	-	-	-	-	-	-	-	-
F	4.25	648	4.0	Volume, Pools	338	15.4	SCG	63.0

*SCG - slow crack growth

However, the average room-temperature flexure strengths varied from 496 to 779 MPa. In almost all cases, the strength-limiting defects were identified to be "pools" of second phase clusters. The origination of such "pools" was related to the reaction of SiC with the second phase and was reported in detail in Task 1. The flexural strength at 1370°C appeared to be proportional to flexural strength at room temperature. Slow crack growth accompanied by glass phase formation has been determined to be the failure mechanism at 1370°C for samples fabricated from each of the powder sources. The slow crack growth mechanism was explained under Task 1. Though the slow crack growth parameter appeared to be high for powders "B" and "F," the strength levels were low. Actually, significant slow crack growth occurred even at higher stress rates for these materials as evidenced from optical fractography.

Samples fabricated from SiC powder source "A" yielded the best properties in terms of densification behavior and flexural strengths at room temperature and 1232°C. The other responses such as K_{Ic} and slow crack growth exhibited only minor variations. Moreover, the SiC powder from source "A" was commercially available and the cost was the second lowest among the six powders considered. Based on these considerations, SiC powder from source "A" was selected for further optimization studies.

Task 3: Development of an Improved Dispersion Process.

The objective of this task was to improve the mixing and dispersion of the sintering aids. Three approaches were explored and are discussed below.

Experimental Procedure and Results

The first approach was to use chemical precursor sintering aids such as soluble nitrides and butoxides, rather than ceramic powders. Three mixes were prepared with additives from the chemical precursors. The total additive levels were 2 wt %, 2.5 wt %, and 3.0 wt %. Sintered densities of 70%, 88%, and 81% T.D., respectively, were obtained. These values were considerably lower than those with the control mix (95.5% T.D.). Chemical analysis for these three mixes showed relatively high free carbon contents (1.20 wt %, 1.15 wt %, and 1.12 wt %). This was believed to cause the low sintered density.

The second approach focused on improving premix uniformity by intensive grinding of sintering additives. Using a lab-size attrition mill, the sintering additives were ground to a median size of 0.50 to 0.55 μm from an as-received size of between 3.0 and 4.9 μm. With these ground sintering additives, three mixes were prepared with total additives of 2 wt %, 2.5 wt %, and 3.0 wt %.

Densities of 72%, 90%, and 93% T.D., respectively, were obtained for these three mixes, again lower than the control mix.

High-energy turbomilling of the sintering-aid powder was the third technique investigated. Professor Dale Wittmer of Southern Illinois University was retained as a consultant for this task. Using the turbomill in his laboratory, high-surface-area mixes (up to 22.8 m^2/gm) were obtained. More significantly, green densities as high as 66% were achieved. These samples were sintered and post-treated to high densities, with the exception of the samples from mix WR5 (Table 4) which were milled with sintered ZrO_2 (Y_2O_3) grinding media. It was suspected that contamination from ZrO_2 (Y_2O_3) media wear was responsible for the poor sintering in that sample. It should also be noted that all the green samples were prepared by a filter-pressing technique without the use of a binder. As a result the samples showed numerous cracks after sintering.

Because of this cracking problem, only a limited number of samples were obtained for MOR evaluation. These results are listed in Table 4. It can be seen that on the average turbomilled SX samples showed higher MOR strengths than those of the traditionally prepared samples. A high single-sample value of 1,227 MPa was obtained. To eliminate the cracking problem, a sixth turbomilled batch duplicating mix WR4 was prepared. Instead of using the filter-cake approach with no binder, spray drying with an added binder was used. With the later technique, the cracking problem was resolved. The average strength of the samples fabricated from spray drying was 903 MPa, with a highest single value of 1,172 MPa. Nevertheless, the sinterability was not consistent. A second plate processed the same way was found to exhibit a low density (<95% T.D.). However, based on the exceptional mechanical properties potentially achievable, turbomilling was chosen as the processing technique to be used for the next task.

TABLE 4

Turbomilling Results

	Milled Time (hour)	Grind Media	Surface Area m^2/g	Postsinter Density g/cc	MOR R.T. MPa	No. of Samples
WR1	2	SiC	22.0	3.224	804	2
WR2	2	SiC	20.5	cracked	NA	
WR3	1	SiC	18.6	3.226	969	3
WR4	4	SiC	22.8	3.226	1231	1
WR5	0.5	$Y\text{-}ZrO_2$	NM*	cannot be densified	NM	
WR6	4	SiC	NM	3.220	903**	4
SX-G1+	NA***	SiC	16.0	3.220	779	20

*Not measured

**Average strength 903 MPa, highest individual strength is 1,172 MPa

***Not applied

+Conventional process

Task 4: Optimize the Current SX-G1 SiC Material Through an Experimental Design Methodology.

Experimental Procedure

The objective of Task 4 was to optimize SX-G1 properties using a designed experiment approach by varying the three most influential processing parameters, namely, the sintering temperature, post-treatment temperature, and post-treatment pressure. The upper and lower limits for each of these factors are listed below:

Sinter Temperature: ST1 and ST2

Post-Treat Temperature: PT1 and PT2

Post-Treat Pressure: P1 and P2

A main factor plus interaction model was used to design this series of experiments[7]. The upper and lower limits of these three factors form a design experiment cube. Totally, there were ten sets of experimental conditions, with eight at the corners of the cube and two at the center of the cube as shown in Figure 29. The responses used to monitor these experiments included MOR and toughness at room temperature and 1232°C, and dynamic fatigue at 1232°C.

Results

Five powder mixes were prepared by turbomilling at Southern Illinois University using a new batch of SiC grit as grinding media. Sintered densities for these five mixes were between 90 and 94% T.D., as compared to >95% for the earlier turbomilled samples. After post-treatment the densities were in the range of 92 to 97%; however, they were still below full density. To understand the reason for this low density, particle size analyses for these five mixes were conducted. Larger particles (>1 mm) were detected with a Horiba analyzer. BET surface area measurements of these turbomilled mixes were between 16.0 and 17.2 m^2/g as compared to >20 m^2/g obtained on earlier turbomilled samples. Both results indicated the presence of large particles which are believed to be from the wear of SiC grit grinding media. A review of powder processing data also confirmed that there was about 250 grams of media wear from the grinding grit for each 1,500 gm batch mix. This excessive wear of grinding media was expected to have a significant effect on retarding the sintering kinetics.

The fracture surfaces of the Task 3 turbomilled sintered samples were also reviewed in light of this information. In one set of samples that densified poorly, excess grit concentration was observed on both the etched microstructure and the fracture surfaces. In another set that

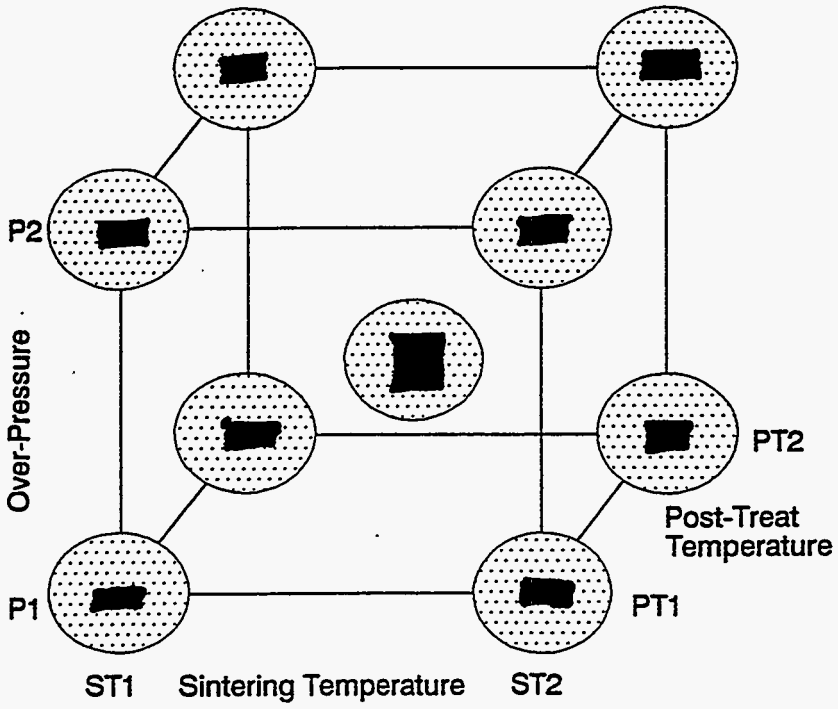

Figure 29. A Schematic of the Experimental Design Matrix.

achieved high densities and exhibited strengths in excess of 896 MPa (as high as 1,227 MPa), the fracture analysis revealed no obvious fracture origin. In other plates that also densified well but with somewhat lower strengths (ranging from 620 to 896 MPa), occasional grits were found and were determined to be the strength-limiting defects.

These results reveal that although turbomilling had shown the potential of achieving exceptionally high mechanical properties, processing reproducibility was a major issue. It was concluded that more effort was required to overcome this problem. It was therefore decided to utilize the traditional milling process as in Task 1 to complete the remaining Task 3 work.

A new SX-G1 mix was then prepared. All of the ten experiments were conducted successfully. The final densities obtained were between 97.7% and 100.0% T.D. with six out of ten conditions showing densities over 99.3% T.D. The appropriate mechanical testing and microstructural characterization were then conducted.

The mechanical properties determined at different combinations of the densification conditions are listed in Table 5. The upper limit of each processing parameter is denoted as +1 and the lower limit as -1. The toughness (K_{Ic}) does not change appreciably as a function of densification parameters. However, the average flexure strength varies from 580 MPa (84 ksi) to 965 MPa (140 ksi) at room temperature.

Two types of strength limiting defects at room temperature were observed. One was a volume defect containing "pools" of second phase reaction product clusters. The other type of defect was surface defects induced by machining. Samples failing from the machining defects have a relatively high strength in excess of about 800 MPa (116 ksi). The flexure strength varied anywhere from about 345 MPa (50 ksi) to 900 MPa (130 ksi) for the volume defects, depending upon the location and size of the flaw. A typical volume defect (i.e., "pool") is shown in Figure 30, while a machining-induced surface failure is shown in Figure 31. Table 3 also lists the fraction of failures due to "pools." It can be seen that a higher mean flexure strength corresponds to fewer "pools" observed.

The strength at 1232°C appears to be directly related to the strength at room temperature. Moreover, no slow crack growth was evidenced at 1232°C since no strength drop at lower stress rates was observed. This observation is consistent with the earlier results observed in Task 1. Hence, the room-temperature flexural strength (MOR) was considered to be the primary response for different densification conditions. The room-temperature flexure strength response is shown schematically in Figure 32 corresponding to different densification conditions explored in the

TABLE 5

Density and Mechanical Properties Determined at Specific Densification Conditions in the Experimental Design Matrix

				MOR, ksi		Fraction of "Pools"	Chevron Notch Toughness MPA $\sqrt{m}$	
Sin. Temp	PT* Temp	PT Pressure	PT Density %	RT	1232 °C	RT	RT	SCG** Parameter N
0	0	0	99.9	128	76	0.53	4.9	No SCG
+1	−1	+1	99.7	84	60	0.80	5.2	No SCG
−1	+1	−1	99.5	103	66	0.40	5.4	No SCG
+1	+1	+1	100.0	124	60	0.53	4.9	No SCG
−1	+1	+1	99.9	140	76	0.27	4.7	No SCG
0	0	0	99.9	115	72	0.53	4.9	No SCG
−1	−1	+1	99.3	105	62	0.33	4.4	No SCG
+1	−1	−1	98.0	79	54	0.66	4.6	No SCG
−1	−1	−1	97.7	80	57	0.33	4.3	No SCG
+1	+1	−1	98.2	84	58	0.73	5.5	No SCG

* **Post Treatment**

** **Slow Crack Growth - No SCG means N is approaching infinity.**

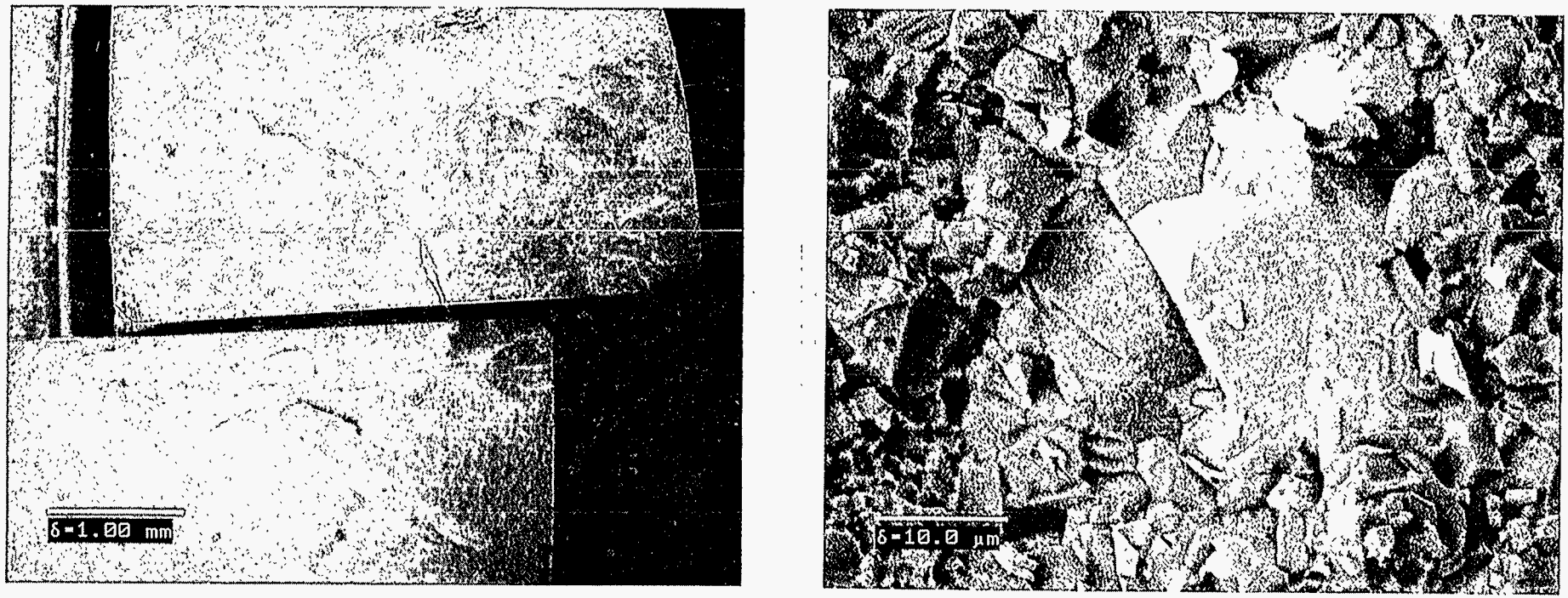

Figure 30. "Silicon-Rich Pool" as Strength-Limiting Volume Defect.
Flexural Strength = 1,020 MPa (148 ksi).

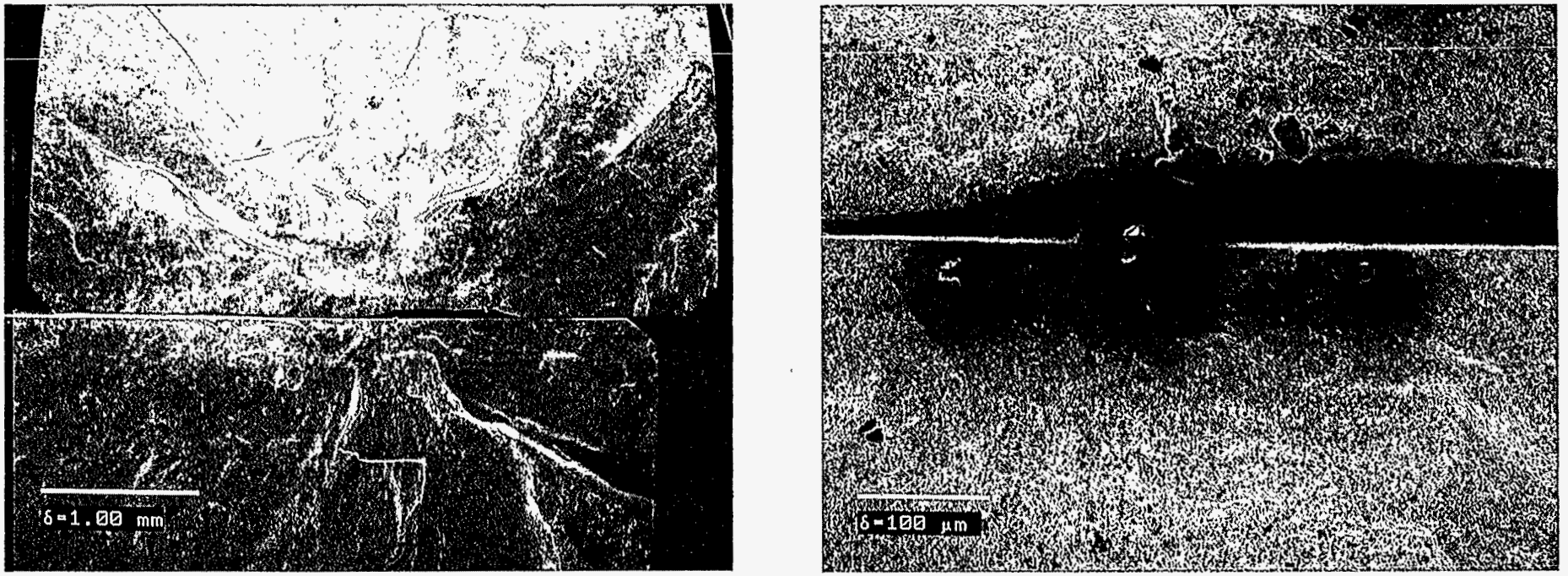

Figure 31. Machining Induced Surface Defect as the Fracture Origin.
Flexural Strength = 938 MPa (136 ksi).

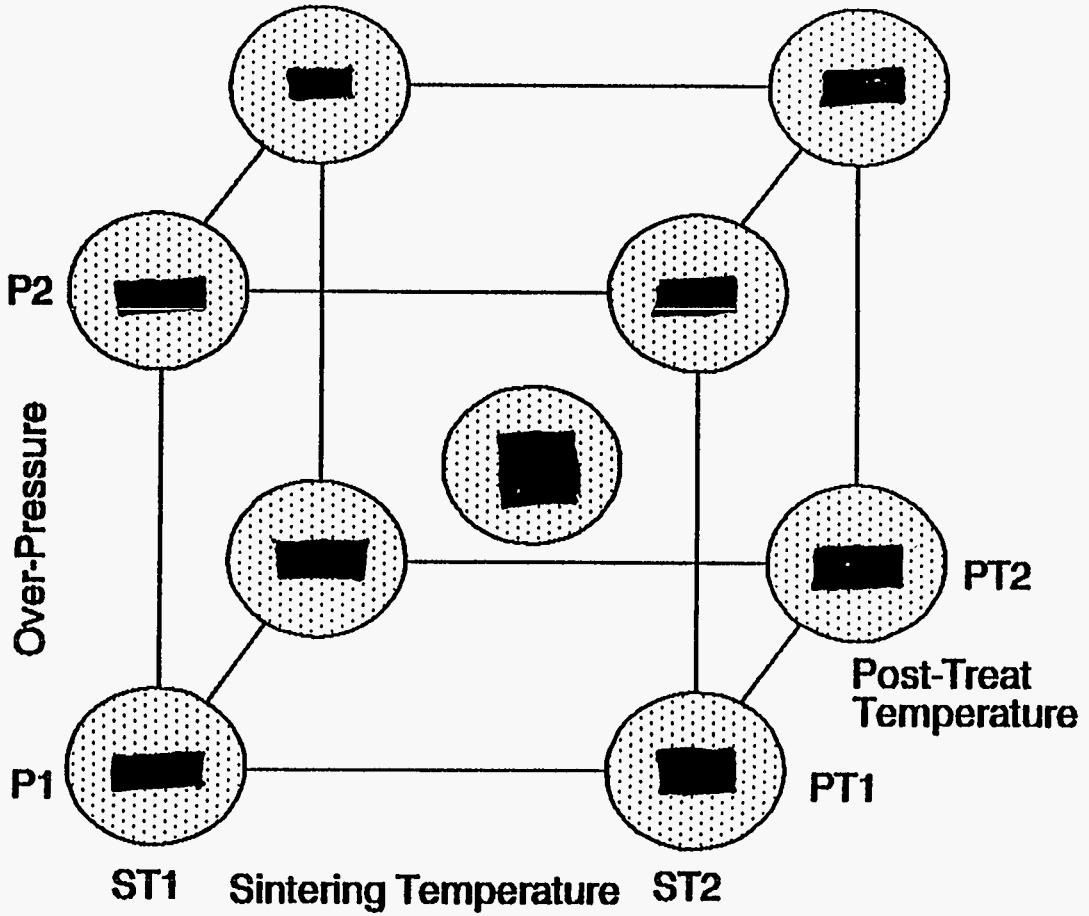

Figure 32. Room-Temperature Flexural Strength Corresponding to Various Densification Conditions in the Experimental Matrix.

statistical process design. The maximum room-temperature average flexural strength of 965 MPa (140 ksi) was observed for the densification conditions corresponding to the lowest sintering temperature, and higher post-treatment temperature and post-treatment pressure. Contour maps are shown in Figures 33, 34, and 35, illustrating the variation of strength with densification parameters.

It is interesting to note that a higher strength was observed with lower sintering temperatures and also corresponded to fewer volume flaws or "pools." The "pool" of second phase clusters appeared to form from the reaction of SiC with yttrium aluminate as suggested by Omori et al.[4], and from the SAM studies in Task 1. It is believed that at lower processing temperatures the extent of reaction was not as severe and thus a lesser number of smaller "pools" were formed.

It was clearly illustrated from the Task 4 experimental results that for the same corresponding post-treatment temperature and pressure, a lower sintering temperature leads to fewer "pools" and higher strength suggesting that sintering temperature was primarily responsible for the "pool" formation. The reaction between the Y-Al-O second phase and SiC matrix also involves the release of gaseous species such as CO and or CO_2. Under higher pressure the reactions would be thermodynamically less favorable. Hence, at higher pressures the reaction may be inhibited.

It was observed from the statistically designed experiments that the highest flexure strength obtained at room temperature was 965 MPa (140 ksi). This value was more than 20% higher than that obtained in the earlier Task 1 studies of 780 MPa (113 ksi). Also, fewer failures were due to volume defects and more failures were from the surface due to machining-induced defects. The higher fraction of surface defects will result in better reliability. Contour plots generated suggested that lower sintering temperatures combined with higher post-treatment temperatures and pressures would lead to improved flexure strengths. In fact, the optimum post-treatment conditions might fall outside the experimental cube indicating that the mechanical properties might be further improved with additional experimental work.

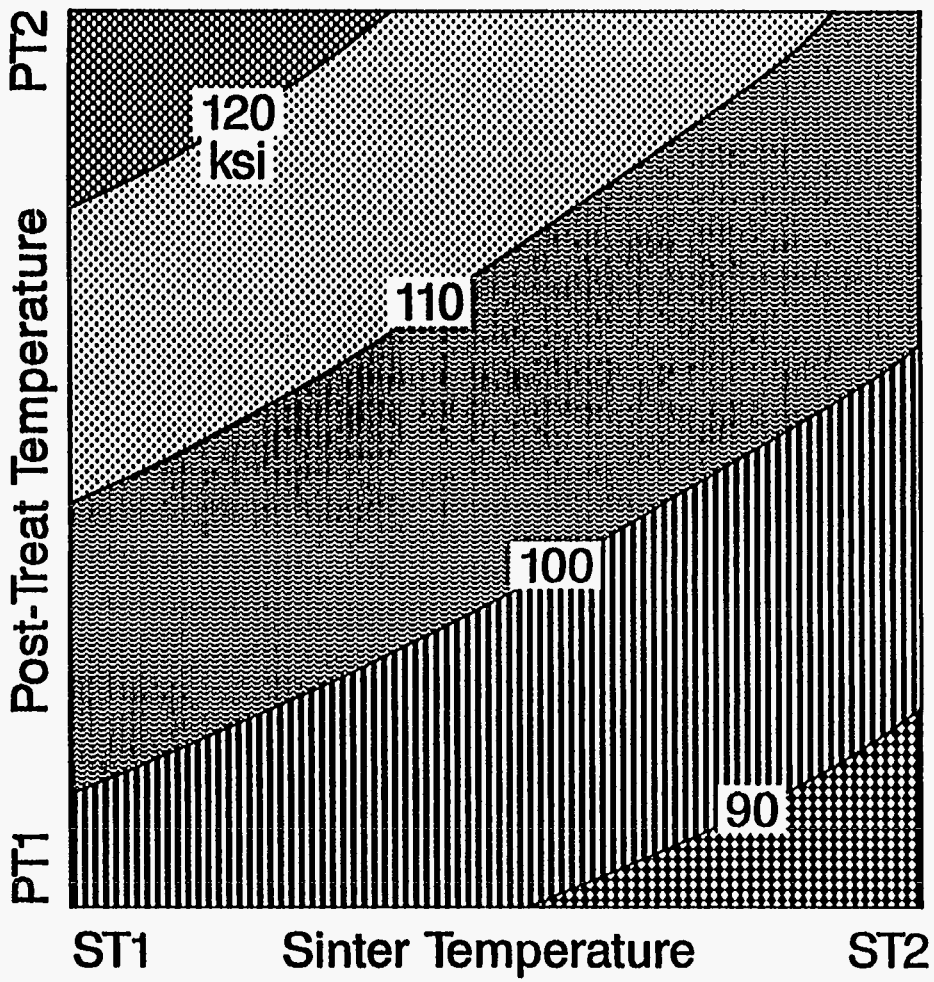

Figure 33. Contour Map of Room-Temperature Flexural Strength as a Function of Sintering Temperature and Post-Treatment Temperature

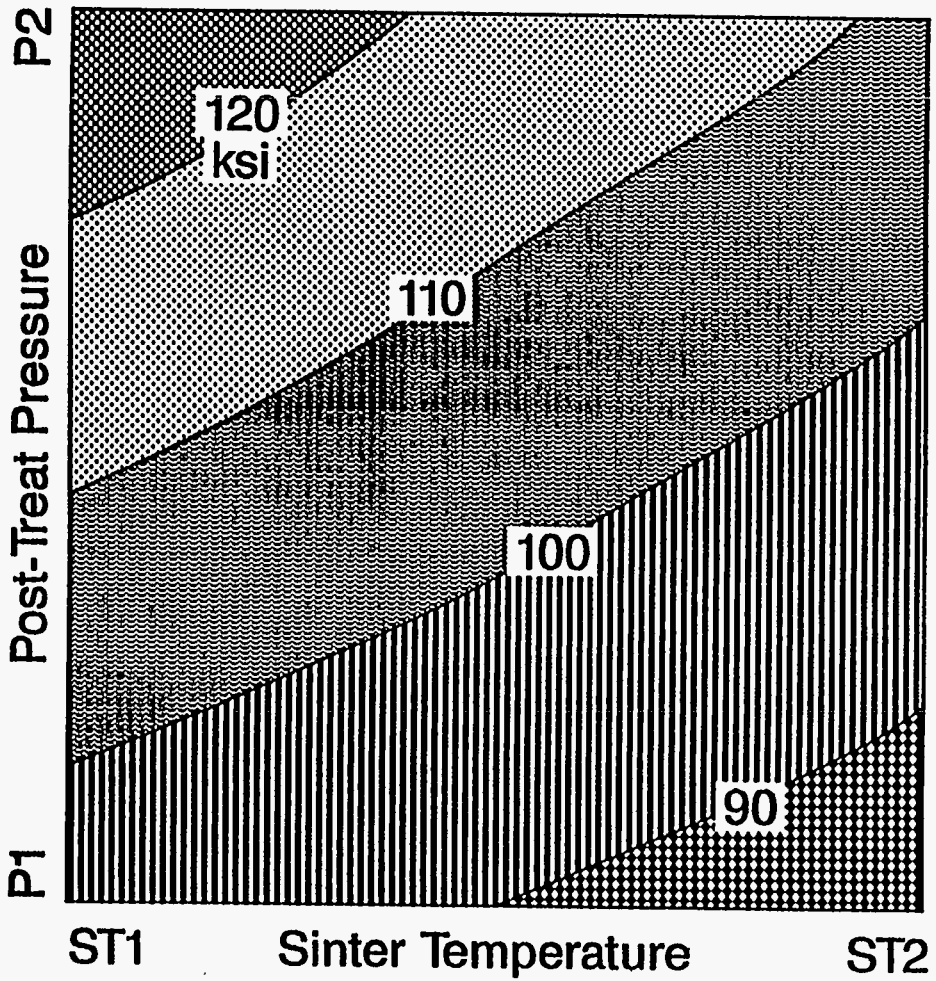

Figure 34. Contour Map of Room-Temperature Flexural Strength as a Function of Sintering Temperature and Post-Treatment Pressure.

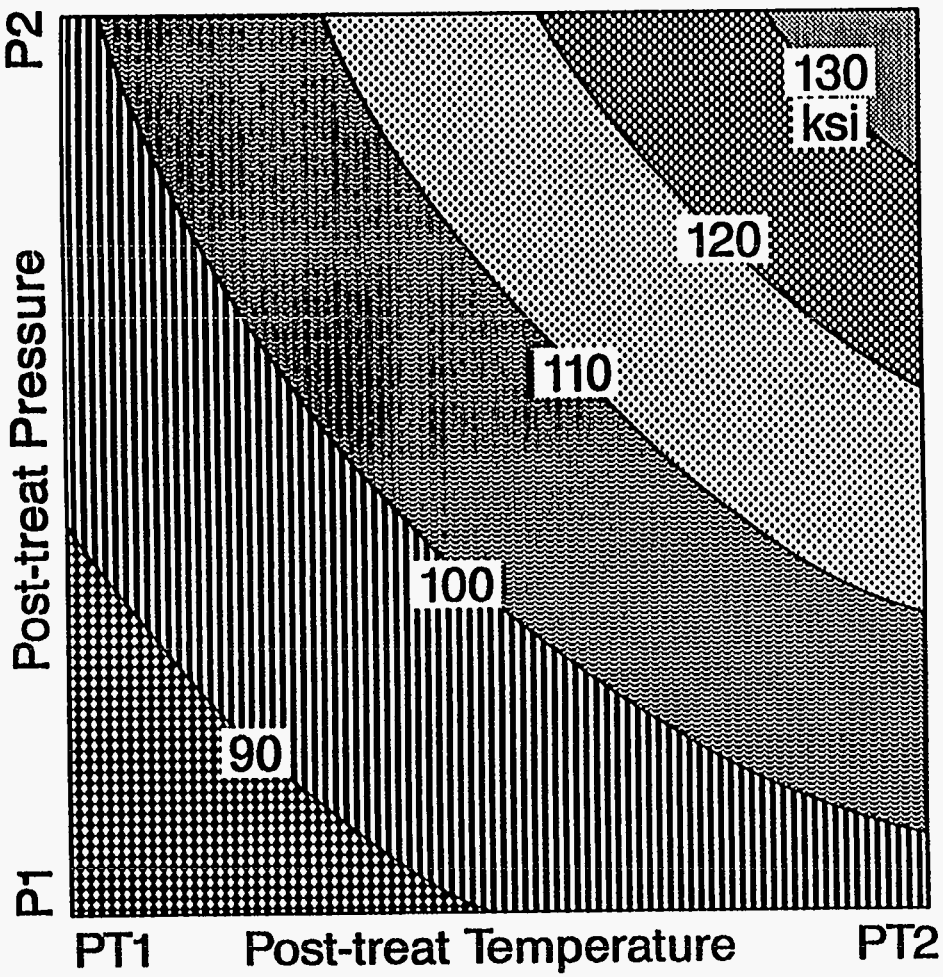

Figure 35. Contour Map of Room-Temperature Flexural Strength as a Function of Post-Treatment Temperature and Post-Treatment Pressure

Task 5a: Further Optimization of SX-G1.

Experimental Procedure and Results

The original objective of Task 5 was to optimize and fully characterize a second generation SX material. Prior to the start of Task 5 under a Carborundum in-house sponsored program, efforts to develop a second generation SX composition (SX-G2) as well as improve the processing conditions of turbomilling were conducted. Results obtained revealed that although there were several compositions identified to possess wider processing windows, the SX-G1 composition developed previously, after several years of intensive work, still provided the best room-temperature and high-temperature strength.

Based on these considerations, the technical approach for Task 5 was modified to conduct a second series of design experiments to further improve SX-G1 using the results described above and those reported under Task 4. The proposed experimental conditions were:

Powder Processing Techniques:	Improved Turbomilling Process
Sinter Temperature:	ST3 to ST4
Post-Treat Temperature:	PT3 to PT4
Post-Treat Pressure:	P3 to P4

The upper and lower limits for each of the furnacing parameters were selected based on the findings described earlier as well as previous Carborundum in-house research data. For example, although it was found that lower sintering temperatures and higher post-treatment temperatures were preferred, Carborundum in-house data has shown that SX-G1 could not be sintered below ST3 to achieve >99% post-treatment density. This defined the lower temperature limit. On the other hand, above T4 excessive silicon pool formation would occur, which defined the upper temperature limit. Regarding the post-treatment pressure, preliminary in-house data revealed that at or above P4 lower mechanical properties were obtained. Similar observations on Si_3N_4 furnacing indicated that too high a gas pressure could have the effect of "pumping" gaseous species into the material microstructure and retarding the sintering cycle. The upper limit for the post-treatment pressure was, therefore, set at P4.

It was estimated that 11 experiments were needed to complete the experimental matrix. Optimum furnacing conditions were to be selected after the evaluation of the resulting mechanical data, including MOR at room temperature and 1232°C, toughness at room temperature and 1232°C, and dynamic fatigue at 1232°C.

After an initial series of experiments to define the optimum turbomilling processing conditions, turbomilling was carried out in several batches at Southern Illinois University to yield 40 pounds of premix. These powder batches were then further processed at Carborundum. The particle size analysis and BET surface area measurements were consistent from batch to batch. The surface area increased from about 15 m^2/g to about 22 m^2/g after this milling process. The powder was then spray dried. Green plates were pressed and baked out. Prior to the start of the designed experiment, two plates were sintered at temperature ST3 since the sintering had never been attempted at this temperature. An acceptable density of 94% T.D. was obtained after pressureless sintering. Post treatment at temperature PT3 and pressure P4 resulted in >99% densification. Flexure bars were machined and an average MOR of 855 MPa (124 ksi) (from 18 samples) was obtained, which was about the order of strength expected from the previous design experiments. It was then decided to use this temperature in the design matrix.

Since a larger quantity of bars was needed for additional characterization and delivery, eight plates per crucible were used during the sintering runs, compared to two plates/crucible for the screening runs, and the sintering temperature was held constant. The density and mechanical property information are summarized in Table 6. The room-temperature MOR is schematically shown in Figure 36. It is surprising to note that plates sintered at temperature ST3 did not achieve density and mechanical property levels expected from the screening trials. For example, an average MOR of 965 MPa (140 ksi) was expected for the center point of the experiment. However, in two trials with a higher furnace loading, the MOR values were 806 MPa (117 ksi) and 813 MPa (118 ksi). The lower strength was attributed to an increased frequency of "pools."

The differences in densification and mechanical properties are believed to be due to the different number of plates used in the crucible for sintering (Table 7). It is possible that the partial pressures of gases are different under the two conditions leading to differences in densification and reaction of second phase(s) with SX SiC. It is realized here that the optimum densification condition is also a function of ratio of mass of green parts to volume of the crucible or, in general, furnacing configuration. This type of furnacing variation is not observed in the processing of HEXOLOY® SA SiC, and is therefore related to the presence of a second phase.

TABLE 6

Density and Mechanical Properties Determined at Specific Densification Conditions in the Expanded Experimental Design Matrix

					MOR, ksi		Toughness MPA. √m		
Sin. Temp	Post Temp	Post Press	Sint. Density %	Post Density %	RT	1232 °C	RT	1232 °C	SCG* Parameter at 1232 °C
–1	–1	–1	85	91.5	57		3.1	2.5	No SCG
–1	–1	1	85	89.9	56		3.1	2.7	No SCG
–1	1	–1	85	93.7	79		4.0	2.7	No SCG
–1	1	1	85	97.4	75		4.0	2.8	No SCG
0	0	0	95.6	99.9	119	78	4.5	3.3	No SCG
0	0	0	94.8	99.8	114	71	4.5	3.1	No SCG
1	–1	–1	97	99.7	95		4.3	3.0	No SCG
1	–1	1	97	99.9	128	69	4.9	3.2	No SCG
1	1	–1	97.8	99.7	104	56	5.0	3.7	No SCG
1	1	1	97.8	99.8	100	55	5.2	4.0	No SCG

* **Slow Crack Growth - No SCG means N is approaching infinity.**

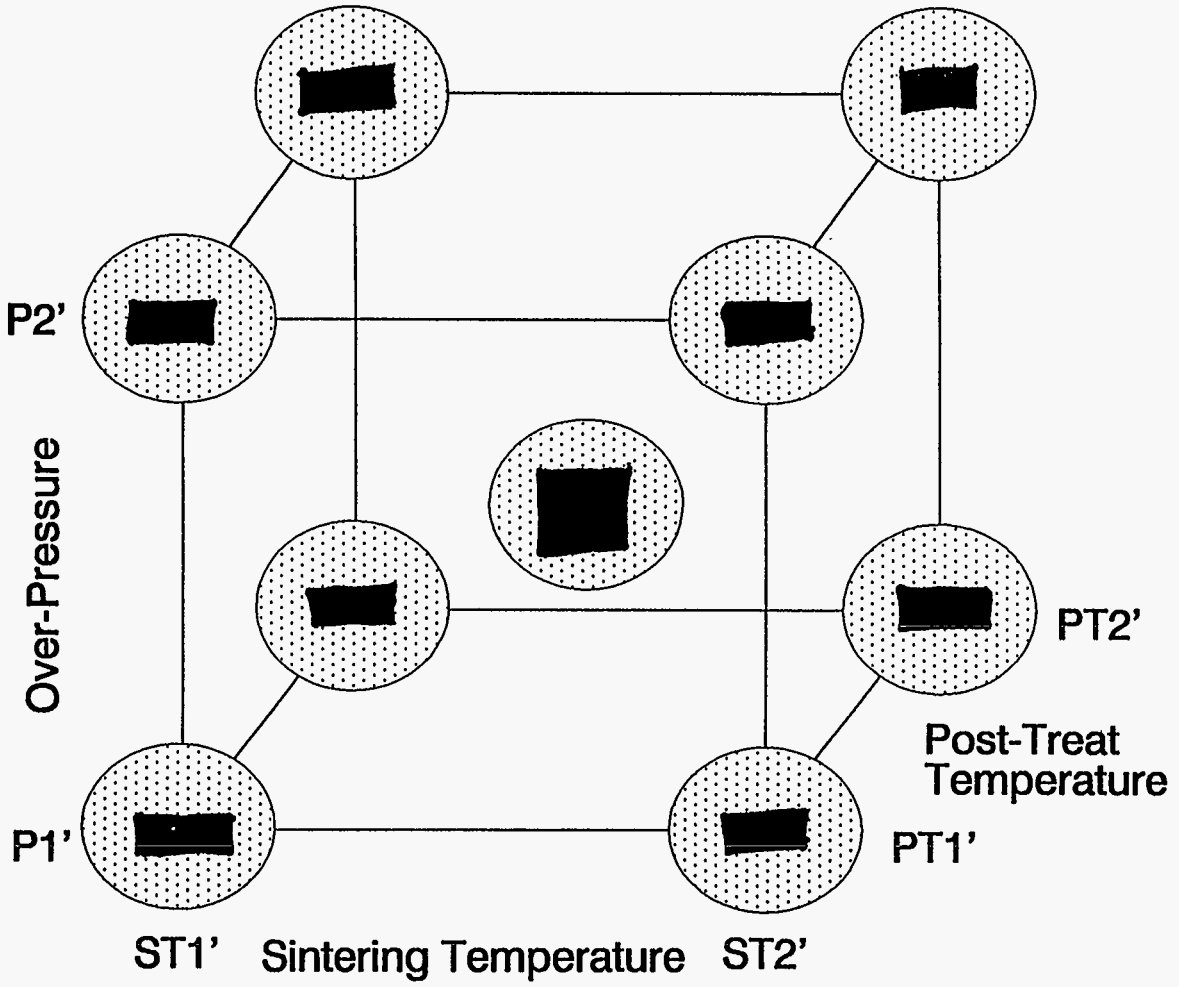

Figure 36. Room-Temperature Flexural Strength Corresponding to Various Densification Conditions in the Expanded Experimental Matrix.

TABLE 7

Summary of Strength and Flaw Data from Task 1, 4 and 5

Task	Fraction of "Pools" as Defects	MOR at RT MPa (ksi)
1	0.7	780 (113)
4	0.27	965 (140)
5	0.3	896 (130)

Task 5b: Complete Characterization of Optimized SX-G1 Composition.

Results

Based on the results obtained in Task 5a, it was determined that the optimum furnacing conditions corresponded to a sintering temperature of ST1 and a post-treatment temperature of PT1. It was realized from both Task 4 and 5 that higher post-pressures were favored. The optimum densification condition was then identified to correspond to a sintering temperature of ST1, post-treatment temperature of PT1, and a pressure of P4, which was higher than the maximum pressure P2 used in Task 3.

The premix powder was compacted into plates and rods. The rods were 216 mm in length and 21 mm in diameter. When densified under the above mentioned processing conditions, the plates resulted in >99% T.D. and rods resulted in 92% T.D. In Task 1 the rods also required a higher sintering temperature than plates for adequate densification. The rods were densified to >99% T.D. using the same conditions described in Task 1.

The stress rupture (static fatigue) and creep tests were not conducted because these properties were not expected to change since the chemistry and microstructure (grain size) were not different from the Task 1 material. However, flexural and tensile strength were evaluated at both room temperature and high temperatures. Dynamic fatigue experiments on flexural samples were conducted as in Task 1 for comparison.

The average room-temperature flexural strength for the optimized SX-G1 was determined to be 896 MPa (130 ksi). This strength is higher than the 780 MPa (113 ksi) obtained for SX-G1 from Task 1, and very close to 965 MPa (140 ksi) obtained from Task 4. The major strength-limiting defect was due to surface defects possibly introduced from grinding. The strength-limiting defects from Tasks 1, 4, and 5b are shown in Table 7 for comparison. It is clear that when densified under the optimum conditions as identified from Task 4, fewer "pools" were observed leading to higher strengths. Acceptable reproducibility in strength was also observed if the furnacing configuration (i.e., number of plates per crucible) was kept the same.

The flexural and tensile strengths determined at elevated temperatures are shown in Figure 37 and compared with the strength measured in Task 1. The strength at elevated temperatures is about the same for starting SX-G1 from Task 1 and optimized SX-G1 from Task 4 and 5 as expected. The dynamic fatigue response is shown in Figure 38 for both Task 1 and Task 5 materials. Again, no difference in slow crack growth characteristics was observed as expected. The uniaxial tensile strengths obtained from Task 1 and Task 5 are also about the same as seen in Figure 37. No

strengths obtained from Task 1 and Task 5 are also about the same as seen in Figure 37. No difference in strength was expected as the tensile rods were densified under the same sintering conditions.

The above characterization study reinforces the fact that sintering conditions, including the furnacing configurations, are very important in obtaining reproducible and improved mechanical properties.

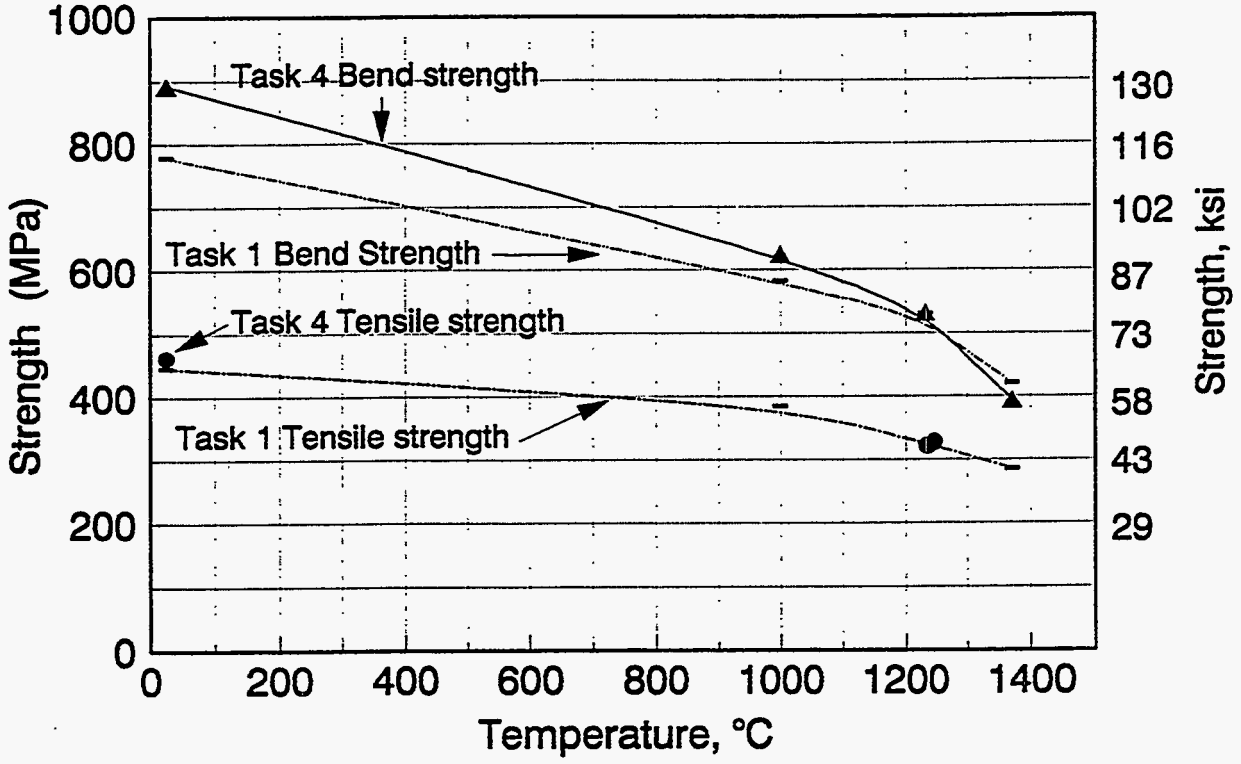

Figure 37. Flexural and Tensile Strength Determined in Tasks 1 and 5b at Various Temperatures.

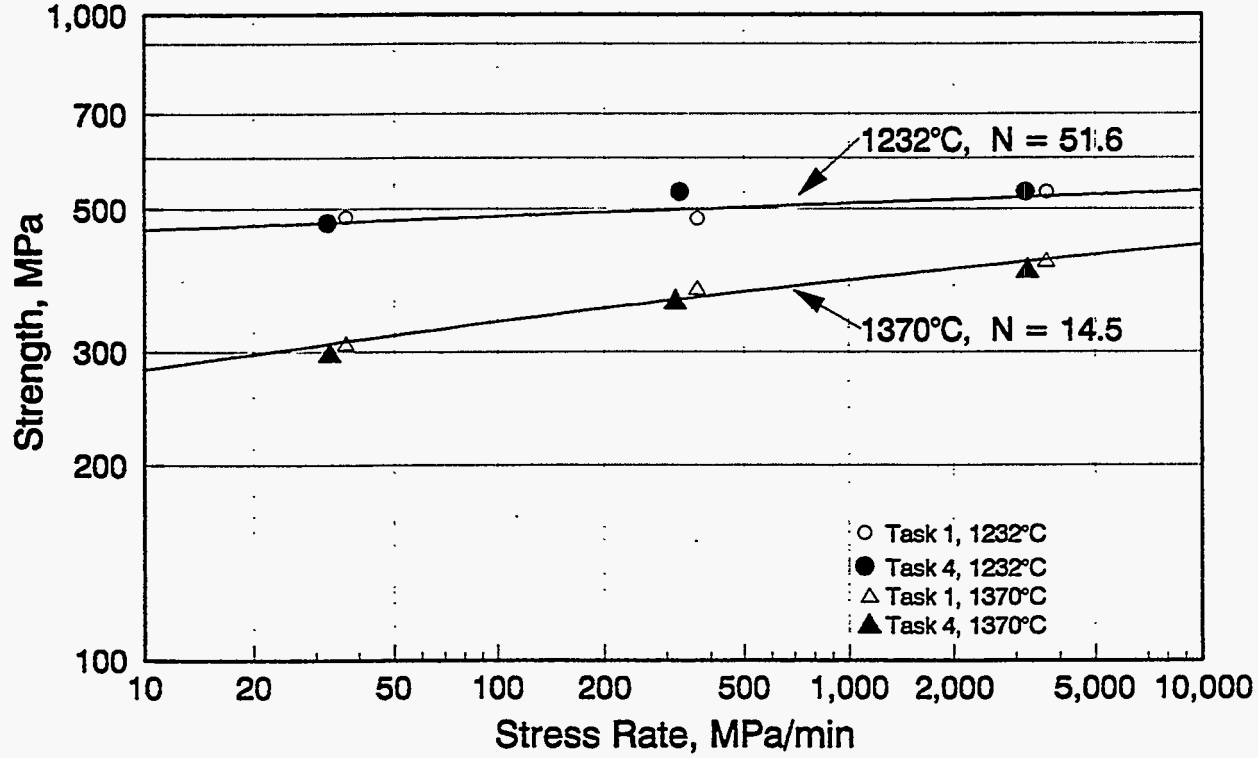

Figure 38. Dynamic Fatigue Response Measured from Tasks 1 and 5b.

SUMMARY

- A complete mechanical property database and structure–property relationship was established for the SX-G1 starting composition.
- From room temperature to 1232°C, the predominant strength-controlling defect was the silicon-rich pool of second phase clusters resulting from the reaction of yttrium aluminates with SiC.
- At elevated temperatures the decrease in strength was primarily due to the decrease in fracture toughness.
- Slow crack growth occurs at temperatures above 1300°C in air because of oxidation and subsequent glass-phase formation.
- The material possesses excellent creep resistance at all temperatures tested (up to 1450°C).
- Reduction in toughness and slow crack growth at elevated temperatures were inherent limitations of this material because of its chemistry.
- Turbomilling provides excellent mixing and dispersion but suffers from reproducibility due to excess wear of grinding media.
- Sintering-condition optimization lead to a reduction in reactivity of second phase and enhancement in room-temperature strength. Average flexural strength as high as 965 MPa (140 ksi) has been obtained.
- However, the optimum densification conditions and reactivity of second phase with SiC also depended upon furnacing configuration.

CONCLUSIONS

The results show that SiC materials sintered with the addition of yttrium and aluminum compounds can achieve the high level of mechanical properties required for their use in heat engine applications. Average MOR values up to 965 MPa (140 ksi) were demonstrated with a room-temperature fracture toughness up to two times that of boron and carbon sintered SiC. However, the reaction of the second phase with the SiC leading to strength-limiting pools results in increased variability in material properties. This variability was observed with both different sample sizes (i.e., plates vs tensile rods) and furnacing loads. If this variability can be eliminated or controlled, the relatively low cost of raw materials and processing for HEXOLOY® SX SiC manufacture would make these materials attractive candidates for commercialization for both low- and high-temperature engine applications.

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