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PROCESS DEVELOPMENT FOR SILICON CARBIDE
BASED STRUCTURAL CERAMICS

January 1983

Edward E. Hucke, Principal Investigator
Materials and Metallurgical Engineering
The University of Michigan
Ann Arbor, Michigan 48109

Interim Report for Contract No. DAAG46-80-C-0056 -P0004

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER
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of 714 MPa which exceeds commercial reaction bonded SiC (NC433) by ~100%, sintered α -SiC by ~85%, and approximately equals hot pressed SiC (NC203) when tested under identical conditions. The fracture toughness, K_{IC} , measured by vickers indentation testing was approximately the same as hot pressed Silicon carbide (NC203).

Efforts to produce finer structured materials have not yet reproducibly achieved uniform microstructures free from silicon veining.

The materials thus far produced give Weibull moduli in the range 5 to 7 compared with 10-15 for the commercial materials. Fracture analysis indicates the lower values are due to machining defects and undesirable microstructures at the surfaces which were incompletely removed in sample preparation.

SUMMARY

The objective of this program is to develop a process for making shaped silicon carbide based ceramic materials with reduced microstructural flaw size by in situ reaction of silicon with fine, ultra-uniform pored carbon skeletons that are produced from liquid polymer solutions without particulate additions.

A very uniform siliconized microstructure has been made from a carbon skeleton with average pore size of $\sim 1.9 \mu\text{m}$ and apparent density of $\sim .85 \text{ gm/cm}^3$. This material had a room temperature, four point Weibull characteristic strength of 714 MPa which exceeds commercial reaction bonded SiC (NC433) by $\sim 100\%$, sintered α -SiC by $\sim 85\%$, and approximately equals hot pressed SiC (NC203) when tested under identical conditions. The fracture toughness, K_{IC} , measured by Vickers indentation testing was approximately the same as hot pressed Silicon carbide (NC203).

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INTRODUCTION

The current experimental program is aimed at developing methods to produce shaped highly reproducible silicon carbide based structural ceramics. The emphasis is toward understanding the processing origins of the various microstructural flaws and devising means for their minimization.

The process chosen for refinement is a variant of reaction bonding, which has the potential advantages of yielding high accuracy shaped objects of substantial size. A detailed rationale and discussion of the approach adopted was given previously.¹ The major difference between this approach and others is that no particulate material is processed in making the shaped body. Instead, the body is made from low viscosity castable liquid polymer which after polymerization is converted to a controlled porosity pure carbon skeleton. Subsequently, the skeleton is reacted with silicon to form the silicon carbide cermet.

The control of pore size distribution and maximum carbon dimension within the skeleton is excellent and allows a greater degree of control of the subsequent siliconization. The manipulation of the skeleton parameters is through variation of chemical variables such as concentrations, times and temperatures and not mechanical processes such as grinding, mixing, and pressing.

Since the microstructural strength limitations in ceramics are determined by the largest defects the present

approach is aimed primarily at producing maximum spatial uniformity and at simultaneously reducing the structural feature size.

Results thus far achieved have demonstrated a high degree of reproducibility in the skeleton parameters and the ability to tailor properties such as skeleton density and pore size.²

EXPERIMENTAL RESULTS

Carbon Skeleton Development

The major efforts have been toward further refining the major skeletal variables as experience in siliconization is acquired. The previous work^{1,2} had indicated the range of variables needed to produce a highly uniform fine grained silicon carbide and demonstrated that generally finer structures result from fine skeletons. The major variables are apparent density, pore size, distribution and carbon solid size distribution.

Skeleton Apparent Density: This factor controls the residual silicon contents and should lie in the range of .82 (14.8 vol% Si) to .91 (5.5 Vol% Si) gm/cm³ depending on the residual silicon content desired. In general, lower silicon content is sought but a compromise is struck since below 5 Vol% Si complete infiltration of large sections becomes very difficult while at higher silicon content the strength and probably toughness are reduced. A value of .865 gm/cm³ (~10 Vol% Si) is typically selected as a goal. Aside from producing the correct average carbon density it is vital that the density be spatially

uniform over a very small scale, preferably less than 5 μm . Current findings continue to confirm¹ that the density can be controlled within 1% (the approximate measuring error) over the extremities of cast blocks 15 cm $\times$ 10 cm $\times$ 5 cm. Reproducibility between various casts of a given batch and repeated batches is within experimental measuring error.

An effort has been made to make the pore size distribution as narrow as possible. It is particularly important that there be no large pores since these can deliver silicon too rapidly ahead of the advancing siliconization interface and cause rapid local heating with distortion and cracking from thermal stress. The pore size determines the rate of silicon penetration into the body and must be large enough to permit flow of at least several cm in reasonable time ($\sim$ 1-2 hours). However, there is a definite advantage to keeping the pore size small since the slower the advance of the reaction interface the more nearly isothermal the reaction can become. This reduces the thermal stresses and allows less local overheating to cause grain growth and silicon lake formation.

Previous experience² had indicated that a skeleton (381-2B) with an average pore size of 1.9 μm could be filled adequately. Additional skeletons in this pore size range have been produced as well as some at a much finer level ($\sim$.5 μm) aimed at generally refining the size of the resulting silicon carbide.

The carbon "solid size" must be carefully controlled since any sections of the carbon skeleton that are too thick

will not be completely reacted. As the rate of reaction is slowed the maximum thickness of carbon must also be reduced since there is a much smaller temperature excursion to aid reaction. While relatively large carbon particles ($>100\text{ }\mu\text{m}$) can be fully reacted with large exotherms this is to be avoided since it generally coarsens both the resulting silicon carbide and causes silicon lakes due to solution and reprecipitation or may cause cracking.

It is also necessary to decrease the maximum particle size as the skeleton density is raised particularly in the range where the residual silicon is below 10 Vol%. In this case the flow is slower due to the lower volume of delivery channels and each particle is further from its reactant supply. Previous efforts² showed that the maximum size should be below $10\text{ }\mu\text{m}$ for complete reaction with a skeleton density in the range of .8 to .85 gm/cm³. It has been found that at higher densities that the maximum size needs to be further reduced, probably to less than $2\text{ }\mu\text{m}$.

In the current period several dozen different batches have been produced aiming at a pore/particle size less than $1\text{ }\mu\text{m}$ and the range $\sim 1\text{-}3\text{ }\mu\text{m}$. Several of these have been made in two density levels so that processing and properties can be evaluated as a function of residual silicon content.

The carbon skeleton can be shaped in several ways. It may be cast or machined in the polymerized state or machined after carbonization. Each has advantages in certain cases.

Machining the polymer to close tolerances is easy but the carbonization shrinkage must be known accurately. Previous work^{1,2} has demonstrated that this shrinkage is quite reproducible for a given processing scheme. It was noted² that very long thin sections warped somewhat during carbonization. Significant progress has been made in reducing this bending. The major factors which seem to be responsible for the improvement are further extension of curing time for the polymer prior to machining, and carbonization with the samples packed in fine sand in the vertical position instead of on horizontal graphite plates. Table I shows results of measuring the centered "bowed" region with a dial indicator on representative sets of samples. The samples were regular bend test samples about .25 x .38 x 4.5 cm. A group of samples machined after carbonization is included for comparison. The figures given are for the averages and standard deviations of the maximum deviation from straightness in two directions. The more recent samples which have a length to thickness ratio of ~15 have been held straight to within about .002 cm over their length. In some cases the result is within .001 cm which is about the same as the result for samples machined after carbonization. This level is adequate for most applications but further improvement and delineation of the important variables will be pursued.

Siliconization

No major changes in the siliconization times or temperatures^{1,2} have been made. However, an additional complication

TABLE I. DISTORTION DATA FOR BAKED CARBON SKELETONS

Mix No.	Machining Baked & Machined	Cured 95°C Days	Baking Support	No. of Spec.	Average Dimensions		
					H./cm	W./cm	L./cm
331-57	Machined	-	-	20	0.260	0.512	3.814
331-27	Machined & Baked	None	V-grooves Gr. Plate + sand	20	0.233	0.322	4.017
			Flat Gr., Plate + sand	20	0.230	0.318	4.048
331-28A	"	None	Flat Gr., Plate + sand	20	0.233	0.317	4.040
331-57	"	49	V-grooves Gr. Plate + Gr., Felt	11	0.245	0.335	4.884
331-57	"	49	V-grooves Gr. Plate + Gr., Felt	15	0.244	0.335	4.895
331-60B	"	8	V-grooves Gr. Plate + Gr., Felt	16	0.240	0.383	4.752
331-60B	"	8	V-grooves Gr. Plate + Gr., Felt	20	0.254	0.509	3.812
331-60B	"	8	V-grooves Gr. Plate + Gr., Felt	27	0.239	0.380	4.744
331-60B	"	8	V-grooves Gr. Plate + Gr., Felt	20	0.239	0.380	4.728
331-67	"	14	Vertical Position in sand	20	0.240	0.300	4.965
332-8	"	16	Vertical Position in sand	24	0.240	0.290	4.840
332-11	"	16	Vertical Position in sand	30	0.238	0.290	4.794
332-12	"	16	Vertical Position in sand	67	0.240	0.295	4.844

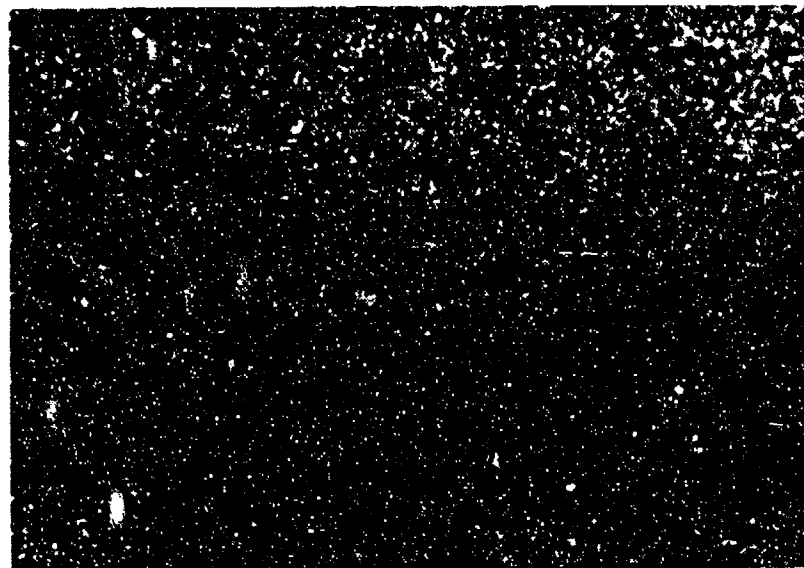
TABLE I CONT.

Mix No.	Avg./Max. Bending			Bending Distribution (%)				
	H. cm $\times 10^{-3}$	Std. Dev.	W. cm $\times 10^{-3}$	Std. Dev.	0-2.5cm $\times 10^{-3}$	2.5-5cm $\times 10^{-3}$	5-7.5cm $\times 10^{-3}$	>7.5cm $\times 10^{-3}$
331-57	1.52	1.27	0.36	1.29	85	15	0	0
331-27	17.09	22.3	40.0	37.31	0	0	15	85
331-28A	4.78	4.34	10.70	7.95	0	24	20	56
331-28B	9.45	6.78	21.33	19.13	5	10	5	80
331-57	5.33	3.40	8.13	6.50	0	27	27	46
331-57	7.77	5.21	8.89	4.47	0	7	7	86
331-60B	4.83	5.43	6.35	4.42	0	44	19	37
331-60B	0.46	0.61	1.27	1.60	95	5	0	0
331-60B	2.29	2.06	4.57	2.79	41	19	25	15
331-60B	2.29	2.49	5.84	4.34	40	20	15	25
331-67	3.3	1.63	4.83	3.56	25	50	10	15
332-6	3.61	4.80	8.58	6.10	17	29	4	50
332-11	1.02	1.42	3.05	3.15	40	40	13	7
332-12	6.35	11.4	4.32	3.45	40	22	21	16

has arisen from efforts to lower the residual silicon density by raising the carbon concentration above $.85 \text{ gm/cm}^3$. It was previously noted^{1,2} that a narrow subsurface region sometimes exists where unreacted carbon balls remain. This is particularly pronounced on small samples (e.g., bend test bars), siliconized at lower temperature $\sim 1460^\circ\text{C}$ and short times (< 10 hours). Reaction heating is apparently not sufficient in these cases to completely react the carbon. When the maximum carbon size is $< 10 \text{ }\mu\text{m}$ and the density $< .85 \text{ gm/cm}^3$, 10 hours is usually long enough to completely react the carbon. However, such long times contribute to a liquid grain growth region at the sample surface so that generally much shorter siliconization time is desirable.

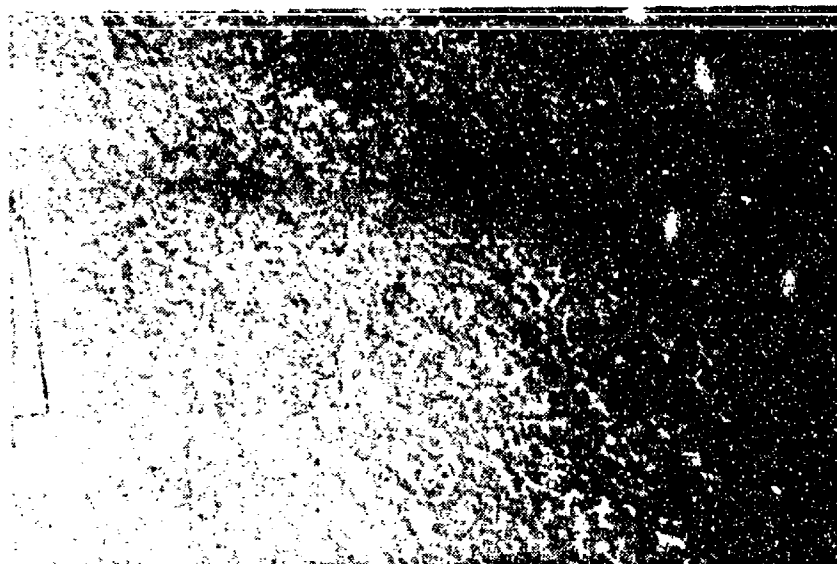
At densities $> .85 \text{ gm/cm}^3$ the tolerable maximum particle size is much smaller (see previous discussion) and must be reduced, probably below $2 \text{ }\mu\text{m}$.

In order to remove the residual carbon two procedures were evaluated. First the siliconization time was extended up to 23 hours and the temperature increased modestly to $\sim 1500^\circ\text{C}$. This procedure definitely decreased the size and frequency of the residual carbon in the subsurface zone as can be seen in Figs. 1 and 2. Very slight coarsening of the structure can be detected and a few $< 2 \text{ }\mu\text{m}$ carbon "balls" can still be seen. These balls need to be avoided since due to their low coefficient of expansion undoubtedly induce residual tensile stresses in the carbide regions immediately surrounding them.



|—|
10 microns

Figure 1. Micrograph shows 331-60B after siliconization at 1450°C for 10 hours with subsurface zone containing residual carbon (dark).



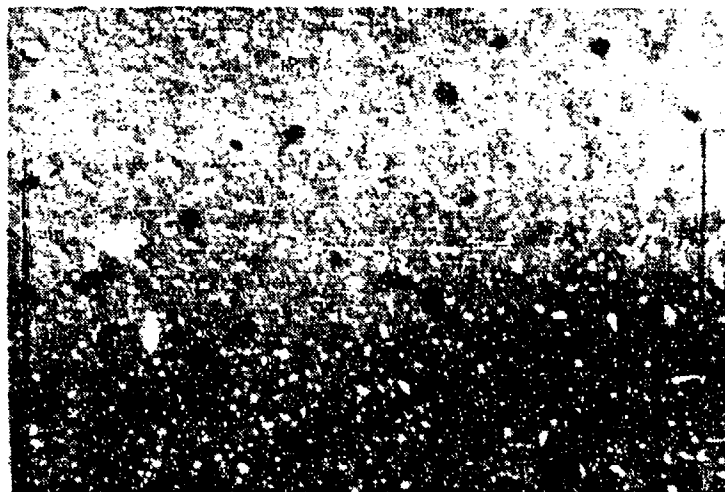
|—|
10 microns

Figure 2. A sample of 331-60B after an additional 23 hours at 1475°C showing almost complete reaction of residual carbon and some agglomeration of the carbide.

The second procedure used an additional 3.5 hour higher temperature ($\sim 1575^{\circ}\text{C}$) heat treatment subsequent to siliconization. In this case the samples were packed in a crushed silicon carbide grit which contained a small amount of residual silicon in order to avoid excessive vaporization loss of silicon from the samples. This procedure eliminated the balls almost completely but gave a rather perceptible coarsening of the structure as can be seen in Figs. 3 and 4.

It was also noted that the samples gained weight during the high temperature heat treatment. No significant size increase occurred, but the approximate weight gain would indicate that the volume % silicon had increased from ~ 11.6 to 12.2 . In addition the samples were very weak as judged by breaking with ordinary finger pressure, which can not normally be done with a good sample of this size. This may be due to very pronounced silicon carbide growth region at the surface.

The long term solution to the residual carbon problems must be sought in refining the skeleton since prolonged treatments are not desirable particularly when only a small region of the sample requires the treatment. This region is particularly important to the strength properties since it is near the surface, but deep enough to be incompletely removed with ordinary grinding. In addition, elimination of the liquid grain growth region² at the surface also requires that processing time be minimized.



10 microns

Figure 3. As siliconized 331-70 showing residual carbon (dark) in subsurface zone and fine (unresolved) silicon carbide (gray).



10 microns

Figure 4. A sample of 331-70 after an additional 3.5 hours at 1565°C shows almost complete elimination of residual carbon and substantial carbide growth and agglomeration.

Many of the problems in optimizing the siliconization process for a given skeleton are expected to be section size dependent since the filling rate and heat released are functions of the section size. Routinely ~2 cm diameter x ~1 cm thick discs are siliconized prior to preparation of much smaller bend test specimens. In some cases different structures result from the same skeleton with the same processing for the two section sizes. A more systematic study of the effects is underway. Recently larger blocks ~1.4 cm thick by 5 cm x 2.5 cm have been filled in 10 hours with satisfactory structure from a carbon ($.89 \text{ gm/cm}^3$) with a pore size ~3 μm .

A recent check of the siliconization furnace showed a much larger temperature gradient over the working volume than had been expected. Often three stacked trays have been used and it was observed that the bottom tray was only about 6°C hotter than the middle tray but ~60°C hotter than the top tray. This large temperature difference has undoubtedly caused some variation in structure and properties and may well be responsible for some differences noted in the strength properties noted (see later discussion) between presumably identical groups of samples treated in the various trays in the same heat. Steps are being taken to remedy the temperature distribution in the working zone. Such steps include vertical repositioning of the heat treating chamber with respect to the heating element and additional insulation of the top.

PROPERTY EVALUATION

Composition and Structural Characterization

A variety of metallographic techniques has been employed to yield information about the structure of both the carbon skeletons and the reacted materials. These are relatively successful in assessing the local structure but due to the fineness of the structures it is difficult to quantitatively establish properties reliably.

A pressing need is to develop a fast but reliable method for determination of the free silicon content. Quantitative metallographic methods either evaluate too small an area or at lower magnifications can not resolve the silicon. An attempt has been made² to measure the free silicon by X-ray techniques. At the present time this method appears able to reproduce results in the range 6-16% Si within $\pm 2\%$ Si. This accuracy needs to be improved in order to be very useful. Several sources of error have been identified. The most important relates to the need for very good alignment of the X-ray equipment and the exact placement of the powder sample in the holder. These rather elementary considerations have presented a major problem in an X-ray laboratory used intermittently by a large number of students. A continued effort will be made to improve the X-ray method. If no further improvement is practical, this method will be dropped in favor of a calculated silicon content from an Archimedes density measurement. At present this method gives the most reproducible results. More rigorous methods³ of

analysis are available but are beyond the scope of the present investigation.

Indentation Testing. In the past few years an impressive series of papers⁴⁻⁸ has analyzed the damage induced by controlled indentation into brittle materials. The type and extent of damage under an indenter is a very important technological quality of the material. Quantitative measures of this quality has obvious uses in developing materials for various service conditions. The complete range of indentation behaviors even under ideal geometric conditions such as a Vickers diamond indenter is quite complicated, ranging from nearly elastic in polymers through a variety of elastic-plastic processes in metals and ceramics.

For many candidate structural ceramic materials a well defined radial/median crack system develops under a Vickers indenter.

For some, but not all ceramics there is no significant growth in the radial crack system in periods of several hours after removal of the load. This has been the case with the silicon carbides under study². It has, however, been possible to notice time dependent growth of the lateral crack system, where severe chipping or spalling due to residual stress has been observed to occur several days after removal of the load.

For well behaved impressions the extent of cracking can be related directly to K_{IC} ⁸ obtained by other means. In order to yield satisfactory results careful determination and handling

of the experimental data is required. Generally unsatisfactory results were obtained² if data were limited to one indenting load and correlation made via the techniques in the earliest paper⁴. If, however, data for the impression diagonal, $2a$, and surface median crack trace, $2c$, are gathered over a range of indenter loads, P , and analyzed statistically, highly reproducible results are obtained.

For materials with no substantial slow crack growth and well developed radial cracks there is a material independent constant⁸ which relates the material properties, toughness, K_{IC} , hardness, H , and elastic modulus, E , to the crack size, c , and the load P ;

$$S_V^R = \frac{K_{IC}(H/E)^{1/2}}{(P/c^{2/3})} = .016 \pm .004 \quad (1)$$

The constant contains all of the geometric, surface constraint and other test variables and has been established within the range quoted by comparison with a variety of test ceramics.

Equation (1) indicates that a plot of $c^{3/2}$ versus P should be linear suggesting that a least squares fit with P as the independent variable would be an appropriate way to establish the slope,

$$\text{Slope} = S_V^R \left(\frac{E}{H}\right)^{1/2} \frac{1}{K_{IC}} \quad (2)$$

It is worth stressing that whether or not Eq. (1) holds precisely with the same constant for all materials is not of crucial importance for materials development purposes since the

slope of the $c^{3/2}$ vs P curve becomes a good figure of merit in its own right provided the slope is highly reproducible for a given material. In short, it takes little imagination to see that a small slope means a tougher material, i.e., less cracking at a given load.

The data gathered for NC203 are plotted in Fig. 5. Since this material is used for comparison purposes a relatively large number of points (25) was measured at 5 loads ranging from 1 to 5 KG. All points were taken on a single modulus of rupture specimen supplied by AMMRC⁹. Additional data on another sample of NC203 are being gathered to check material variability.

The fit shown in Fig. 5 is typical of that obtained on most of the samples. The error bars shown are the standard deviations for $c^{3/2}$ while the line shown is that using all experimental points with equal weighting.

Typical standard deviations for 2a and 2c were <1.5% and <6%, respectively. The resulting equation for NC203 is:

$$c^{3/2} = (-3.34 \pm .66) + (7.47 \pm .19)P \quad (3)$$

where P is in KG and C is $\text{mm} \cdot 10^{-2}$. The linear fit gives a coefficient of determination, $r^2 > .99$ for 25 points.

With the appropriate unit conversion and the same choices of properties taken in Ref. 8, i.e., $K_{IC} = 4.0 \text{ MPa} \cdot \text{m}^{1/2}$, $H = 24 \text{ GPa}$, $E = 436 \text{ GPa}$ the experimental slope is,

$$S_V^R = .023 \quad (4)$$

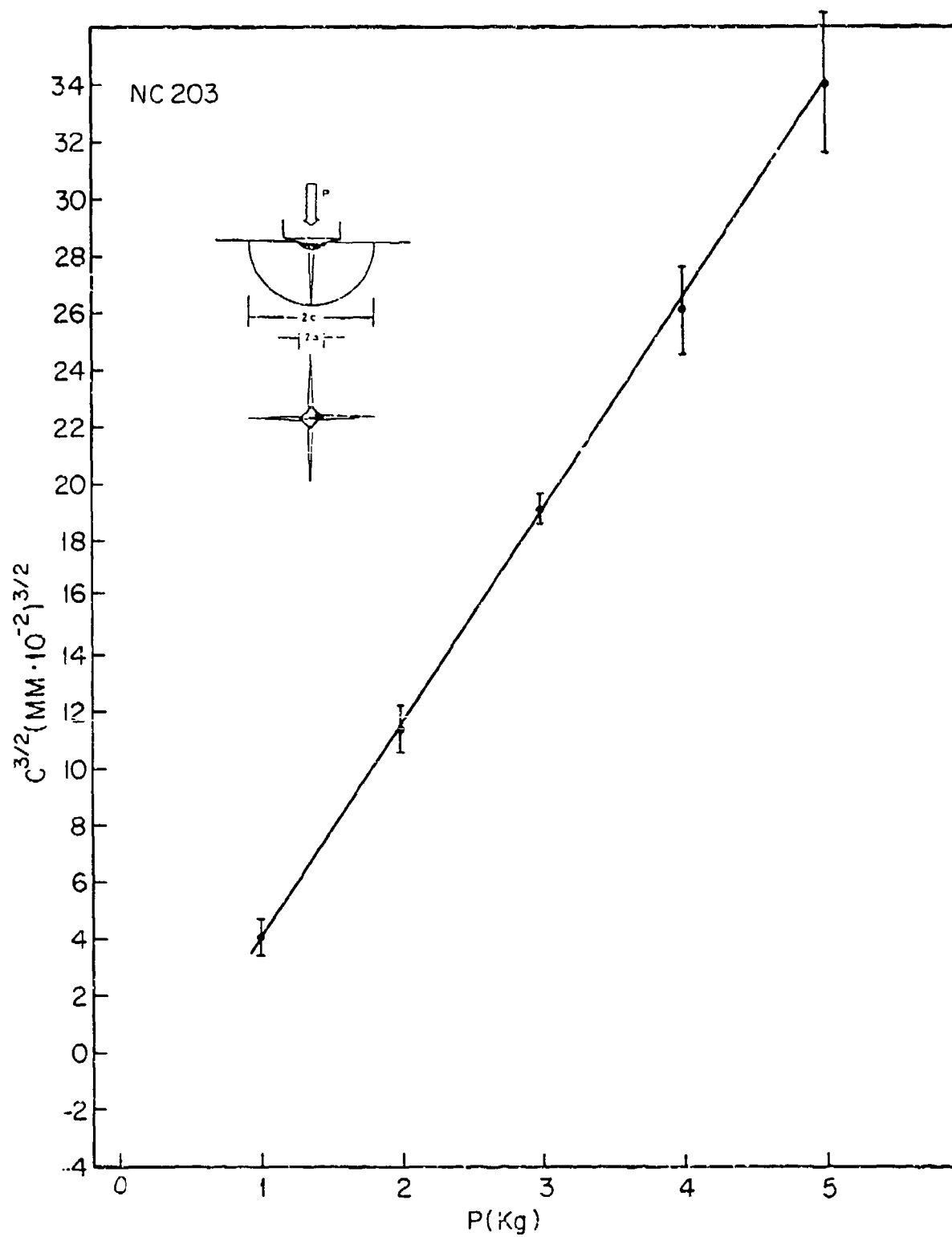


Figure 5. Median cracking versus load in a Vickers test for hot pressed silicon carbide (NC203).

The data indicate a standard error for the slope of about 2.7% which would still not reduce the value of S_V^R enough to fall within the range stated in Ref. 8,

$$.012 < S_V^R < .020 \quad (5)$$

It should be noted that there is a good deal of uncertainty in the values to assume for K_{IC} , E and even H . For NC203 the values used by reputable authors vary considerably, i.e., $H = 19.3$ to 24.0 GPa; $E = 420$ to 448 GPa; $K_{IC} = 2$ to 5.1 MPa $\cdot$ m $^{1/2}$. While the absolute value for S_V^R would be affected by the choice of constants this would not account for the disagreement since the same values have been used for each calculation.

The standard error estimate for the experimental slope is less than 3% and was typical of the error found in this study for other silicon carbide based materials. While NC203 was one of the materials studied in Ref. 8, the logarithmically plotted data given in the publication can not be read accurately enough for a direct comparison. This material was not one chosen as one of the calibration materials for calculating S_V^R .

It is obvious then that quite small changes in the crack propagation behavior as measured by the slope of $c^{3/2}$ vs P can be significantly differentiated but that reflecting these changes into K_{IC} values is much less certain due to uncertainties in the other constants.

By least square fitting of the square of the impression diagonals, a^2 , versus load, P , as the independent variable the

Hardness is $H_{V\infty}$, which is the hardness at large load and is independent of load and inversely proportional to the slope of the a^2 vs P line. The constant of proportionality is determined by the geometry of the Vickers indenter and had value 4636 for the choice of units given with eq. (6).

For NC203 the fit of a^2 vs P is given in Fig. 6 and is typical of the fit obtained on the other materials studied. The best fit equation determined over the range 1 to 5 KG is:

$$a^2 = (.53 \pm .07) + (2.21 \pm .02)P \quad (5)$$

where P is in KG and a is $\text{mm} \cdot 10^{-2}$. The linear form gave a coefficient of determination, $r^2 > .99$, for 25 points. For NC203 the hardness is then,

$$H_{V\infty} = \frac{4636}{2.21} = 2097 \pm 20 \text{ KG/mm}^2 \quad (7)$$

Accurate determination of the hardness still leaves the modulus, E , and K_{IC} to be selected from the literature or accurately measured. Until such reliable E values are available for all the developmental materials, the K_{IC} will be determined by comparing slopes of the $c^{3/2}$ vs P curves with the assumption that $K_{IC} = 4.00 \text{ MPa} \cdot \text{m}^{1/2}$ for NC203 and that $(E/H)^{1/2}$ is the same for all the materials.

The correlations of $c^{3/2}$ and a^2 to the independent variable, P have obvious physical bases. In the case of a^2 vs P the usual definition of Vickers hardness would demand that the constant term in Eq. (6) be zero. However, a least square fit passing through the origin may be tested with the Gauss test¹⁰

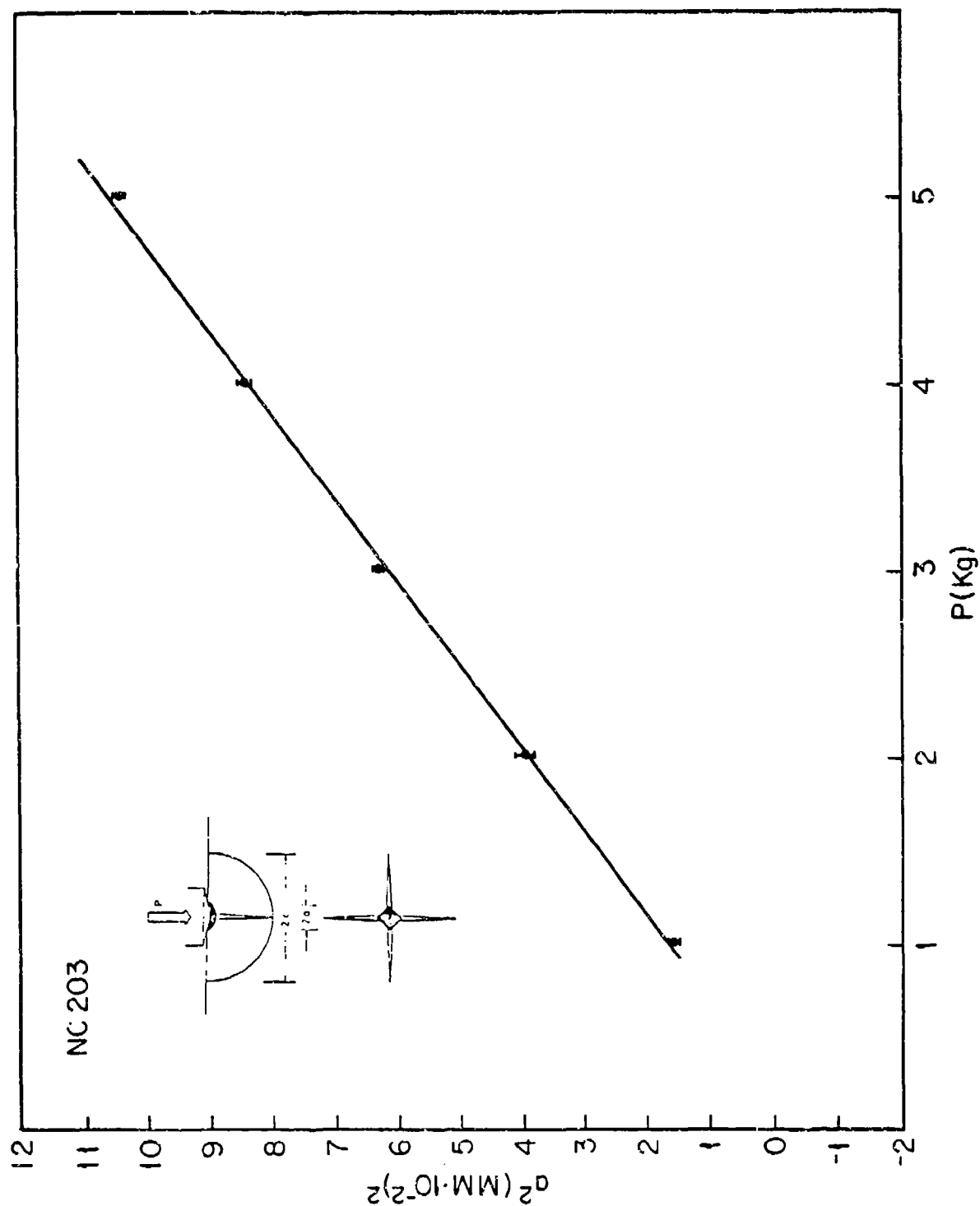


Figure 6. Square of impression diagonal versus load in a Vickers test for hot pressed silicon carbide (NC203).

to determine whether or not the constant is more appropriate. For NC203 and all other materials tested the constant should be included, which means that the linear form can not hold for very low loads and that the conventionally determined hardness will be higher and quite load sensitive at small loads². Such behavior is often noted with microhardness data on ceramics at low loads i.e., <1 KG.

In the case of $c^{3/2}$ vs P the linear behavior is expected to be limited to large P and it is therefore somewhat surprising to observe that the linear behavior exists to rather low loads and $c/a < 2$. In this case also the constant term in (3) gives a significantly better fit than a curve forced through the origin.

While the slopes of the $c^{3/2}$ and a^2 vs P lines measure the crack propagation behavior and hardness directly, it is not clear what physical significance the respective constant terms may have. At the least they may be regarded as additional statistical characterizations of the respective fits. These constants will have somewhat larger standard errors than the respective slope terms. It is possible that they are related to some more recognizable physical qualities of the material. Such a possible interpretation has been pointed out² and can be seen by expressing $c^{3/2}$ as a function of a^2 by either eliminating P algebraically from Eq. (3) and (6) or by considering a^2 as independent and least square fitting the $c^{3/2}$ data against a^2 .

The latter is probably more desirable, but the former clearly shows the role of each of the constants in the new relationship.

An example of the result of a least squares fit is shown in Fig. 7 for NC203 and is typical of the other materials studied. The fitted curve is quite linear over the data range, having a coefficient of determination $r^2 > .98$ for 25 points. The best fit is:

$$c^{3/2} = (-1.49 \pm .63) + (3.36 \pm .09)a^2 \quad (8)$$

and c and a are $\text{mm} \cdot 10^{-2}$.

A very similar result is obtained from the algebraic elimination of P between Eq. (3) and (6). In Fig. 7 the locus of points where $a = c$ is also plotted. This curve represents the limiting case where indentation takes place without developing any radial cracks. If the experimental curve follows the same functional form until it intersects the $a = c$ curve, this intersection would represent the cracking threshold in terms of $a^* = c^*$, the impression diagonal that must be exceeded to get a radial crack. Mathematically $a^* = c^*$ can be solved from Eq. (8) while the value of load, P , corresponding can best be solved from substituting a^* into Eq. (6). The values would appear to have the same significance as the values discussed in Ref. 5. More forgiving and therefore more desirable materials would have higher values for both P^* and c^* .

Table II shows the results of indentation analysis on a number of materials where strength data is also known. In most cases the data were taken on the rupture bar of maximum strength

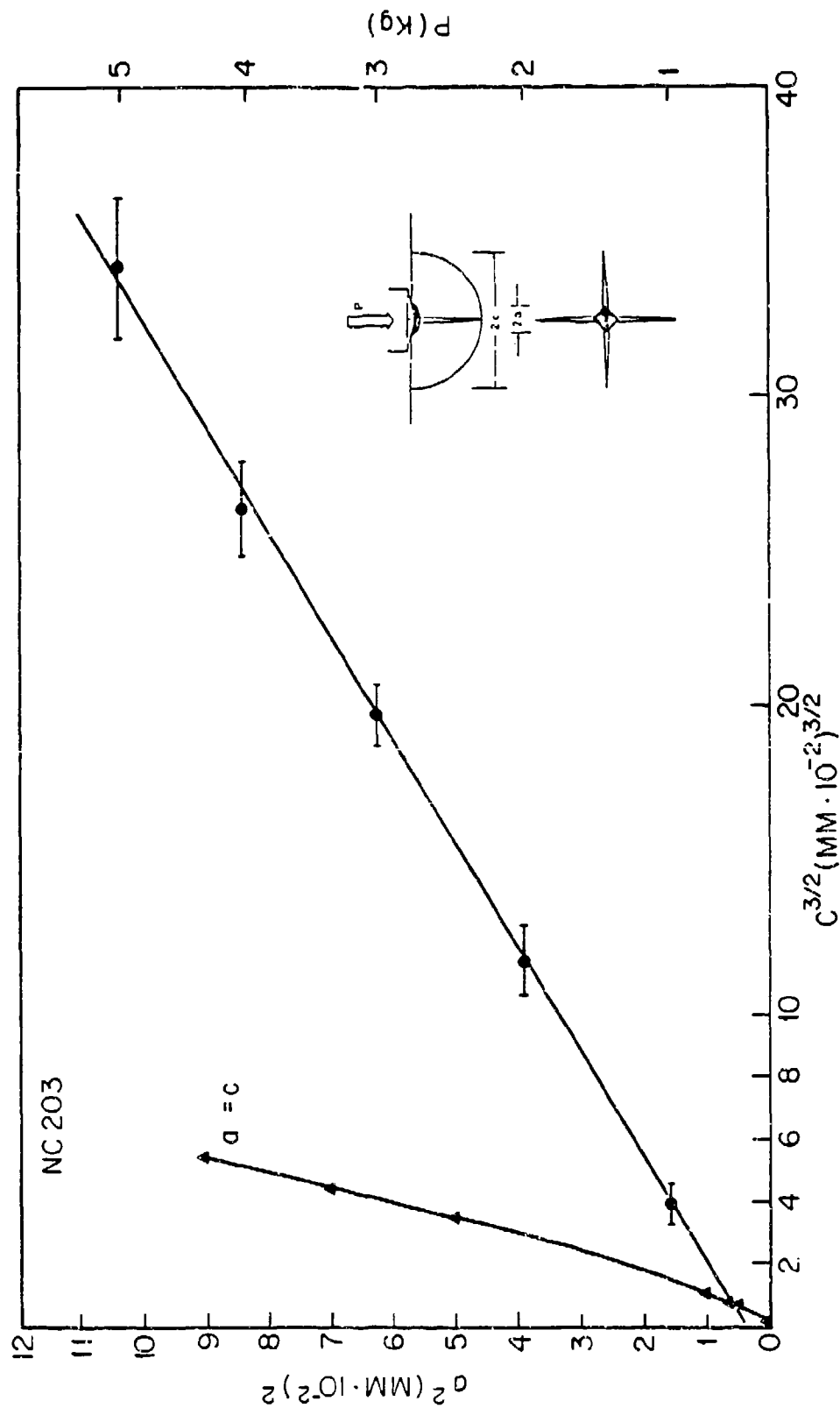


Figure 7. A least squares fit of $c^{3/2}$ against a^2 as independent variable for hot pressed silicon carbide NC203.

TABLE II. SUMMARY OF RESULTS OF INDENTATION TESTING

MATERIAL	c^* μm	P^* KG	$H_{V\infty}$ KG./mm ²	K_{IC} MPa $\cdot\text{m}^{1/2}$	Si Vol. %	Highest Strength MPa	Brittleness $H/K I_C^{1/2}$ (μm)
NC203(1)	8.3	0.5	2100	4.00(2)	0	825	5.11
α -SiC(78)(1)	6.8	0.4	2202	3.82	0	449	5.68
NC433(1)	3.9	0.3	1747	4.15	13-25(1)	383	4.13
331-2B	12.3	0.6	2072	4.72	11.51(3)	855	4.39
331-2A Top	9.0	0.4	1941	4.0	12.88(3)	899	4.85
Middle	10.0	0.4	1930	4.67	10.59(3)	803	4.13
Bottom	4.8	0.4	1909	4.40	11.26(3)	791	4.33
331-28A	10.1	0.5	1873	3.81	17.84(3)	596	4.93
331-28B (7/23-25/81)	12.0	0.6	1636	3.81	20.63(3)	538	4.29
331-28B (8/25/81)	5.1	0.2	1783	4.27	13.21(3)	557	4.18
331-27 (9/29-30/81)	10.5	0.6	1524	3.26	21.32(3)	501	4.69
331-34 (7/23-25/81)	5.4	0.2	1674	3.87	~45(3)	346	4.33
331-34 (7/30-31/81)	10.0	0.7	1824	3.21	16.12(3)	627	5.68
331-37	9.2	0.3	1826	3.90	15.48(3)	868	4.67
330-12	13.3	0.6	1928	4.59	8.85(3)	690	4.20
330-64	7.6	0.2	1681	3.91	16.58(3)	2	4.29

¹Samples studied in AMMRC TR-80-15; Ref. (9).²Constants set so $K_{IC} = 4.00 \text{ MPa}\cdot\text{m}^{1/2}$; Ref. (8).³Calculated from infiltrated density.

from a group. Included is the residual silicon vol% as calculated from the infiltrated density and the highest bending strength measured for the group. In addition, the brittleness, H/K_{IC} as defined in (5) is shown. A larger value signifies a more brittle material. Most of the materials measured are less brittle than NC203 by about 15%.

Several of the developmental materials show modest (~18%) improvements in toughness over NC203. The best results seem to be at low residual silicon content, i.e., less than 15 vol%. For all the materials studied the K_{IC} value lies within $\pm 20\%$ variation of that for NC203. However, the differences between samples are experimentally significant since in general the data indicate standard errors less than 5%. As pointed out previously this should not be interpreted as determining the absolute value of K_{IC} within 5% but merely comparative values for materials similar to NC203. However, a range of Si_3N_4 materials has been measured by the same techniques, and experimentors, and found to give the expected values for K_{IC} using the calibration data for NC203.¹¹

An effort has also been made to use the scatter in the crack length data for a given sample over a range of loads to indicate the structural uniformity of a material. Since the measuring error for c is in general much smaller than its variation, it is concluded that local microstructure causes much of the variation in crack length. Efforts to correlate data on this basis are incomplete at this time but will continue.

The parameters c^* and p^* show considerable variation among materials. However, at this time the significance is still uncertain since both c^* and p^* are inherently imprecise since they are determined from intercepts of two fitted lines.

Strength Testing. The strengths reported previously² were somewhat tentative since they were for the most part determined using a load cell on its most insensitive scale. In addition the loading rate was $4.2 \cdot 10^{-3}$ mm/sec which was somewhat faster than planned ($8.5 \cdot 10^{-4}$ mm/sec).

A new 4440 Newton load cell was obtained for use in the 4 point rupture testing. The strengths, sample sizes and spans used result in a breaking load of ~500 newtons which is conveniently determined with this cell. A group of 10 bars of 331-2B was tested with the new cell to compare with a group of 12 previously tested with the former cell. From an analysis of the results it was concluded that no significant errors were introduced by the old cell. However, the greater sensitivity of the smaller capacity cell warrants its use in all future testing.

It was also found that loading rate changes from $8.5 \cdot 10^{-5}$ mm/sec to $4.2 \cdot 10^{-3}$ mm/sec did not have a noticable affect on the breaking strengths. At room temperature loading rate variations in this range should not be very important, but all future testing is to be done with a load rate of $8.5 \cdot 10^{-4}$ mm/sec.

A summary of the room temperature strength determinations is given in Table III. In several cases samples of a given carbon skeleton type were siliconized with different times

TABLE III. SUMMARY OF ROOM TEMPERATURE STRENGTH PROPERTIES

MATERIAL	Density	Tests	H ₁₃	w	Avg.	Weibull Modulus, m	Weibull		Strength Control
							Char. Str., σ ₀ MPa	MPa	
NC203(1)	3.36	127	825	477	683	10(2)	610; 712(2)		Machining Defects
α-SiC(78)(1)	3.13	36	449	224	363	10.6		384	Large Grains
NC433(1)	3.06	37	383	169	317	14.9(2)		350(2)	Large Agglom.; Pores
331-2B Mixed (7/6-8/81)	3.10	10	760	329	510	4.4		558	Large Grains;
331-2B All	3.11	22	855	329	555	4.3		609	Large Si Lakes
331-2A Top	3.09	9	899	582	712	6.3(2)		764	
27 Middle	3.11	8	803	388	598	3.8		661	
Bottom	3.10	9	791	518	668	7.3		710	
331-2A All	3.10	26	899	388	662	5.7		714	
331-28A (9/24/81)	3.05	25	596	230	366	5.8		394	
331-28E (7/23-25/81)	3.02	9	538	298	405	5.3		434	
331-28B (8/25/81)	3.08	27	621	326	464	7.5		493	Surface Defects
331-27 (9/29-30/81)	3.01	8	501	310	432	5.0(2)		469	Large Si Lakes
331-34 (7/30-31/81)	3.06	11	627	352	485	5.3		526	
331-37	3.06	31	868	278	619	4.6		678	

Test Conditions: One third-4 point bending; spans - 0.95 cm; nominal volume under stress $1.6 \cdot 10^{-7} \text{ m}^3$; Nominal surface area under stress $3.10 \cdot 10^{-4} \text{ m}^2$; Ground Samples 600 grit finish.

¹samples and Results, Ref. (9).
²Poor Fit.

or temperatures. In some cases the variation was inadvertant due to differences between trays in the furnace chamber. The data for such separate processing has been grouped in batches and treated with Weibull statistics even though the number of tests for the subgroups is too small to rigorously justify such treatment. In fact even in the cases where greater than 25 tests were made the Weibull plots shown in Figs. 8-12 indicate that the results do not really fit a Weibull distribution. On these plots and in Table III the Weibull modulus, $\{$, is that determined from a leasts square fitting of the data. The resulting line is plotted on Figs. 8-12 and the Weibull characteristic strength, σ_0 , is determined from this line. With so few tests and the relatively poor fit to a Weibull curve the average strength is probably as reliable an indicator of performance as the characteristic strength.

Several points stand out in the results. First, the strength levels achieved are remarkably high for this type of material. With the exception of one batch of 331-34 all of the strengths are higher than comparable values for both reaction bonded Si (NC433) and sintered α -SiC (78). In one case, 331-2A, the strength slightly exceeds hot pressed SiC (NC203). Secondly, the Weibull moduli are significantly lower than the commercial materials. In Figs. 8-12 lines representing the results reported in 9 are plotted for comparison with three commercial materials NC203, NC433, and α -SiC. The first observation is quite encouraging while the second raises concern. However, it

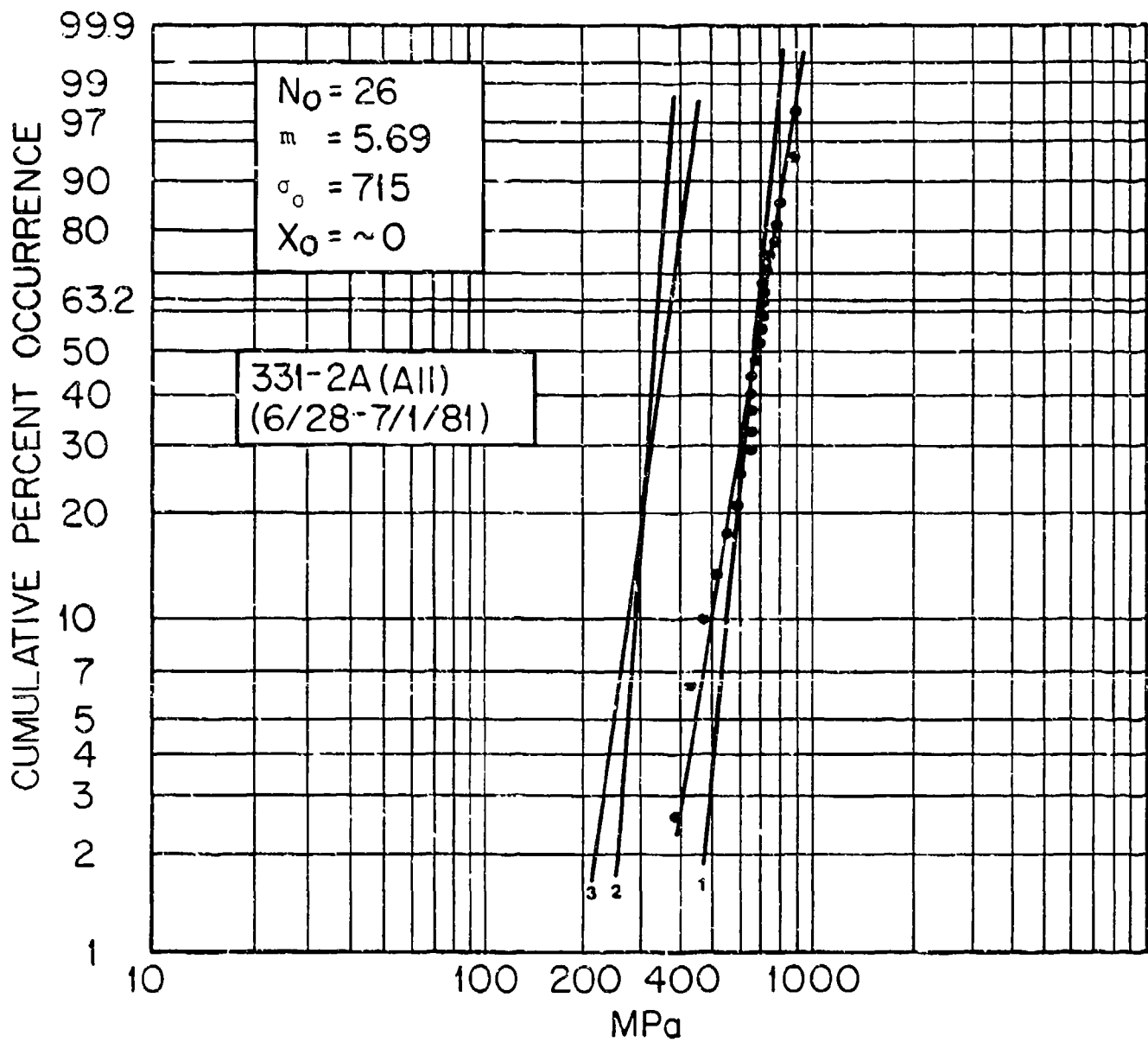


Figure 8. Weibull plot for 331-2A samples from three furnace trays in a single batch. Lines 1,2,3 are for NC203, NC433, and α -SiC as reported in Ref. 9.

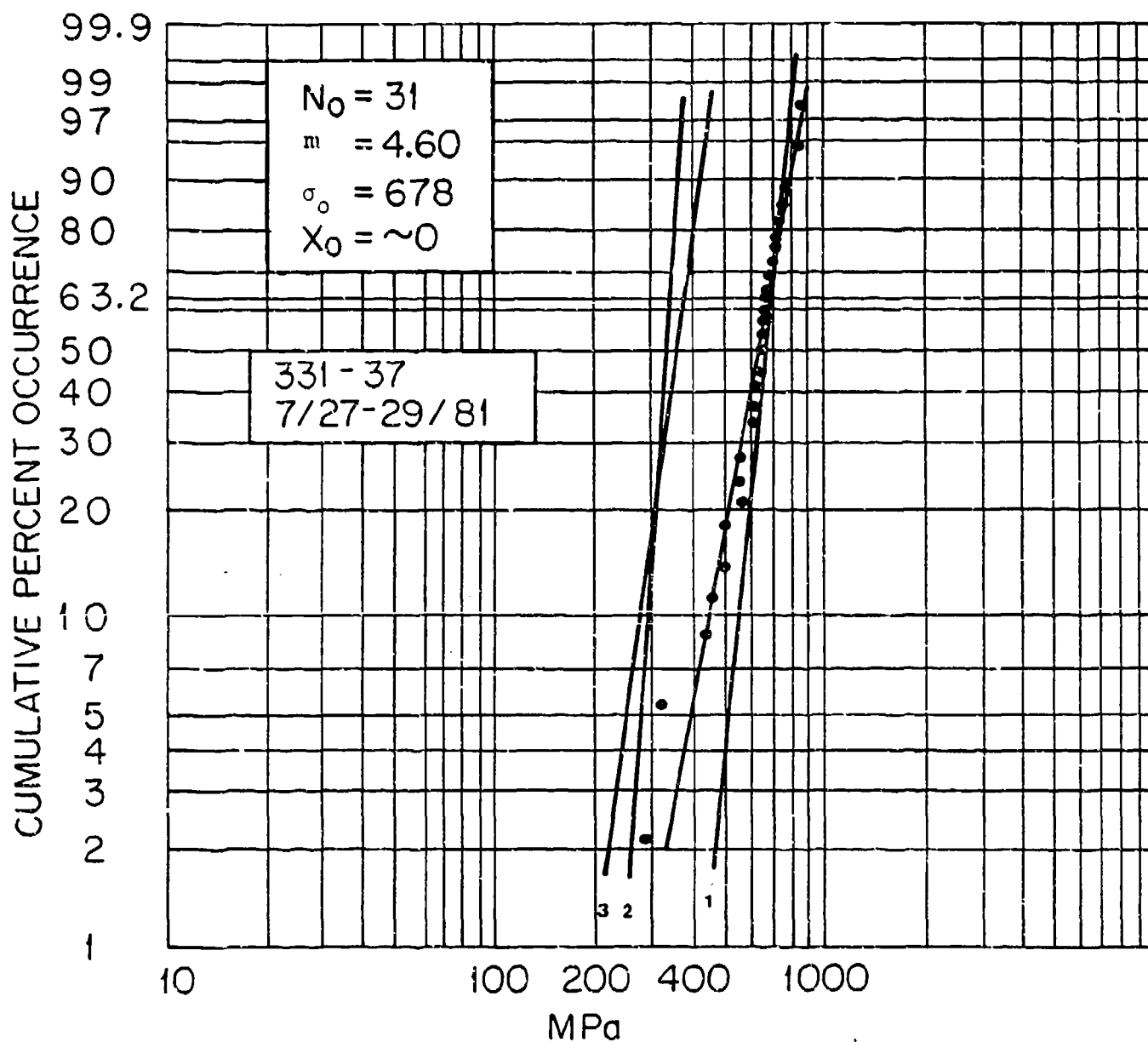


Figure 9. Weibull plot for a single batch of 331-37. Lines 1,2,3 are for NC203, NC433, and α -SiC as reported in Ref. 9.

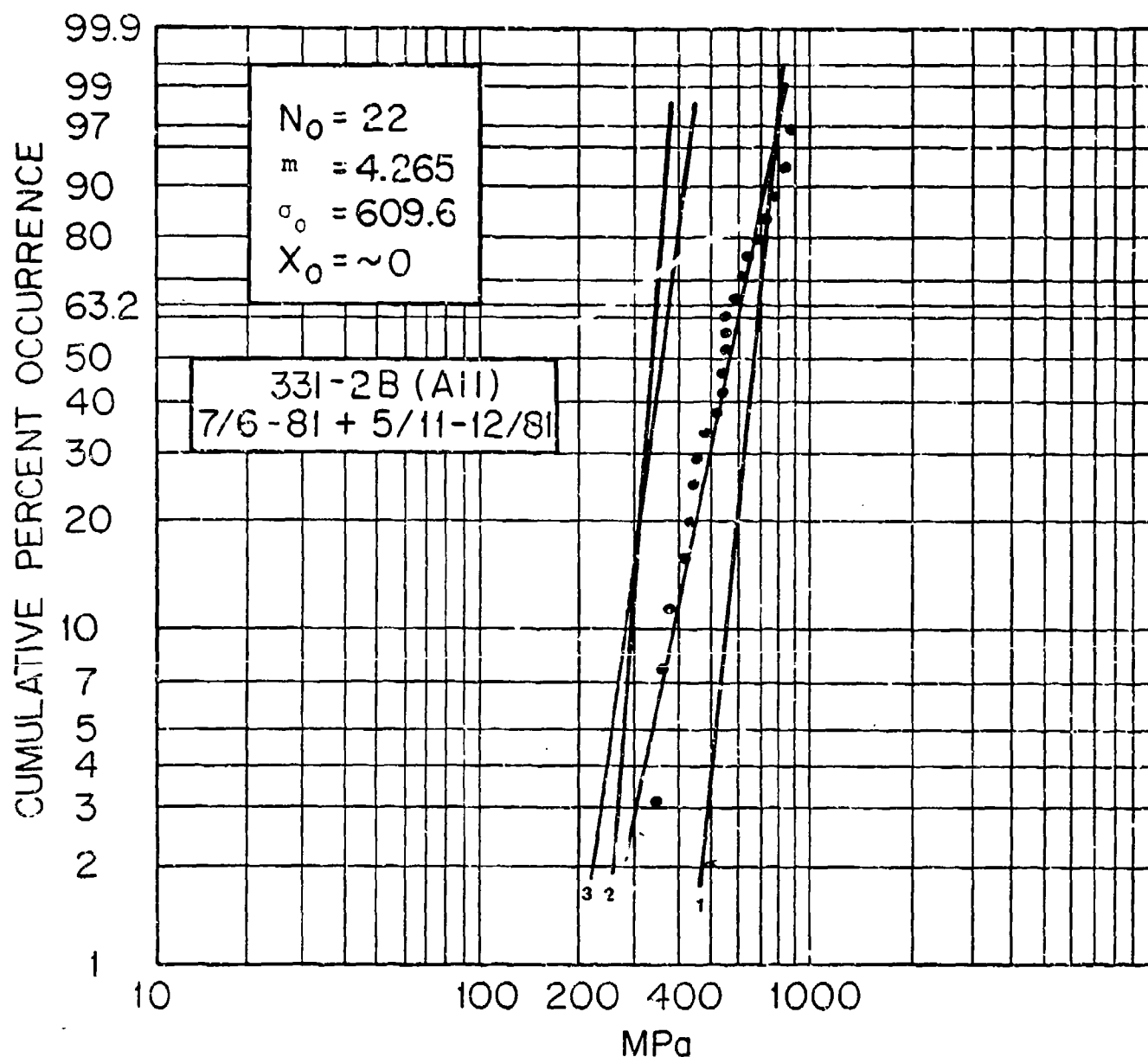


Figure 10. Weibull plot for 331-2B samples from various furnace trays in two different batches. Lines 1,2,3 are for NC203, NC433, and α -SiC as reported in Ref. 9.

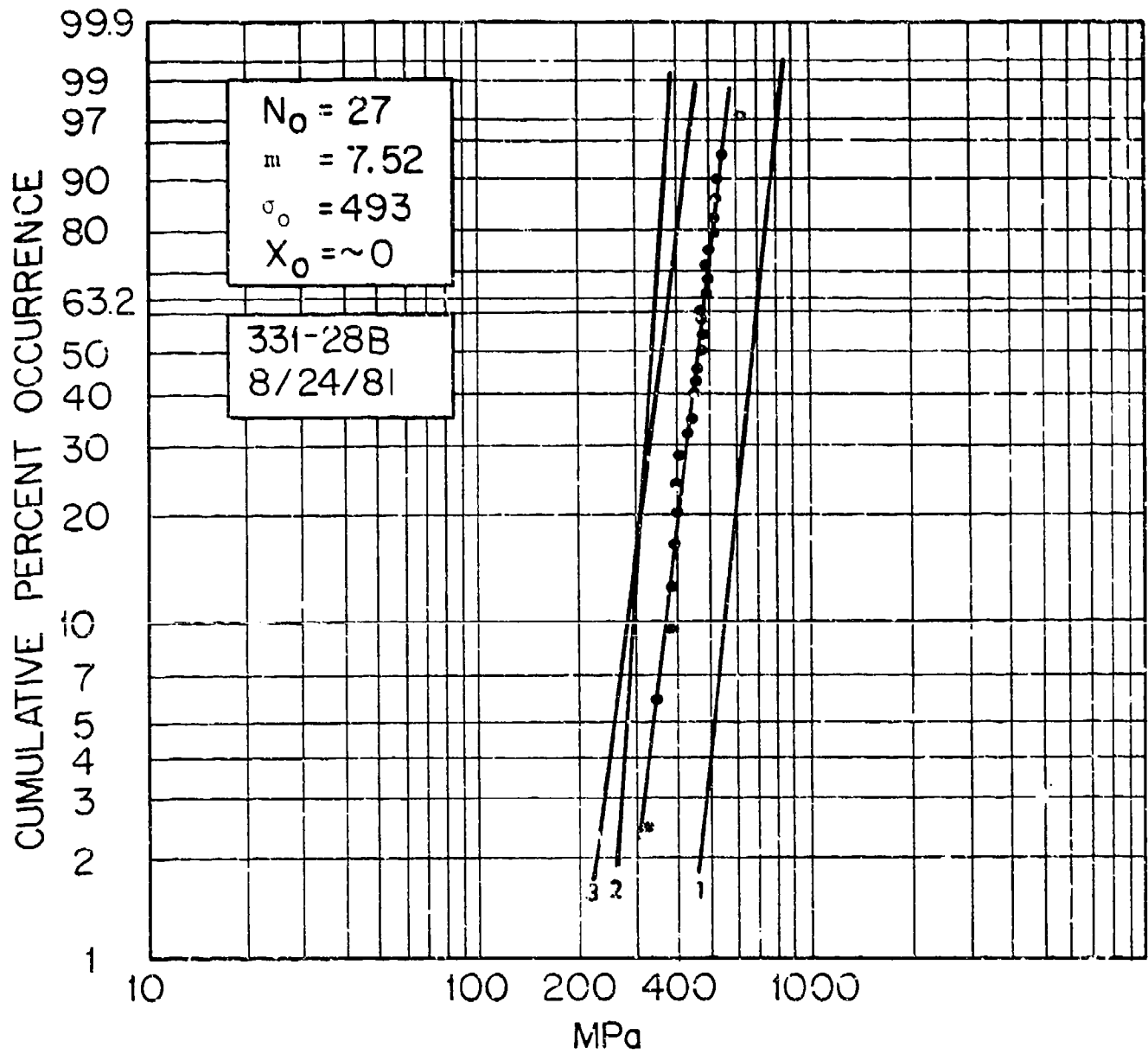


Figure 11. Weibull plot for 331-28B samples from various furnace trays in a single batch. Lines 1,2,3 are for NC203, NC433, and α -SiC as reported in Ref. 9.

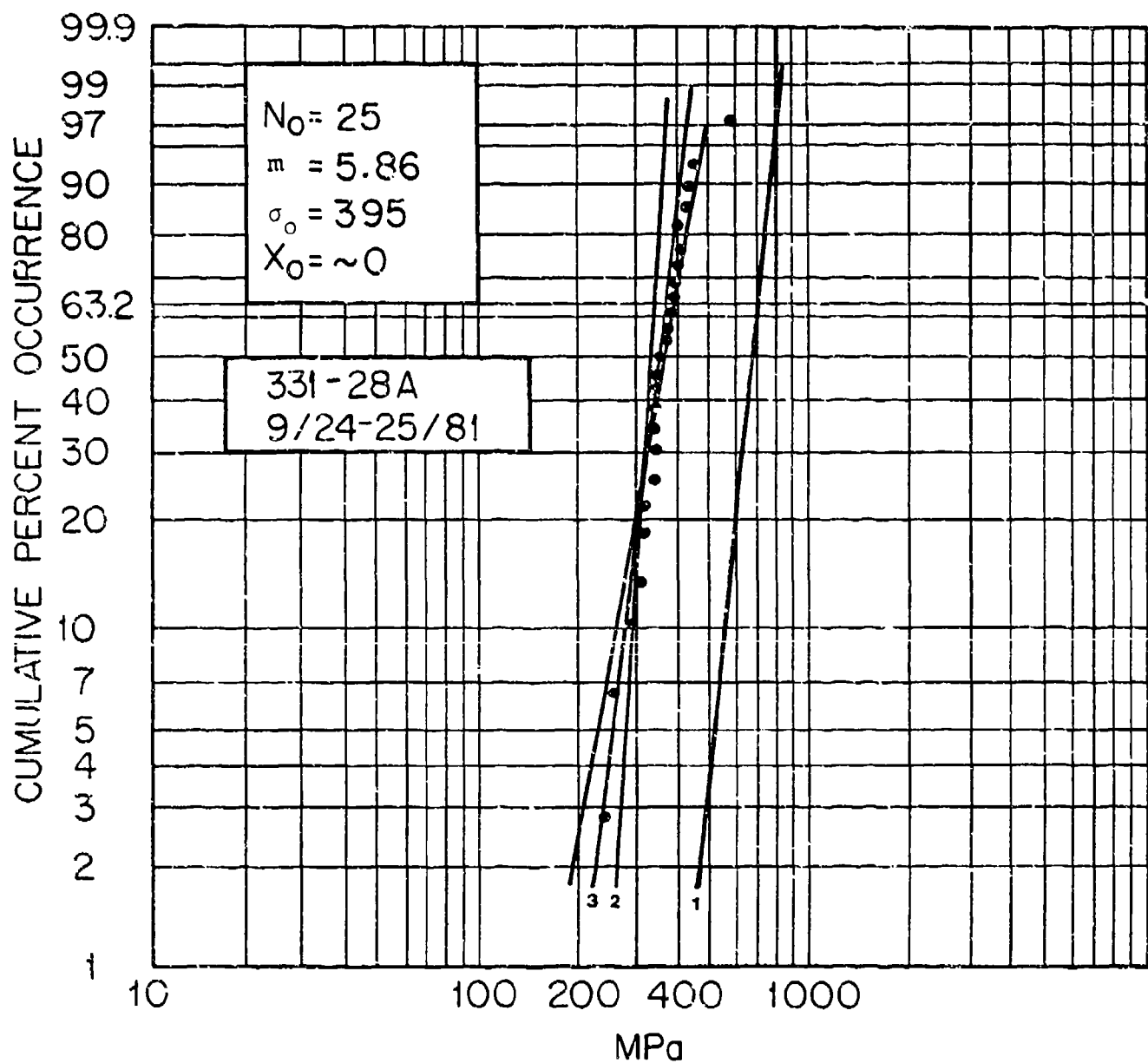


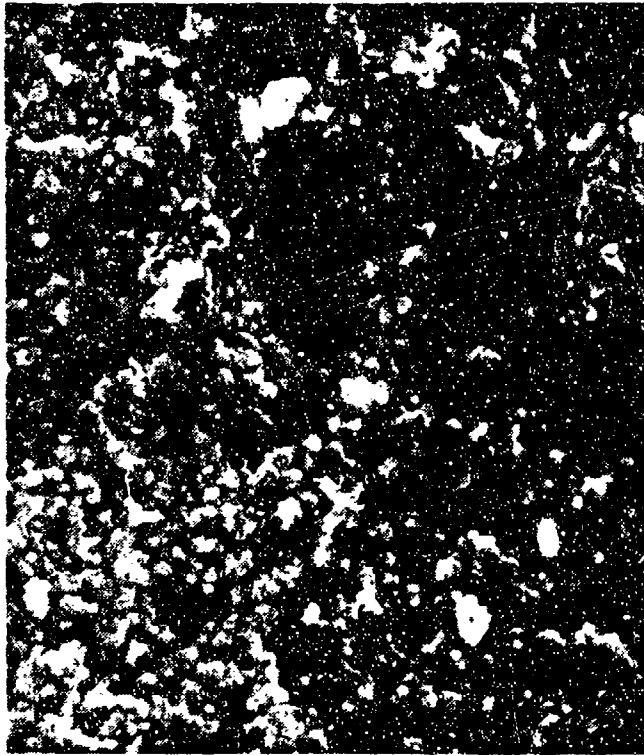
Figure 12. Weibull plot for 331-28A samples from several furnace trays in a single batch. Lines 1,2,3 are for NC203, NC433, and α -SiC as reported in Ref. 9.

should be noted that due to the higher strength levels that the developmental materials in almost all cases show minimum strength levels exceeding the Weibull characteristic strength for NC433. The typical structure of the strongest samples thus far tested, 331-2A, is shown in Fig. 13. These samples were quite uniform in structure.

All fractured bars from each batch were examined with a low power optical microscope while several bars from each batch including the strongest and weakest were examined with the scanning electron microscope. Particular scrutiny was given the very low strength bars since their strengths (~ 300 MPa) would indicate that the critical flaw was $\sim 125 \mu\text{m}$ ($K_{IC} \sim 4 \text{ MPa}\cdot\text{m}^{1/2}$). Flaws of this size should be relatively easily detected on the fracture surface. However for the strongest bars (~ 900 MPa) the indicated critical flaw size is about $14 \mu\text{m}$ which is very difficult to locate with certainty.

Figure 14 shows a SEM view of the fracture surface of the weakest (338 MPa) samples of 331-2A. It shows a small region that was not filled with Si. The size ($\sim 100 \mu\text{m}$) is about that expected from the very low strength of this sample.

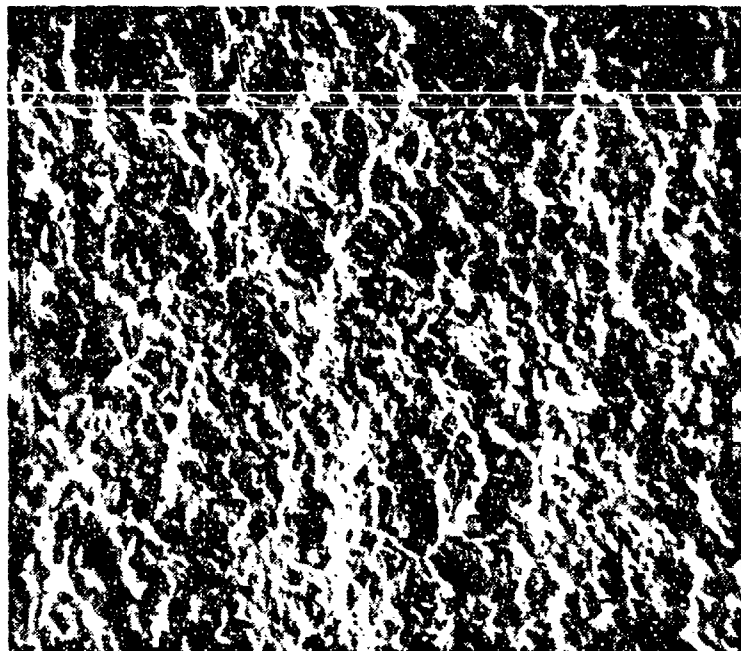
The fracture surface of one of the better samples of 331-2B (760 MPa) is shown in Fig. 15. The tension surface is at the top where the fracture surface is much coarser. This is believed to be a residual region of liquid coarsened SiC that was not completely ground away at this location. Optical examination was unable to confirm this since a polished cross section



|—|
10 microns

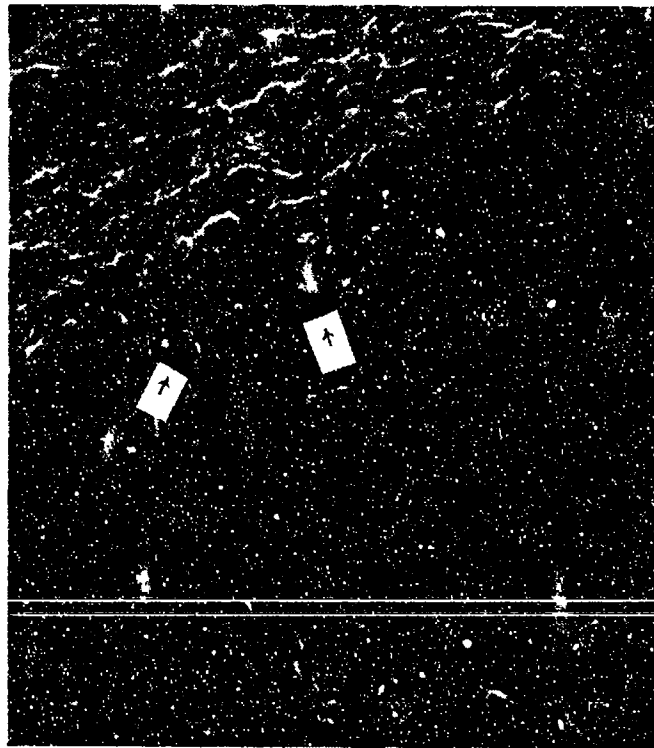


|—|
10 microns



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10 microns

Figure 13. Structures of 331-2A. Top Left: Carbon skeleton-SEM. Top Right: Siliconized-Optical. Bottom: Fracture Surface of strongest sample-SEM.



10 microns

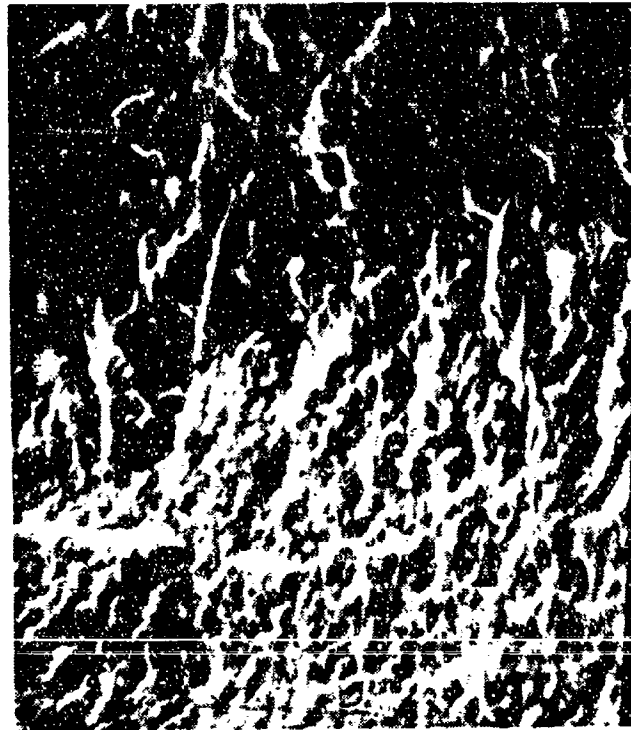
Figure 14. Fracture surface of weakest sample of 331-2A with rupture strength of 388 MPa. Tension surface at upper left. Defect area believed to be unfilled with Si.

could not be prepared at this cross section without removing considerable material. An example of this type of layer is shown optically for another sample (331-37) in Fig. 16.

Other examples of critical flaws were occasional chips on the machined surface and cracks associated with the chamfered edges. Typical examples are shown in Fig. 17.

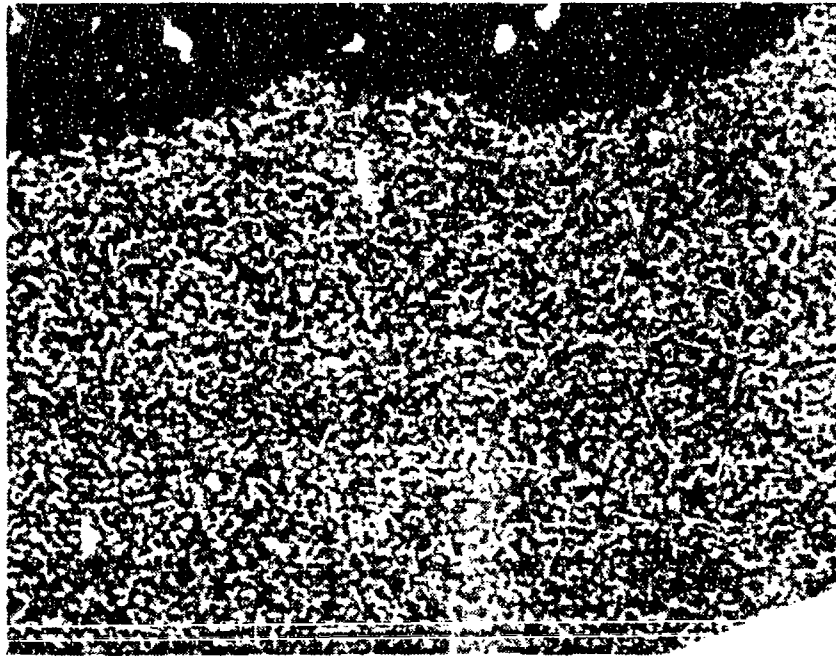
It is probable that at the present time the strength limiting factors for lower strength samples are associated with surface layers and machining and not with the bulk microstructural characteristics. This would indeed account for the relatively poor fit to a Weibull distribution and also for the low Weibull moduli.

Efforts have been made to prepare disks for a punch test². These efforts have been discontinued at the present time since the sample preparation costs have been much higher than originally anticipated.



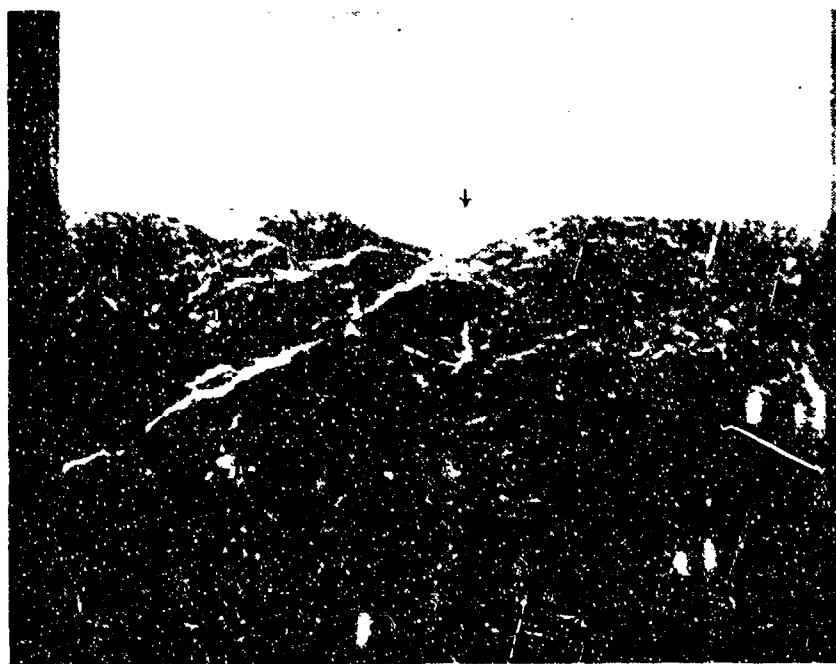
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Figure 15. Fracture surface of a sample of 331-2B with rupture strength of 760 MPa. Tension surface on top with coarse grained structure believed to result from a grain coarsened area not completely removed in grinding.

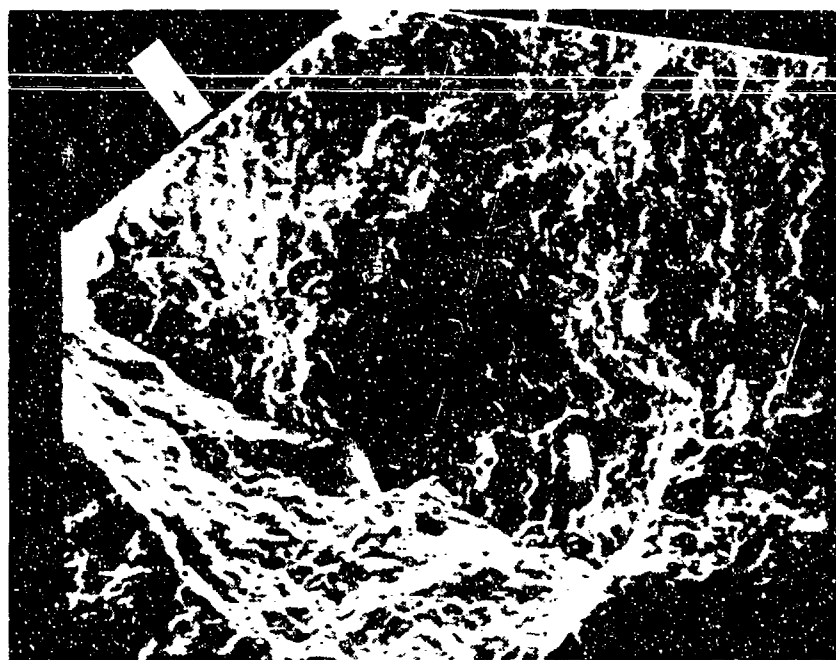


10 microns

Figure 16. Optical Micrograph of 331-37 with sample surface at top. A region of coarsened SiC shown at the top.



|—|
10 microns



|—|
10 microns

Figure 17. Top: Fracture surface of 331-37 showing chip on tension surface (681 MPa). Bottom: Fracture surface of 331-28B showing a fracture initiating at an edge chamfer crack (374 MPa).

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PROJECT DEVELOPMENT FOR SILICON CARBIDE
BASED STRUCTURAL CERAMICS -
Edward E. Burke, Materials & Metallurgical
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Ann Arbor, Michigan 48109

Technical Report AMRC TR 82-10, February 1982,
60 pp - Silicon, Contract DAAH4-80-C-0056
Interim Report 1 Jan 1981 to 31 Aug 1981

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Unlimited Distribution
Key Words
Silicon Carbide
Reaction Bonded Silicon Carbide
Structural Ceramics
Ceramics
Ceramic Processing

The objective of this program is to develop a process for making shaped silicon carbide based ceramic materials with reduced microstructural flaw size by in situ reaction of silicon with flame, ultra-ultra-pure carbon skeletons that are produced from liquid polymer solutions without particulate additions. Secondary objectives are: 1) delineation of the reaction section size, predictable while maintaining microstructural uniformity; 2) production of microstructures with characteristic features reduced from the current level of approximately 5-10 microns to the level of 1 micron or less; 3) characterization of microstructures, corresponding strength levels, and statistical uniformity; 4) delineation of dimensional tolerances and surface finish that can be held during processing without finished machining.

Very uniform and reproducible carbon skeletons have been produced and modified to provide for easy sintering. Complete carbon reaction during sintering without extensive grain coarsening or formation of silicon veins and/or laves has been achieved from 1 cm thick samples of carbon skeletons with particle size of 3.2 μ m and pore size of 1.9 μ m. The sintered material has a minimum grain size of 410 μ m and a well-defined characteristic four point bend strength of 840 MPa, which significantly exceeds other reaction bonded silicon carbide materials and compares favorably with hot pressed SiC (HC203) tested under similar circumstances.

Sections about 6 mm thick which a minimum grain size of 1 μ m have been prepared.

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Very uniform and reproducible carbon skeletons have been produced and modified to provide for easy sintering. Complete carbon reaction during sintering without extensive grain coarsening or formation of silicon veins and/or laves has been achieved from 1 cm thick samples of carbon skeletons with particle size of 3.2 μ m and pore size of 1.9 μ m. The sintered material has a minimum grain size of 410 μ m and a well-defined characteristic four point bend strength of 840 MPa, which significantly exceeds other reaction bonded silicon carbide materials and compares favorably with hot pressed SiC (HC203) tested under similar circumstances.

Sections about 6 mm thick which a minimum grain size of 1 μ m have been prepared.

Army Materials and Mechanics Research Center
Watertown, Massachusetts 02172
PROJECT DEVELOPMENT FOR SILICON CARBIDE
BASED STRUCTURAL CERAMICS -
Edward E. Burke, Materials & Metallurgical
Engineering, The University of Michigan,
Ann Arbor, Michigan 48109

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