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Processing and characterization of ceramic fiber reinforced porous matrix composites

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Processing and Characterization of Ceramic Fiber Reinforced Porous Matrix Composites

by

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ABSTRACT

The development of ceramic fiber reinforced ceramic matrix composites has been shown to be a promising approach for high toughness ceramic materials. The purpose of this work was to develop a new ceramic matrix composite(CMC) system based on a porous mullitic matrix reinforced with ceramic fibers. Unidirectional alligned fiber reinforced mullitic matrix composites were fabricated by a filament winding process. Prepregs were laminated and thermal processed in air. SEM fractographs of the composites were characterized and related to the observed load-deflection behaviors under 3-point flexure. The fracture surface expositions changed from extensive fiber tear-out to limited fiber pull-out as the firing temperature increased from 1250°C to 1450°C. Porosity of the current CMC system appeared to be less insensitive to the firing temperature than did the strength. Increase in strength as the firing temperature increased from 1150°C to 1250°C was considered to be the result of development of a stronger bond between fibers and matrix. However, strength of the composite decreased as the firing temperature increased from 1250°C to 1450°C due to the degradation of the reinforcing fibers. SEM fractography and load-deflection behavior of a Tyranno fiber reinforced porous mullitic matrix composite, fired at 1250°C, showed

characteristics of ceramic matrix composites toughened by matrix/fiber interaction mechanisms. Although the flexure strength of this CMC system was fairly low(178 MPa), the results provide a basis for further investigation of low to medium temperature systems, toughened by the tear-out mechanism.

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I. INTRODUCTION

Ceramic materials provide a number of attractive properties over metals and plastics. These properties include high strength, stiffness at very high temperatures, chemical inertness, and low density. The combination of these highly desirable properties makes them potentially useful for high temperature structural applications. However, ceramic materials are brittle and prone to catastrophic failure under applied loading. Several approaches for toughening ceramics have been proven[1] and led to the development of cermets, zirconia toughening, and fibrous toughening technologies. Among these methods, fiber or whisker reinforcement has been shown to provide maximum levels of fracture energy[2].

In fiber-reinforced ceramic matrix composites, high strength and high elastic modulus fibers are combined with a relatively weak matrix. This gives rise to mechanisms such as fiber debonding and pull-out which impede crack propagation and absorb energy imparting toughness to the materials.

Most methods of processing of fiber reinforced ceramix matrix composites often require high termperature processing to consolidate the matrix phase. Hot-pressing methods which are commonly used to densify ceramic matrix composites, are

expensive and limited to simple shape specimens. There are other difficulties accompanied with the high temperature processing of ceramic matrix composites. Commercially available ceramic fibers suffer from thermal or chemical degradation of their properties at high teemperature[3]. Also fibers without any coating are very likely to chemically react with the matrix materials and to form a strong bond between the fiber and the matrix. This will usually result in a catastrophic failure mode(brittle failure). The fiber/matrix interface is of critical importance in these materials. If there is neither chemical bonding nor frictional resistance between fiber and matrix, there will be no load sharing or property enhancement. If the fiber is either too strongly chemically bonded to the matrix or too strongly held by radial compressive stress (mechanical bonding) imparted by the matrix, an increase in strength will occur but the composite will fail abruptly without fiber pull-out.

Fiber coating methods have recently been successful in the improvement of ceramic matrix composites. The interface properties are adjusted by the coating materials[4], such as BN or carbon.

The criticial interface role can be greatly reduced through the use of porous matrices. Porous materials are usually weak having low elastic modulus and low shear

modulus characteristics[5]. If the reinforcing fibers are bonded to a porous matrix, then a weak "interface" situation will be established. When encountering a strong fiber, an advancing crack will deflect along the porous interface causing fiber debonding and fiber "tear-out" from the matrix, thus imparting fracture energy. This process, unique to those active in highly dense matrix CMC systems, is referred to as "porosity toughening"[6]. The object of this study was to fabricate fiber reinforced porous matrix composites and to characterize their mechanical properties. In the porous composites, the porous matrices function only as the bridging medium between the fibers. Most of the applied loading is carried by the fibers and the stength of the composite depends mainly on the performance of the fibers, while the toughness is provided by the "tear-out" of the fibers.

II. LITERATURE REVIEW

A. General Introduction:

The high toughness of fiber reinforced ceramic composite was first reported by Phillips and his co-workers in 1972 describing carbon fibers in glass and glass-ceramic matrices[7]. Since then, substantial advances have been made in the field of ceramic fiber composites. Recently, the development of continuous silicon carbide fiber has made these CMC composites more resistant to high temperature oxidation than the carbon fiber composites[8]. Prewo and Brennan[9,10] reported a study on silicon carbide fiber reinforced glass and glass-ceramic matrix composites showing that glass and glass-ceramic matrices may be strengthened and toughened with SiC fibers. However, not much success has been achieved with fully dense refractory ceramic composites due to the severity of hot-pressing or very high temperature sintering conditions which cause degradation of the fibers[11]. The method of CVD infiltration(CVI), which provides low temperature processing and avoids severe fiber degration, is an alternative route for fabricating ceramic matrix composites[12]. In the CVI process, reactant gas precursors are decomposed and deposited, at temperature less than 1200°C, within the fiber preform to form the matrix.

B. Theoretical background:

1. Strengthening:

Fiber reinforcement is usually employed to strengthen a low strength matrix by transferring the load to high modulus, high strength fibers. The longitundial tensile strength of a composite can be predicted by the rule of mixture as follows[13]:

$$\sigma_0 = \sigma_f V_f + \sigma_m(1-V_f) \qquad (1)$$

or

$$\sigma_0 = \sigma_m[1 + V_f(E_f/E_m - 1)] \qquad (2)$$

where E_m : elastic modulus of the matrix

σ_m : matrix strength

E_f : elastic modulus of the fiber

σ_f : fiber strength

V_f : volume fraction of fiber

σ_0 : composite strength

It is clear from eqn(1) that the strength of the composite is mainly controlled by the fiber when the strength of the fiber is much higher than that of matrix. Also, eqn(2) shows the effect of elastic modulus mismatch on the composite strength. For effective fiber strengthening to occur, E_f must be considerably greater than E_m. The interface bonding, either chemical or mechanical, is an important factor in effecting the strength of composite[14,15]. The interface functions to transfer load and strong bonding is essential

for a high strength composite. If bonding is too weak, the strength of the composite will be reduced. If no bonding exists, then the fibers behave as if they were merely bundles of fibers and no compositing can occur. The thermal expansion coefficient(CTE) mismatch between matrix and fiber may have a profound effect on the composite strength[2,16,17]. If the CTE of the fiber is moderately higher than that of the matrix, the matrix is placed in tangential compression and radial tension. The fiber is in radial tension, which may result in debonding of the fiber from the matrix and thus is detrimental to the composite strength. If CTE_f is lower than CTE_m, the matrix is placed in tangential tension and radial compression on cooling. If the mismatch is large enough ,these stresses may cause radial cracking of the matrix and a considerable decrease in composite strength.

2. Toughening Mechanisms:

Several concepts of energy-dissipating processes contributing to the work of fracture in ceramic matrix materials have been proposed[13,15,18]. These include (a) matrix microcracking, (b) fiber/matrix debonding, and (c) fiber pull-out

(a) microcrack toughening

When strong fibers are placed in a relatively weak matrix where a strong fiber/matrix bond exists or when $\alpha_m > \alpha_f$, matrix microcracking is induced in the stress field of an advancing crack and further penetration of the crack is retarded.

(b) fiber debonding and pull-out

When the fiber/matrix interface is weak or when $\alpha_f > \alpha_m$ which induces interfacial tension due to thermal stress, an advancing crack will deflect along the interface and thus cause debonding. When the applied loading reaches the maximum fracture stress of the fibers they will break at their weak points. Further load application is accompanied by fiber pull-out. Cottrel[19] showed that the work of fracture due to fiber pull-out is

$$\Gamma_{fp} = (l_c/l)(V_f^2 \sigma_f^2 r_f/12\tau_f) \qquad (3)$$

where Γ_{fp}: work of fracture due to fiber pull-out

l_c: critical fiber debonding length

V_f: volumn fraction of fiber

σ_f: tensile strength of fiber

r_f: radius of fiber

τ_f: interface shear strength

(c) Delamination

Delamination occurs in the form of splitting between the matrix and fiber/matrix interface. This occurs when the maxium shear stress at the crack tip exceeds the matrix ultimate shear stress. The maximum shear stress is defined as(20)

$$\tau_{max} = (2K_T/b)^{\frac{1}{2}} \qquad (4)$$

where K_t is egual to the matrix toughness, b is the crack opening displacement.

C. Interface Properties

1. The role of the fiber/matrix interface in the performance of ceramic matrix composites

A key processing issue for ceramic composites is the development of methods and materials to obtain an interface between the fiber and matrix such that toughness is obtained through mechanisms of crack deflection, debonding and fiber pull-out. If there is a strong fiber/matrix interface bond, an advancing crack through the matrix will extend on through the fibers resulting in brittle failure of the composite. If the interface bonding is appropriately weak, crack deflection causes debonding of the fiber from the matrix to occur producing a tough composite.

2. fiber coating and its effect on the performance of ceramic matrix composites

Ceramic fibers are likely to react with matrix materials, especially those of oxide matrices during high temperature processing. Fiber coatings provide a diffusion barrier to inhibit the fiber from reaction with the matrix as well as to prevent oxidation. R.W.Rice et.al have published studies[4,20] on BN coating of fibers in various matrices. Results showed that both strength and toughness can be improved; however, BN will oxidize to B_2O_3 at elevated temperature in an oxidizing atmosphere, resulting in the development of a glassy layer bonded to the matrix.

D. The role of raw materials

(1) Nicalon fiber

There have been intensive investigations of Nicalon fiber reinforced ceramic matrix composites[9,10]. Nicalon fiber developed by Yajima et.al[8] is based on a SiC composition that was initially obtained by thermal decomposition of a polymer precursor. Because of the production method, Nicalon fiber contains some impurities, such as SiO_2 and free carbon, which are responsible for severe strength degradation and poor creep resistance at temperature above 1000°C[21].

(2) Tyranno fiber

Tyranno fiber is a new SiC type fiber containing 0.5-5 wt% TiO_2 that has been developed by UBE Industrial Ltd. Tyranno fibers are characterized by a noncrystalline structure and strength characteristics that are stable up to $1300^{o}C$[21].

(3) Nextel fiber

Nextel fibers based on mullitic compositions were developed by 3M Co. using the polymer precursor method. Because of their oxide chemistry, Nextel fibers are relatively stable in an oxidizing atmosphere. However, Nextel fiber will recrystallize and lose strength at temperatures above $1000^{\circ}C$[23].

Table I

Properties of three commercial ceramic fibers

Name	Tyranno	Nicalon	Nextel 440
Composition	25-33 C	30.0 C	70 Al_2O_3
	.1-.2 N	11.8 O	28 SiO_2
	15-20 O	54.3 C	2 B_2O_3
	45-53 Si		
	.5-5 Ti		
Diameter(μm)	8-10	10-20	10-12
Cross Section	round	round	oval
Density(g/cm3)	2.3-2.5	2.56	3.10
C.T.E(10^{-6}/°C)	3.1	3.1	4.38
range(°C)	0-500	NL	25-1000
Elastic modulus(GPa)	190	180-200	200-240
Ultimate Tensile Strength(GPa)	2.8	2.5-3.2	1.4-2.1
Ultimate Strain to failure %	1.4-1.7	1.5	NL
Manufacturer	Sumitimo	Dow Coring	3M

(b) Mullitic matrix materials

Usually, the selection of the matrix materials depends more or less on the eventual applications of the composites. For high temperature structural applications, the desired properties include: refractoriness, high temperature strength and creep resistance, toughness, and compatibility with the fiber. In this study, the matrix only functions as a bridge between the fibers, and the requirements for the matrix are much less critical. A mullitic matrix from a clay precursor was selected in this study for its low cost and potential to bond to the fibre.

Kaolinitic clay with the composition $Al_2O_3 2SiO_2 \cdot 2H_2O$ provides a very important source of mullite for the refractory industries. The dynamic thermal decomposition sequence of clay materials has been extensively investigated[24-26]. On heating, clay will dehydroxylized to metakaolinite at about 500^0C. Further heating to 980^0C results in separation of amorphous silica from aluminosilicate, which is accompanied by a prominent exothermic reaction. In a recent study[27], the 980°C exotherm is concluded to be caused by the formation of a spinel phase. The second exothermic peak observed at a

temperature around 1250°C is correlated to the conversion of the spinel component to mullite. Further heating cause mullite growth and crystallization of free silica.

III. Experimental Procedure

A. Raw materials characterization

1. DTA and TGA analysis

The thermal behavior of kaolinitic clay and fibers was characterized by DTA and TGA analysis. Samples were heated to 1450°C at a heating rate of 10°C/min in air.

2. Microstructural characterization

The characteristics of the fibers after heating were also examined by Scanning Electron Microscopy(SEM). Fiber yarns were exposed at 600^{0}C and 1300^{0}C in air for 4 hours before examination.

B. Composite fabrication

1. Infiltration method

This is one of the first known attempts to produce porous matrix composites through the combination of alligned fiber with mullitic matrix materials. Nicalon fiber, after the sizing was burned off, were cut into lengths of about 2" and manually stacked into a steel die. A slurry consisting of 20% of clay powder and 80% of binder solution was poured into the die. The whole fixture was vibrated on a small vibration table to assist distribution of matrix in the fiber preform. This method was not ultimately sucessful because some fraction of the yarns always remained

unimpregnated.

2. Filament Winding

To prepare the slurry for filament winding, all constituent materials were carefully weighed and then mixed by wet milling. The PH value of the slurry was adjusted to about 9.5. The fiber tows, after the sizing was removed, were drawn through the slurry and wound on a mandrel to form unidirectional layer prepregs. After being dried prepregs were laminated by stacking into a steel die and pressed at 750 psi. The green laminated composites were then sintered in air at the desired temperatures for 4 hours.

C. Composite characterization

All samples were subjected to the same characterization procedures.

1. Physical property measurement

After firing, the samples were weighed and measured for density and porosity. The open porosity of the fired specimens was measured by using the method of water replacement according to ASTM 20-83. The density was measured by dividing the weight by the volume of the specimen.

2. Strength measurement

Specimens were tested in three point bending employing an mechanical test unit (Model TSM-10, Instron Co.) Three-point flexure testing was performed at room

temperature, the crosshead speed was maintained at 0.02"/min for all tests, and a span to depth ratio(S/d)≥20 of the specimens was chosen to minimize the tendency of shear failure. The load-deflection curves were automatically recorded on a chart recorder. The modulus of rupture(MOR) was calculated on the assumption that the materials behave elastically and the peak tensile stress at the ultimate load is $M.O.R.= 1.5(LP/bh^2)$

where L equals the testing span, P is the applied loading, and b, d are the specimen width and depth respectively.

Qualitative estimations regarding toughness were inferred from the nature of the load-deflection curves obtained in the flexure test.

4. Microstructure development

Because of porous nature of the composites, specimens were very easily damaged during sectioning. To minimize damage, samples were impregnated with epoxy in a vacuum system prior to sectioning. Samples were sectioned by using a diamond blade and then mounted in epoxy resin. Coarse grinding and fine polishing were performed using silicon carbide grinding paper and diamond paste respectively. The microstructures of the fracture surfaces and polished sections were examined by Scanning Electron Microscopy(SEM) and Optical Microscopy.

IV. RESULTS AND DISCUSSION

A. Characterizations of fibers

The results of the DTA and TGA analyses of ceramic fibers-Nicalon, Tyranno, and Nextel 440 are represented in fig.1-3. Both the Nicalon and Tyranno fibers, which are SiC type fibers, showed an exothermic reaction at around 1100°C. This reaction, accompanied by a weight gain, was assumed to be the oxidation of SiC to form SiO_2. The oxidation of SiC type fibers was also observed by Scanning Electron Microscopy(SEM) examination. When the fiber yarns were thermal exposed at 600°C in air for 4 hours, the surfaces of these fibers remained smooth and no oxidation layers were observed. However, when fiber yarns were exposed at 1300°C, Tyranno fiber showed an oxidation layer of about 2μm thickness and Nicalon fiber, in contrast, exhibited a low degree of oxidation (Fig.4,5). Fig.7 shows cracking of the oxidation layer of Tyranno fiber probably due to the difference in coefficients of thermal expansion between the oxide and fiber. Nextel fibers, because of their oxide composition, are very stable in air and SEM photography showed that no distinction can be made between fibers exposed at 600°C and 1300°C (Fig.6).

B. Composite fabrication:

Filament winding provided the most successful route for the production of alligned continuous fiber reinforced composites. Fig.8 represents a typical cross section of a unidirectional fiber composite, The fiber volume content is estimated to be about 50%. Fig.9a and Fig.9b shows the surface of the specimen before flexure testing and the surface of failed 3-point bend specimen respectively. Fig. 10 represents typical microstructures of the porous matrices after various firing temperatures

C. Mechanical testing results

The specimens used for mechanical testing were composed of unidirectional alligned fiber and thus were highly anisotropic. When testing these systems and analyzing results, two points have to be taken into consideration. First, unlike a monolithic ceramic, the compressive strength of the ceramic matrix composite may be similar to or less than the tensile strength. Secondly, the shear strengths between planes parallel to the fibers are generally very much less than the tensile or compressive strengths[22]. The maximum tensile stress at the surface under 3-point flexure is given to a first approximation by

$$\sigma = 1.5Fl/bd^2$$

and the maximum shear stress occuring on the neutral plane

is

$$\tau = 3F/4bd$$

To an approximation, tension or compression failure will occur at $l/d > 20$ and shear failure at $l/d < 5$[22].

Test results of individual prepreg and of laminated composites

The load bearing behavior of individual prepregs was measured by 3-point flexure testing and results are presented in Fig. 10.

Fig.11 shows the relation between flexural strength and firing temperature of the composites. Flexture testing of both the individual prepreg and laminated composite showed the same trend in the relation between flexture strength and firing temperature,ie, at firing temperatures below 1250°C, strength increased as firing temperature increased. At temperatures above 1250°C, the strength decreased as firing temperature increased.

Generally, as firing temperature increases, the reaction between fiber and matrix becomes more pronounced and stronger bonding is likely to occur. A strong interface bond is usually more efficient for load transference. However, as firing temperature increases, the strength and elastic modulus of fibers decrease and the overall strength of the composite will be reduced due to the reduced load sharing by the fibers.

D. Analysis of stress-strain curves and microstructure development

Three types of stress-strain behavior were observed during this study.

Fig.13 represents the first type of stress-strain behavior, in which the strength, after reaching a maximum value, decreases gradually. SEM fractography showed that the fracture surface exhibited extensive fiber pull-out (Fig.16). Higher magnification examination (Fig. 17) of the pulled out fibers displays some free matrix materials remained attached to the fibers. This constributes evidence of a certain degree of bonding between the fiber and matrix and supports a fiber "tear-out" mechanism of toughening.

Fig. 14 represents the second type of stress-strain behavior, which is characterized by a jagged curve. Examination of SEM fractography (Fig. 18) showed that the fracture surface is made up of matrix plus fiber protrusions in addition to individual fiber pull-outs. This type of fracture has been reported by K. M. Prewo[27]. Higher magnification examination (Fig. 19) of the fractured surface displayed some delamination cracking along the fiber/matrix interface (Fig.19).

Fig.15 represents the third type of stress-strain behavior in which the strength dropped immediately after

reaching its maximum value. Examination of SEM fractography revealed evidences of fiber debonding but very little fiber pull-out (Fig.20)

E. Results of Porosity Toughening

As mentioned, the mechanism of porosity toughening in ceramic matrix composite is characterized by a fiber tear-out mode derived from interface bonding between the fiber and the porous matrix. Fig. 13 presents a typical load-deflection curve of a porosity toughened ceramic matrix composite. Visual exhibitions of fiber tear-out are represented in Fig. 16 and Fig. 17. In contrast to traditional fiber pull-out which is displayed as a very clean pulled-out fiber surface, the tear-out fibers of porosity toughened ceramic matrix composite show fiber surface with some adherent matrix materials.

Adequate fiber strength is required for the porosity toughening to occur. As the strength of the fiber decreases due to thermal degradation, cracks are likely to propagate through the fibers and fiber tear-out will not occur(Fig. 18).

V. CONCLUSION

(1) A ceramic matrix composite(CMC), investigated during this study exhibited characteristics of moderate strength and high toughness when tested in flexure.

(2) The predominant failure modes in this porous matrix composite system are deduced to be delamination cracking and fiber bundle failure.

(3) Fiber pull-out from the surrounding matrix is apparently prevented due to a sudden local decrease in fibre strength or an increase in fiber/matrix interfacial strength.

(4) A low strength shear path may provide the achievement of some crack growth resistance through low fiber/matrix interfacial strength.

(5) A Tyranno fiber reinforced porous matrix composite, which was sintered at 1250°C for 4 hours, yielded a composite with a flexure strength of 178 MPa and high toughness.

(6) A porosoity toughening mechanism with fiber tear-out mode characteristics was demonstrated by SEM fractography as well as by the nature of the load-deflection curve of a Tyranno fiber reinforced mullitic matrix composite.

VI. REFERENCES

1. Wiederhorn,S.M.,"Brittle Fracture and Toughening Mechanisms in Ceramics", Ann. Rev. Mat. Sci., 14, 1984, pp. 373-403
2. R. W. Davidge, "Fibre-reinforced ceramics", Composites, Vol.18, No. 2, April 1987, pp.92-98 1429-1437.
3. T. Mah, M. D. Mendiratta, A. P. Katz and K. S. Mazdiyasni "Recent Development in Fiber-Reinforced High Temperature Ceramic Composites", American Ceramic Society Bulletin, 66 [2] 304-308 (1987)
4. R. W. Rice, J. R. Span, D. Lewis III, and W. S. Coblenz, "The Effect of Ceramic Fiber Coating on the Room Temperature Mechanical Behavior of Ceramic Fiber Composites", Metal Matrix, Carbon and Ceramic Matrix Composites", 1984, 217-231
5. W. D. Kingery, H. K. Bowen and D. R. Uhlmann, "Introduction to Ceramics, 2nd Edition", John Wiley and Sons, New York 1976.
6. Dr. Michael Millard, Personal communication.
7. D. C. Phillips, "The Fracture Energy of Carbon-Fibre Reinforced Glass", Journal of Materials Science, [7], 1175-1191 (1972).

7. S. Yajima, J. Hayashi, M. Omori, and K. Okamura, "Development of a Silicon Carbide Fiber with High Tensile Strength", Nature [261], June 24, 1976, 683-685.
8. K. M. Prewo and J. J. Brennan, "Silicon Carbide Yarn Reinforced Glass Matrix Composites", Journal of Materials Science,[17] 1201-1206 (1982).
9. J. J. Brennan and K. M. Prewo, "Silicon Carbide Fiber Reinforced Glass-Ceramic Matrix Composites Exhibiting High Strength and Toughness", Journal of Materials Science, [17] 2371-2383 (1982).
10. R. Wills, M. Pascucci and F. Jelinek, "Ceramic-Ceramic Composite, State-of-the-Art Report", MCIC 86-51, Battle Columbus Division, January 1986.
11. Naslain, R., et al., "An Analysis of Properties of Some Ceramic-Ceramic Composite Materials Obtained by CVI-Densification of 2D C-C preforms", The Metallurgical Society, PP. 499-514, 1985.
12. K. K. Chawla, "Composite Materials", Springer-Verlag New York Berlin Heidelberg.
13. B. D. Agarwal and L. J. Broutman, "Analysis and Performance of Fiber Composites", John Wiley and Sons, New York 1980.
14. R. W. Davidge, "Mechanical Behavior of Ceramics", Cambridge University Press, 1979, 11-117.

15. I. W. Donald and P. W. McMillan, "Review: Ceramic-Matrix Composites", Journal of Materials Science, [11] 949-972 (1976).

16. R. Pampauch, W. Slomka, and J. Chlopek, "Determination of the Influence of Matrix-Ceramic Fibre Reactions on the Strength of Composites", Ceramics International, [12] 9-18 (1986).

17. J. K. Wells and P. W. R. Beaumont, "Debonding and Pull-out Processes in Fibrous Composites", Journal of Materials Science, [20] 1275-1284 (1985).

18. A. H. Cottrell, "Strong Solids", Proc. Roy. Soc.., A282, pp. 2-9,1964.

19. O. Sbaizero and A. G. Evans, "Tensile and Shear Properties of Laminated Ceramic Matrix Composites", Jounal of American Ceramic Society, 69[6], 481-86(1986).

20. D. Lewis and R. W. Rice, "Further Assessment of Ceramic Fiber Coating on Ceramic Fiber Composites", Metal Matrix, Carbon and Ceramic Matrix Composites", 1985, 13-16.

21. G. Simon and A. R. Bunsell, "Mechanical and Structural Characterization of Nicalon Silicon Carbide Fibre", Journal of Materials Science, [19] 3649-3657 (1984).

22. D. B. Fischbach, P. M. Lemoine, G. V. Yen, "Mechanical Properties and Structure of a New Commercial SiC-Type Fibre(Tyranno)", Journal of Materials Science, [23] 987-993 (1988).

23. D. D. Johnson, A. R. Holtz, and M. F. Gunther, "Properties of Nextel 480 Fiber", Presented at the 11th Annual Conference on Composites and Advanced Ceramic Materials, Cocoa Beach, FL, January 18-23, 1987.

24. G. W. Brindley and M. Nakahira, "The Kaolinite-Mullite Reaction Series: I, A Survey of Outstanding Problems", Journal of American Ceramic Society, July, 1, 1959, pp. 311-314.

25. G. W. Brindley and M. Nakahira, "The Kaolinite-Mullite Reaction Series: II, Metakaolin", ibid., pp. 314-318.

26. G. W. Brindley and M. Nakahira, "The Kaolinite-Mullite Reaction Series: III, The High-Temperature Phases", ibid., pp. 319-324.

27. B. Sonuparlak, M. Sarikaya, and I. A. Aksay, "Spinel Phase Formation During the 980°C Exothermic Reaction in the Kaolinite-to-Mullite Reaction Series", Journal of American Ceramic Society, Vol. 70, No. 11, pp.837-842, 1987.

28. D. C. Phillips and R. W. Davidge, "Test Techniques for the Mechanical Properties of Ceramic Matrix Fibre Composites", Britich Ceramic Society Transactions and Journal, 85, 1986, pp. 123-130.

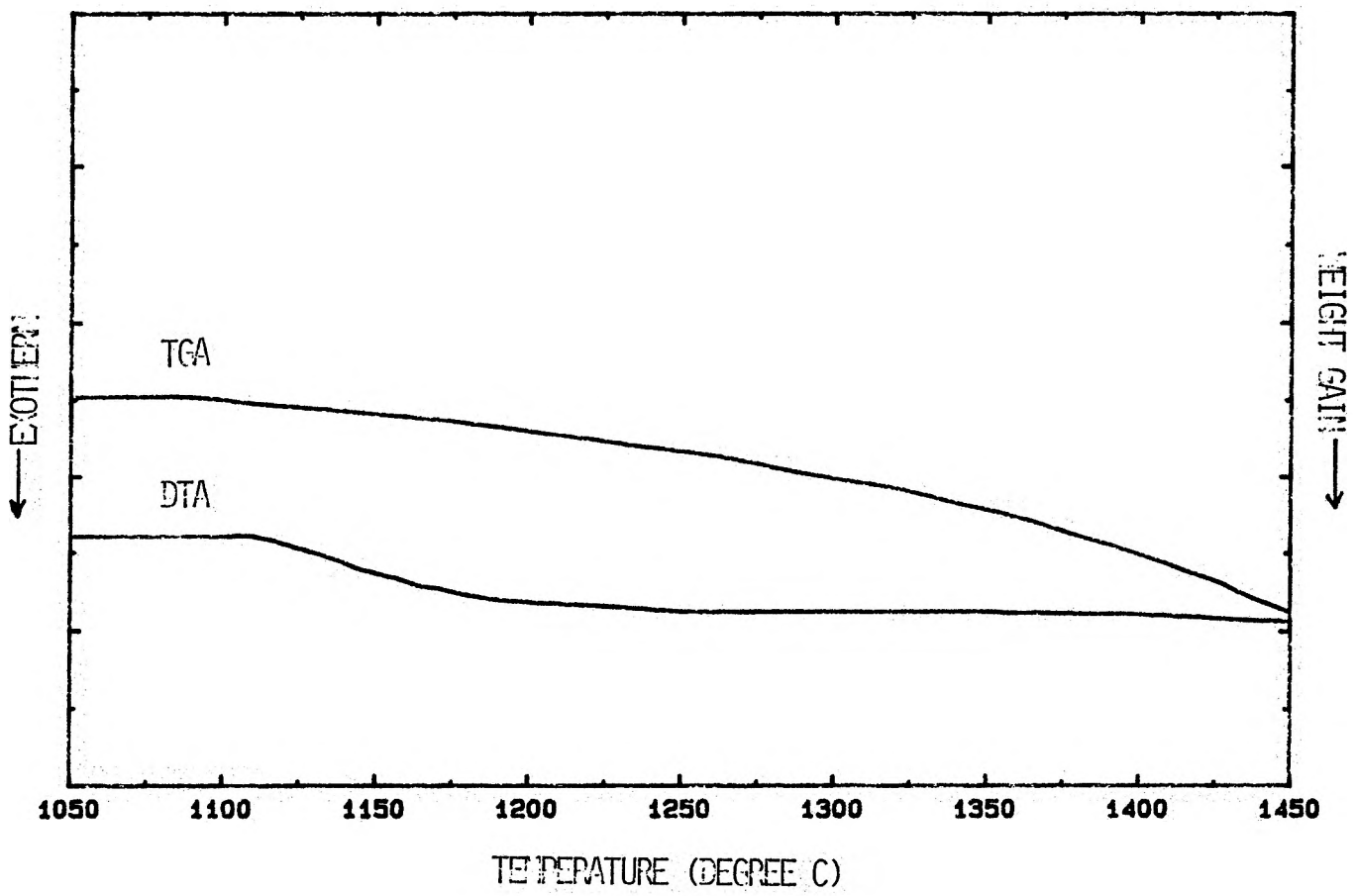


Fig.1 DTA and TGA CURVE of TYRANO FIBER.

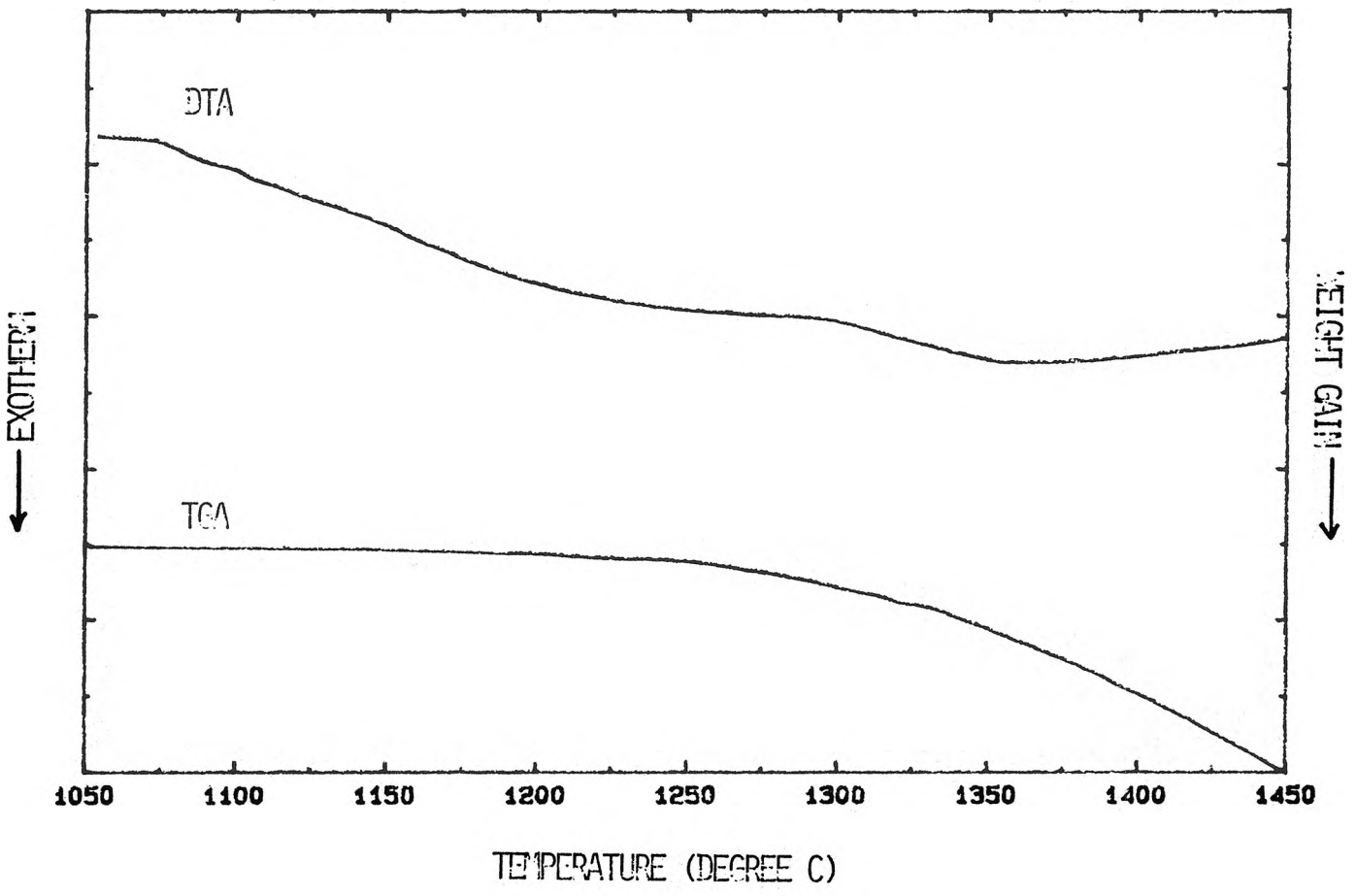


Fig.2 DTA and TGA CURVE of NICALON FIBER.

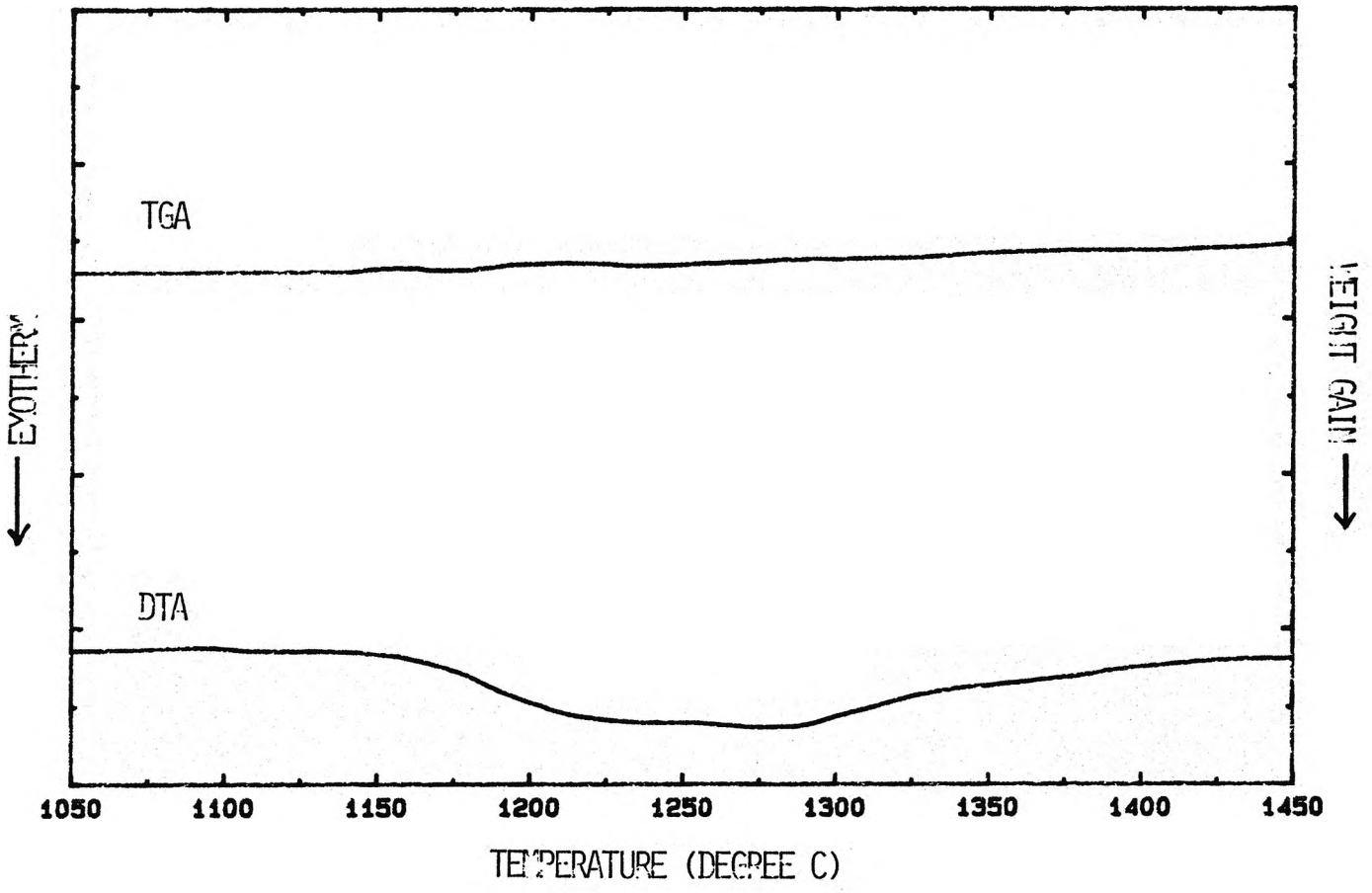


Fig.3 DTA and TGA CURVE of NEXTEL 440 FIBER.

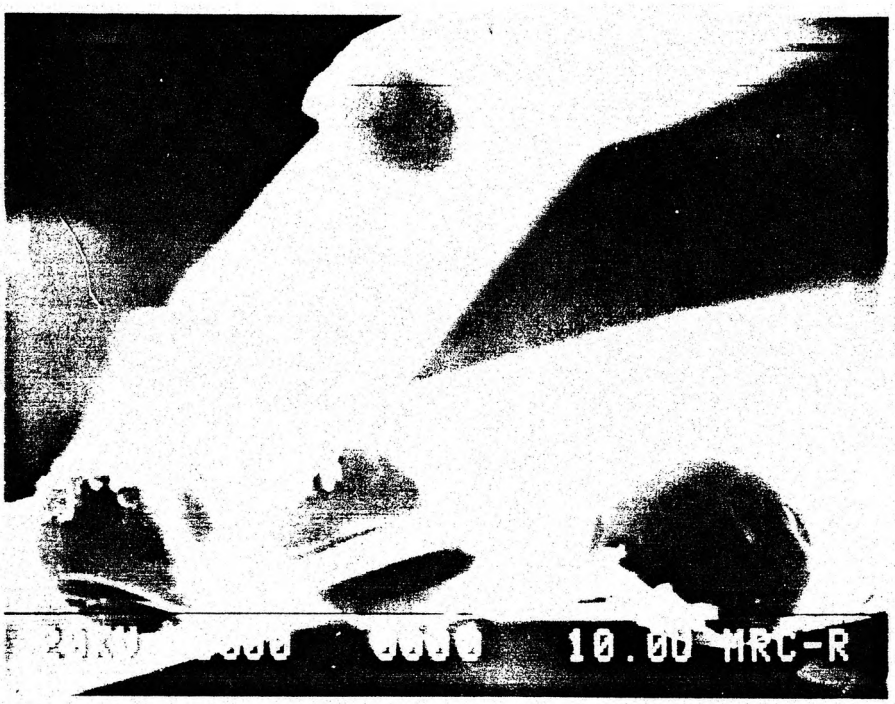


Fig.4a Tyranno fiber exposed at 600°C in air

Fig4b. Tyranno fiber exposed at 1300°C in air

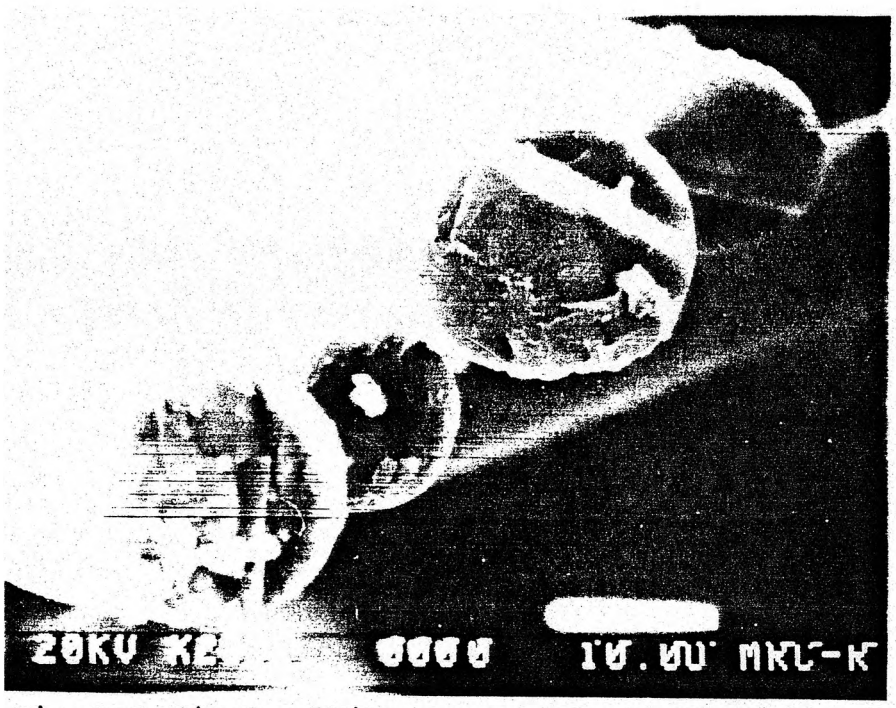


Fig.5a Nicalon fiber exposed at 600°C in air

Fig.5b Nicalon fiber exposed at 1300°C in air

Fig6a. Nextel fiber exposed at 600°C in air

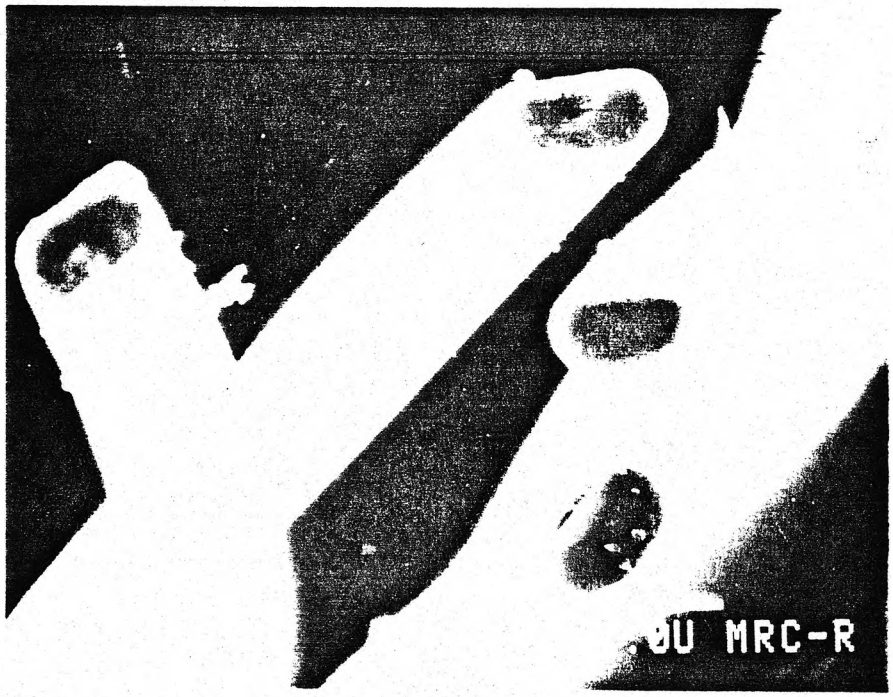


Fig6b. Nextel fiber exposed at 1300°C in air

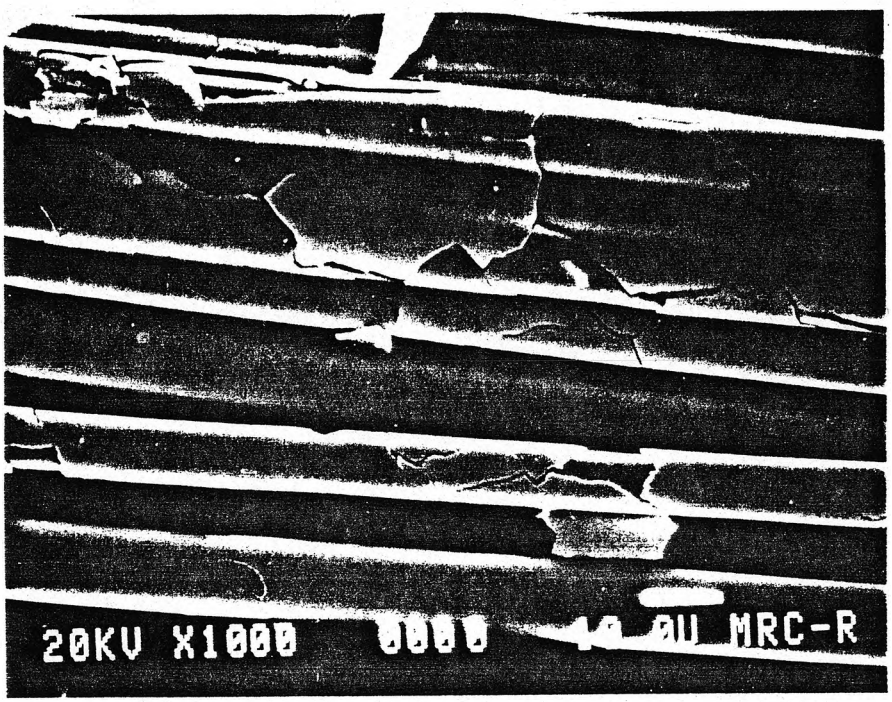


Fig.7a Surface of Tyranno fiber exposed at 1300°C in air showing oxide layer cracking

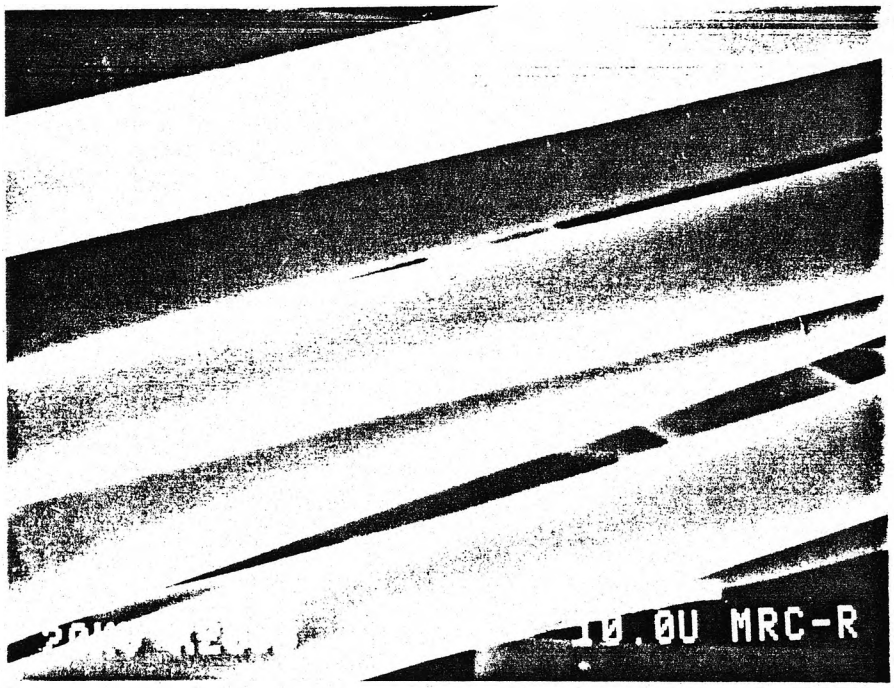


Fig.7b Surface of Nicalon fiber exposed at 1300°C in air

Fig.8a Cross section of unidirectional alligned Tyranno fiber reinforced mullitic matrix composite

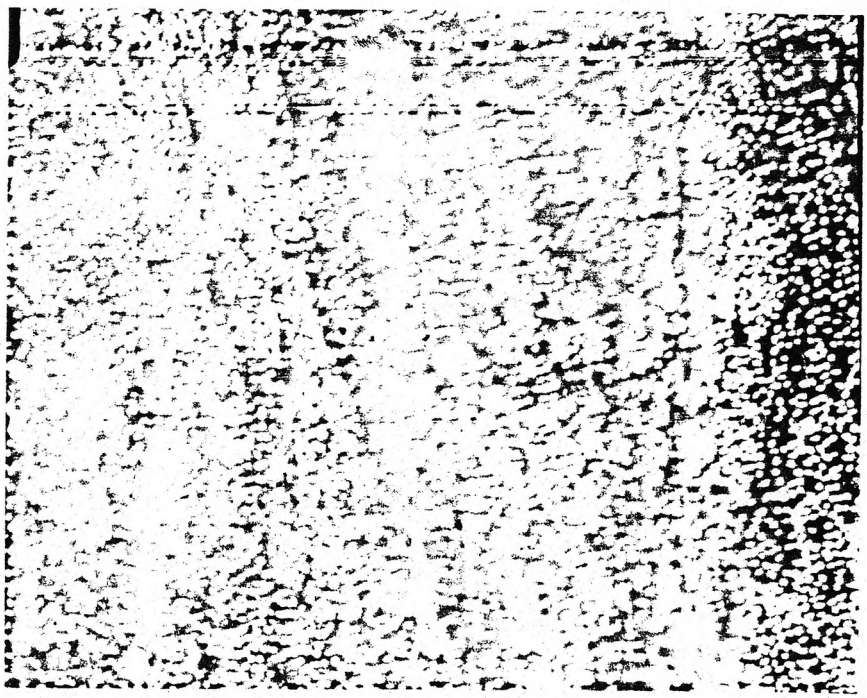


Fig.8b Cross-section of unidirectional alligned Nextel fiber reinforced mullitic matrix composite

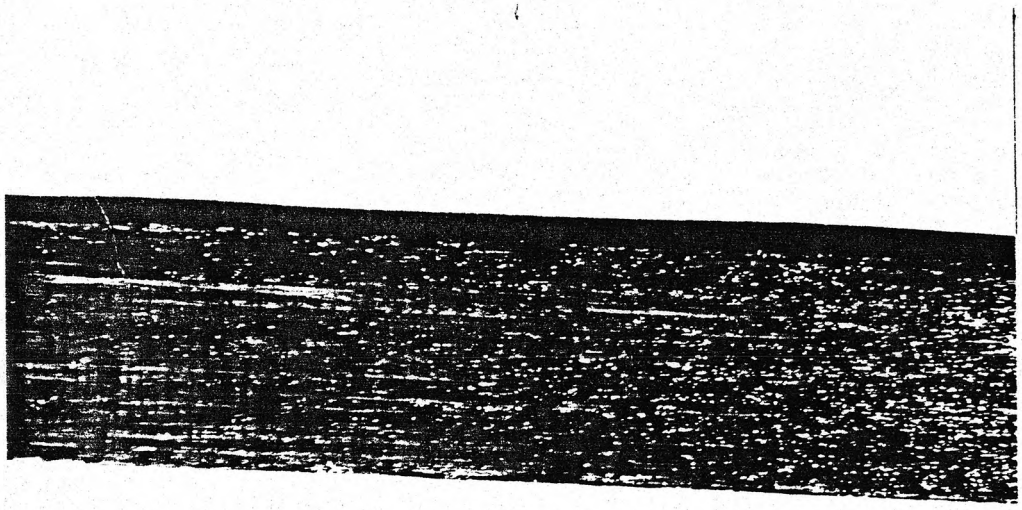


Fig.9a Surface of unidirectional alligned Tyranno fiber ceramic composite

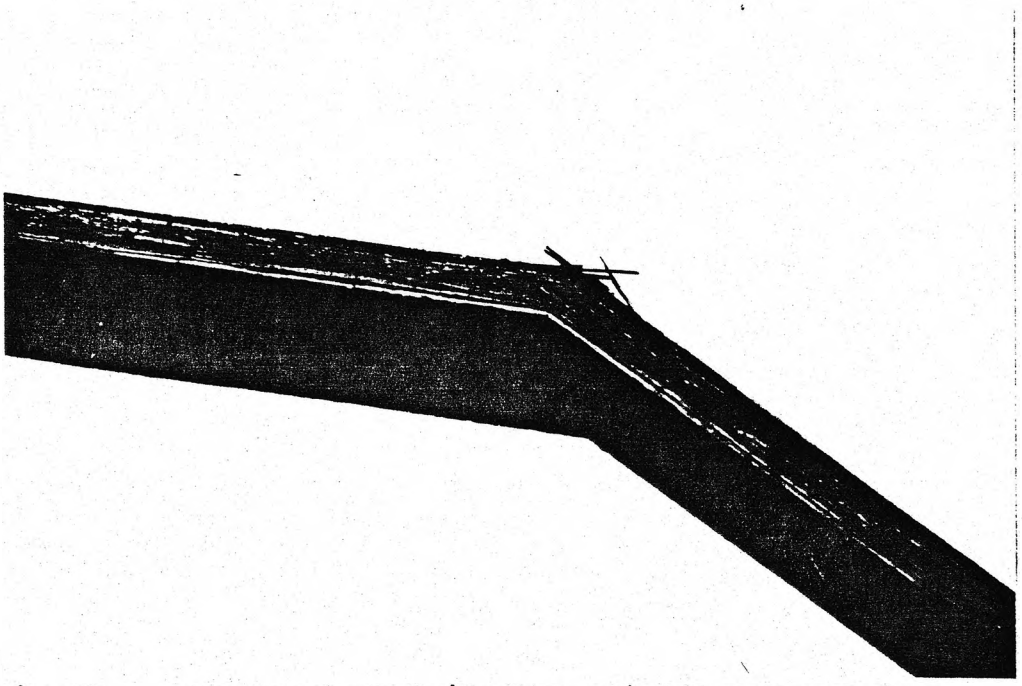
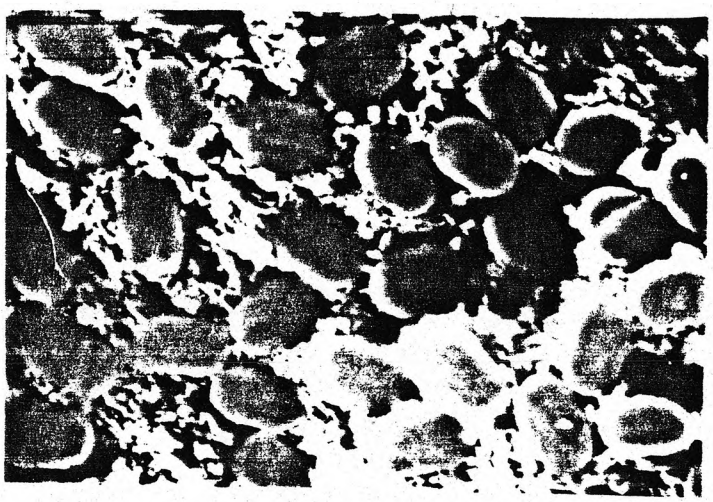
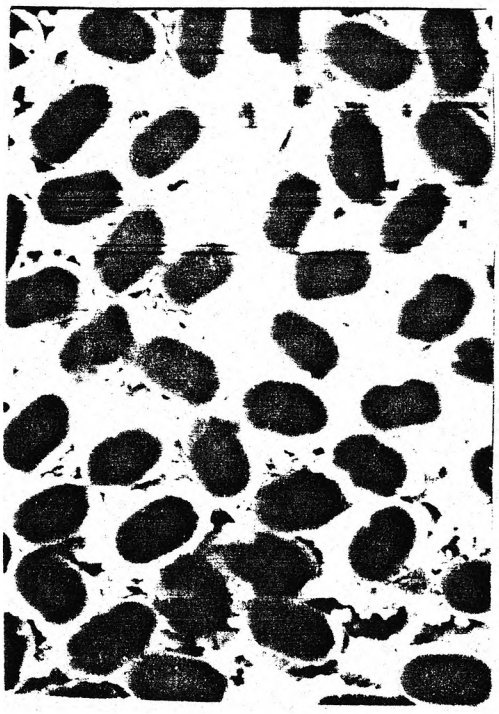


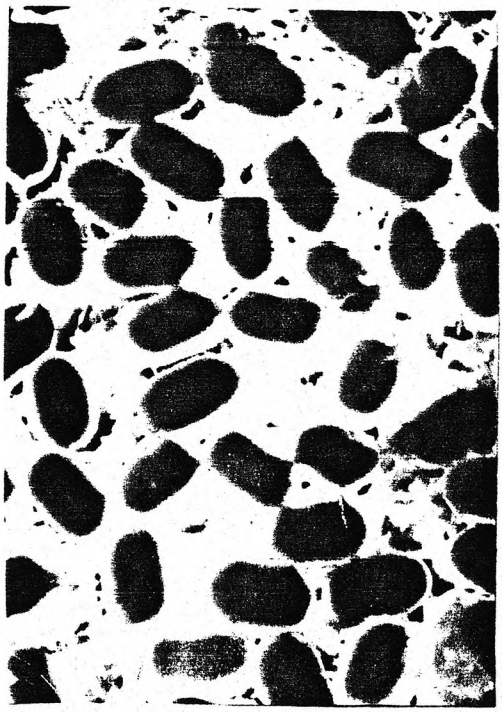
Fig.9b Surface of failed 3-point bend specimen of unidirectional alligned Tyranno fiber mullitic matrix composite



(A)



(B)



(C)

Fig. 10 SEM micrographs showing porous matrices after firing to various temperatures, (a) 1250°C , (b) 1400°C, (c) 1450°C

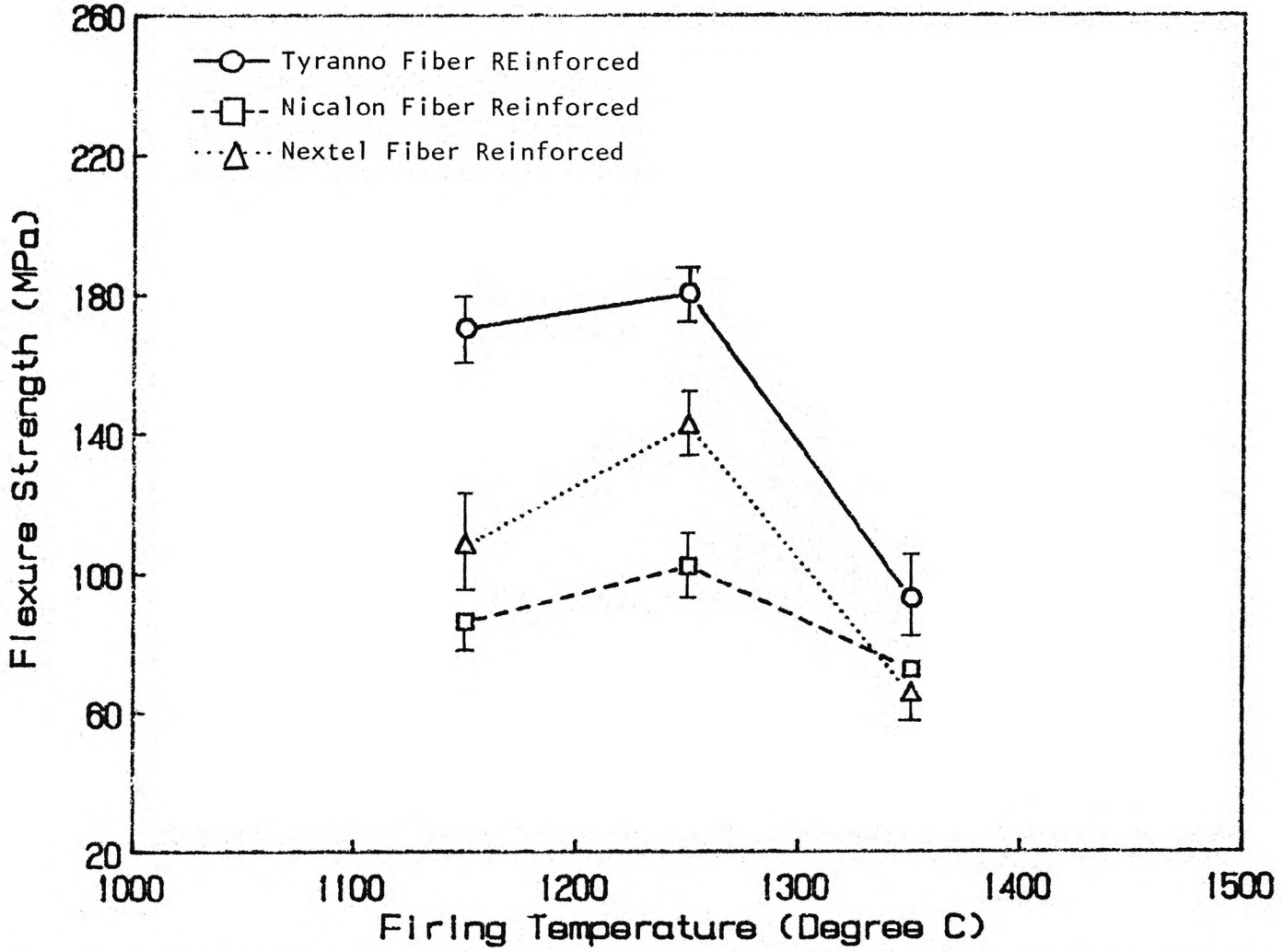


Fig.11 VARIATION of FLEXURE STRENGTH with FIRING TEMPERATURE (INDIVIDUAL PREPREG).

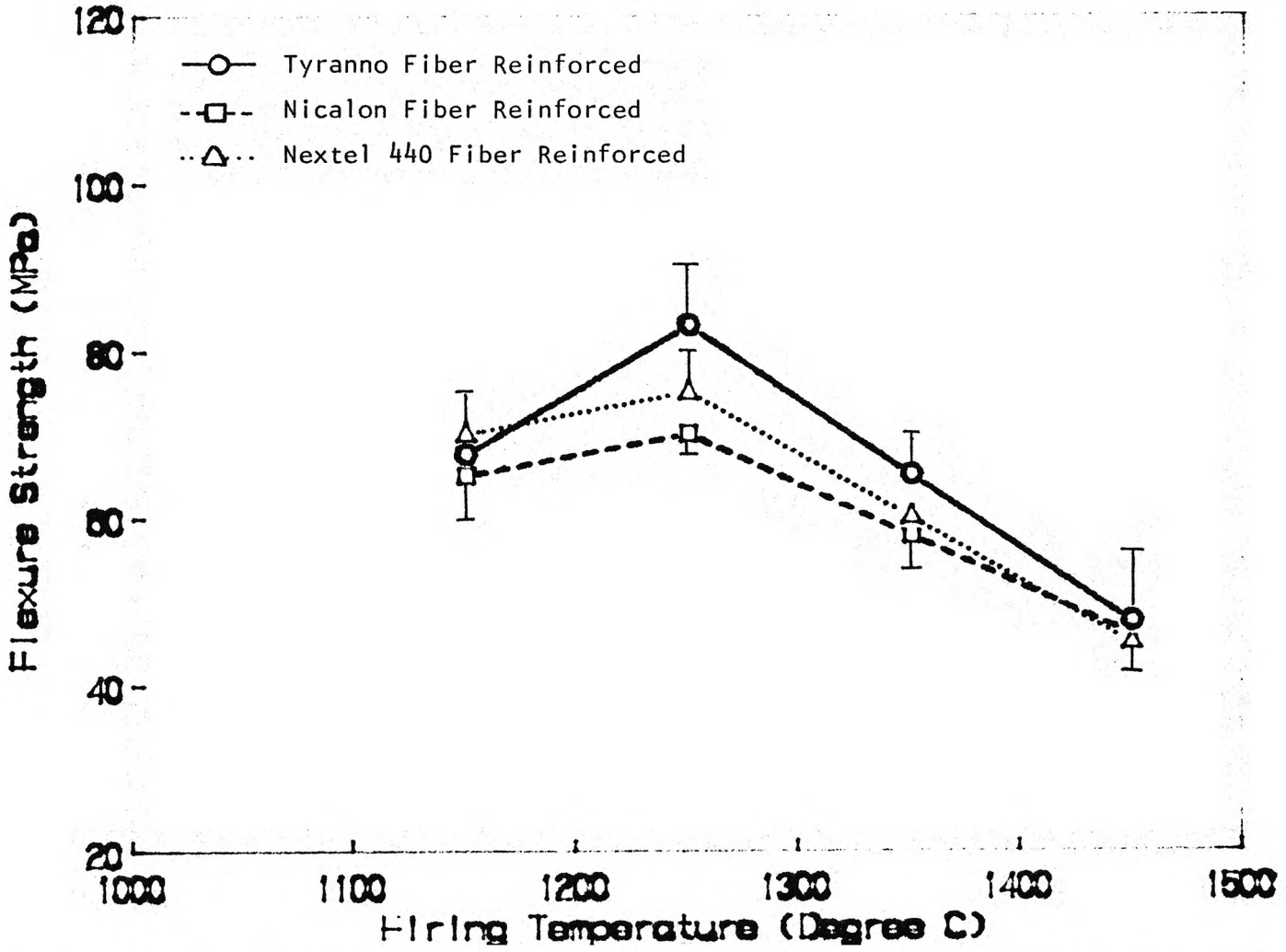


Fig.12 VARIATION of FLEXURE STRENGTH with FIRING TEMPERATURE (LAMINATED COMPOSITES, S/d = 20).

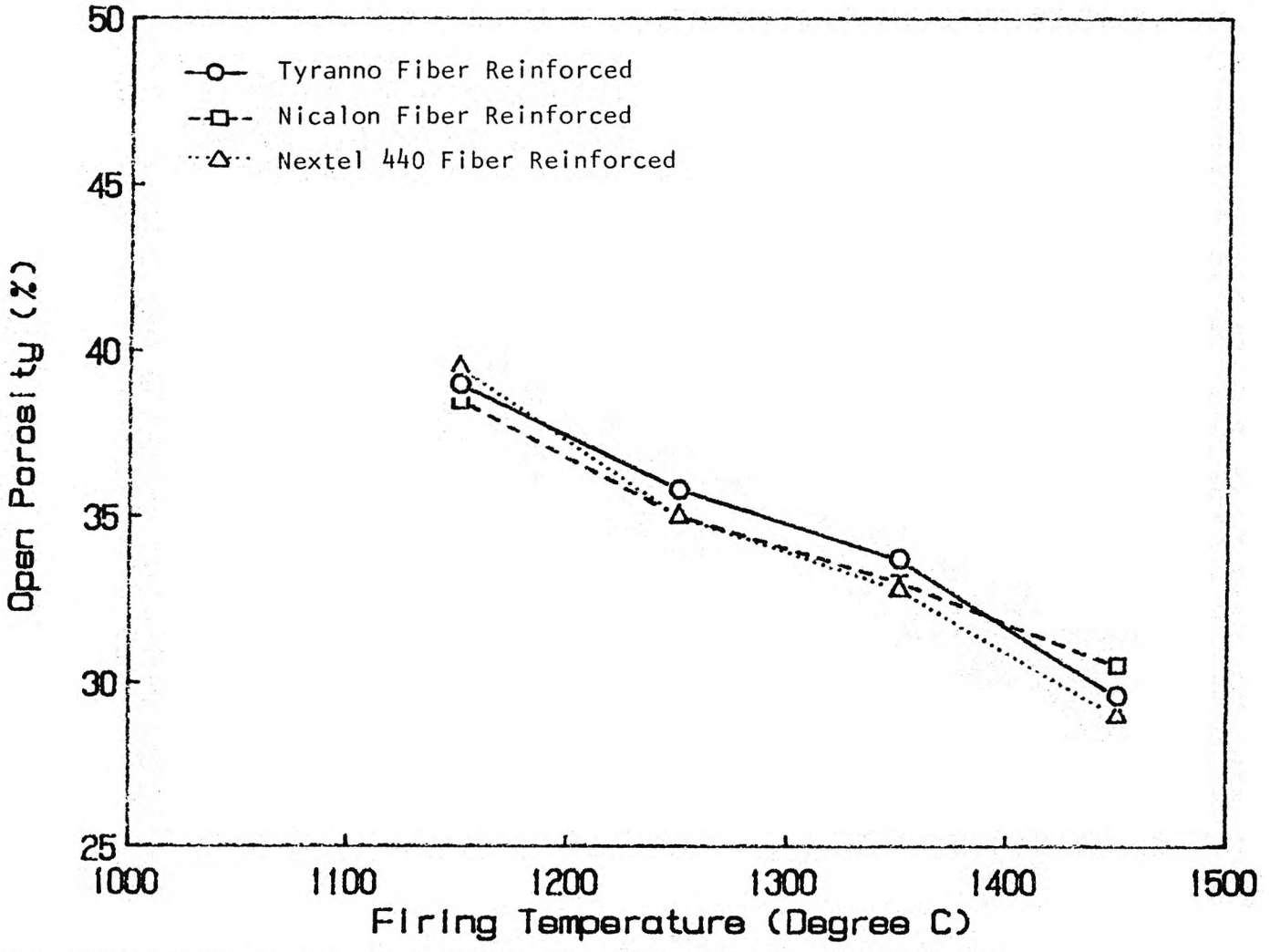


Fig.13 VARIATION of OPEN POROSITY with FIRING TEMPERATURE.

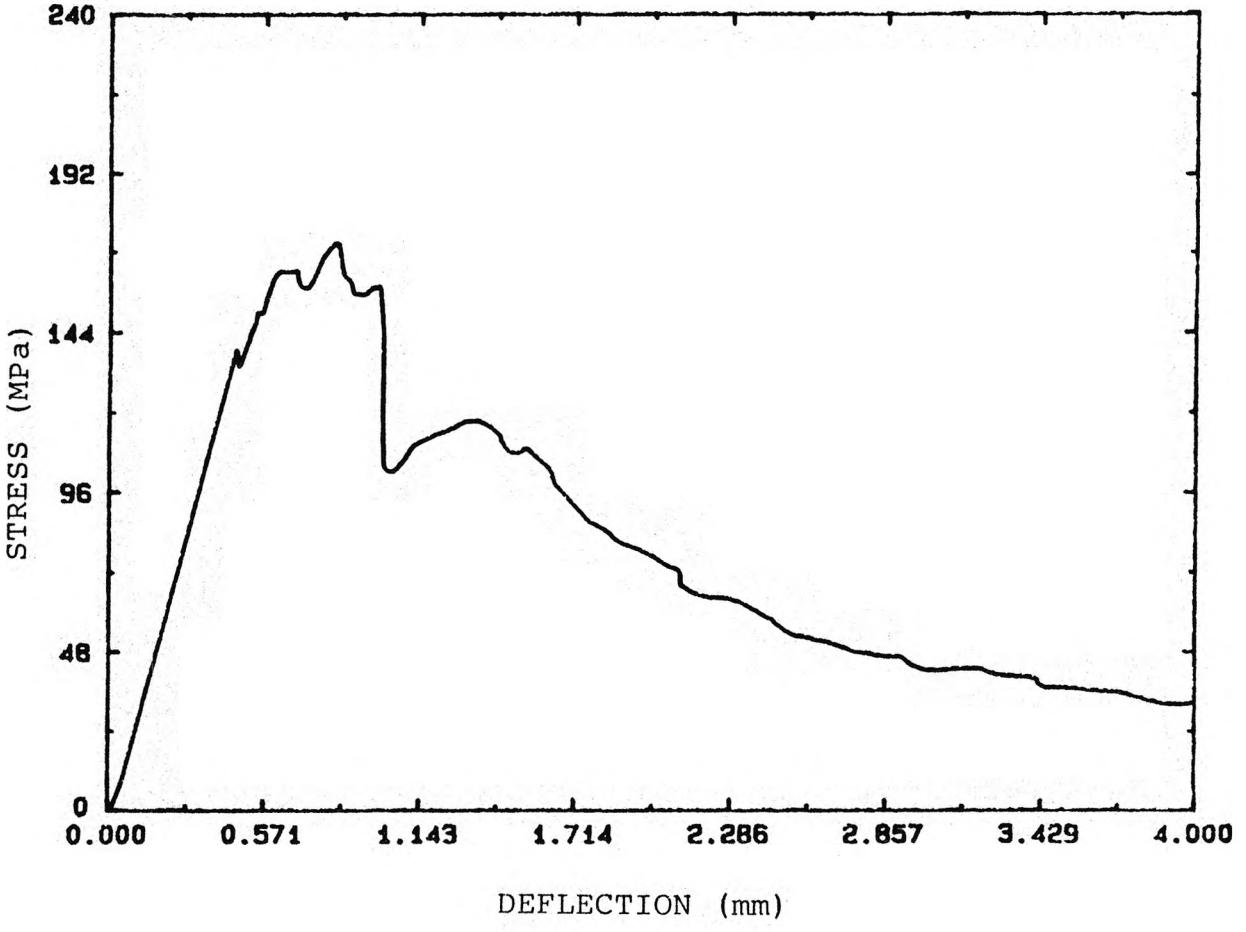


Fig.13 Load-deflection curve under flexure of a unidirectional alligned Tyranno fiber reinforced mullitic matrix composite(fired at 1250°C)

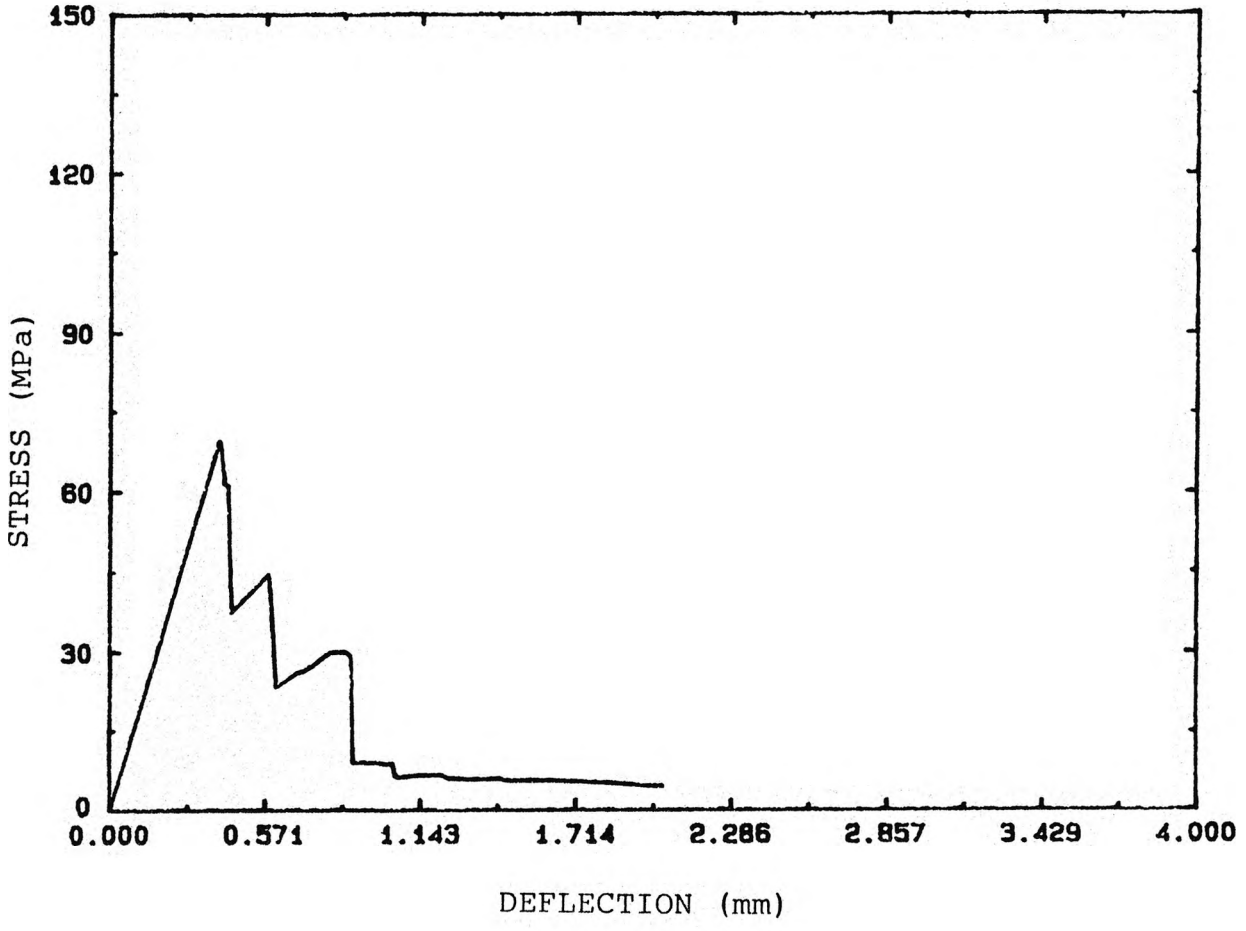


Fig.14 Load-deflection curve under flexure of a unidirectional alligned Tyranno fiber reinforced mullitic matrix composite(fired at 1350°C)

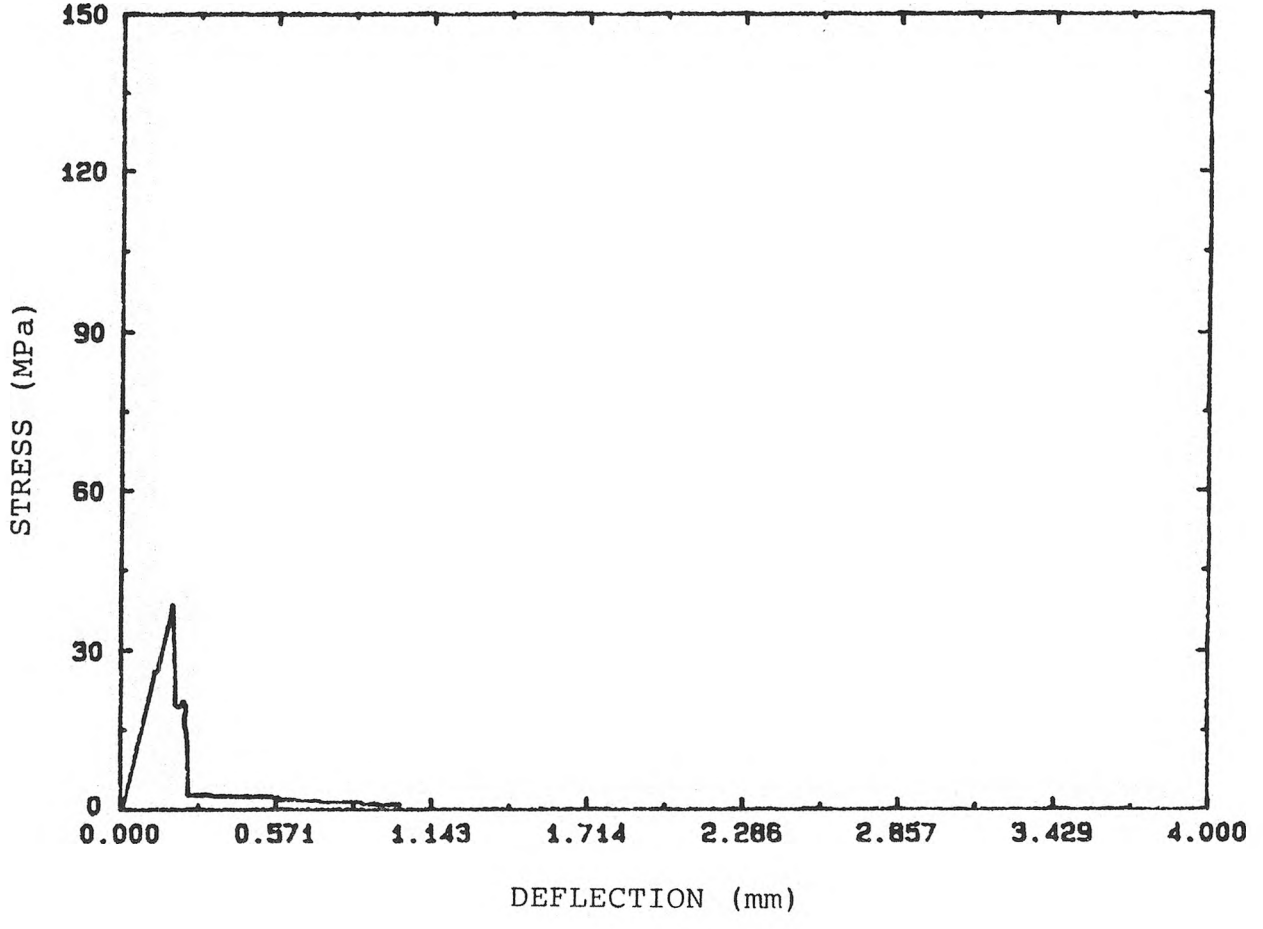


Fig.15 Load-deflection curve under flexure of a unidirectional alligned Tyranno fiber reinforced mullitic matrix composite(fired at 1450°C)

Fig.17 SEM fractograph of a unidirectional alligned Tyranno fiber reinforced mullitic matrix composite, fired at 1250°C, showing extensive fiber pull-out

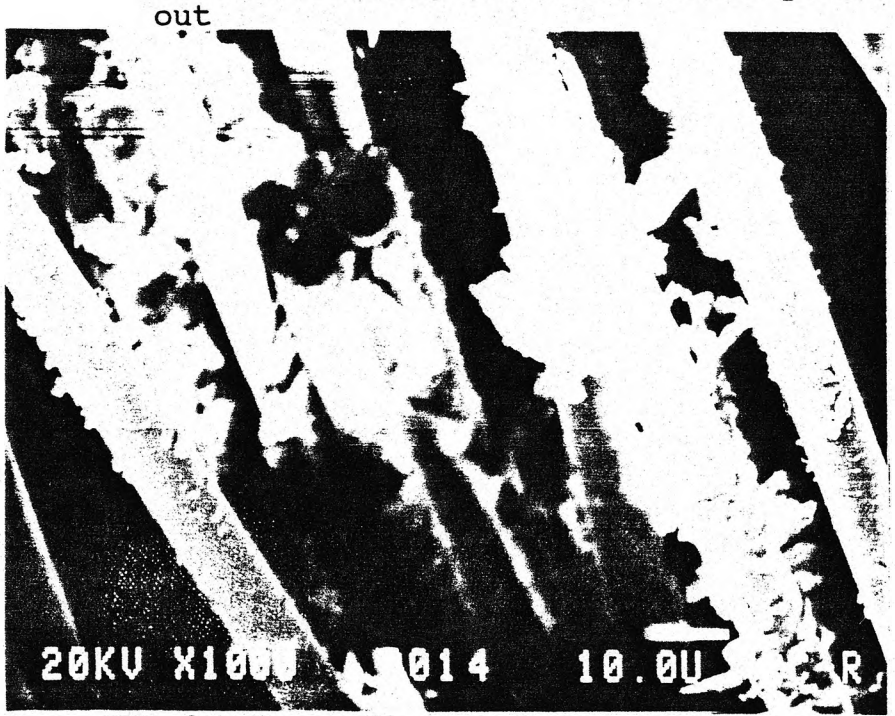


Fig.18 SEM fractograph of a Tyranno fiber reinforced mullitic matrix composite showing fiber tear-out from the matrix

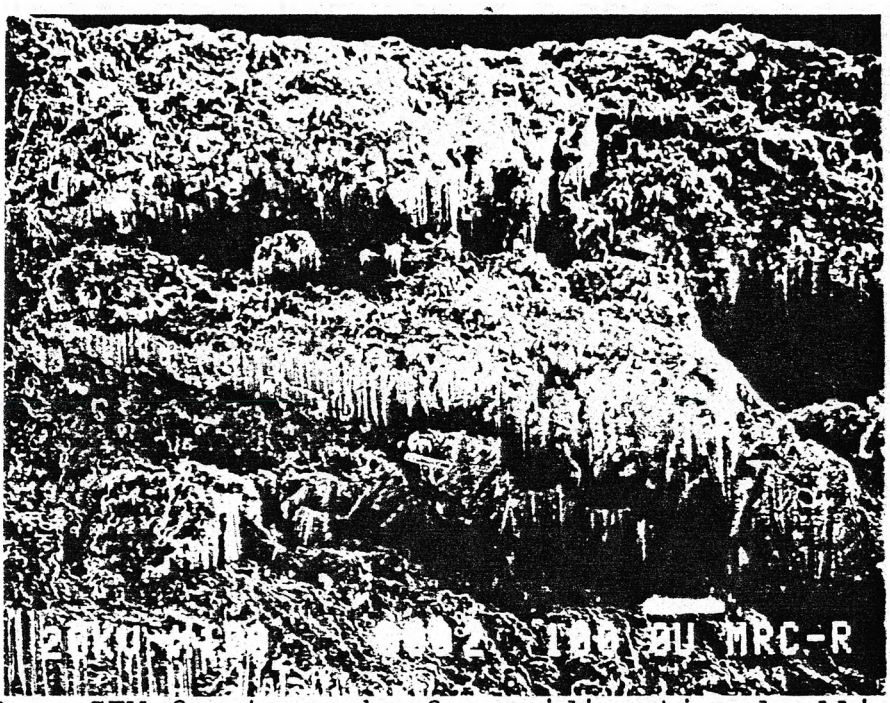


Fig.19 SEM fractograph of a unidirectional alligned Tyranno fiber reinforced mullitic matrix composite, fired at 1350°C, showing limited fiber pull out

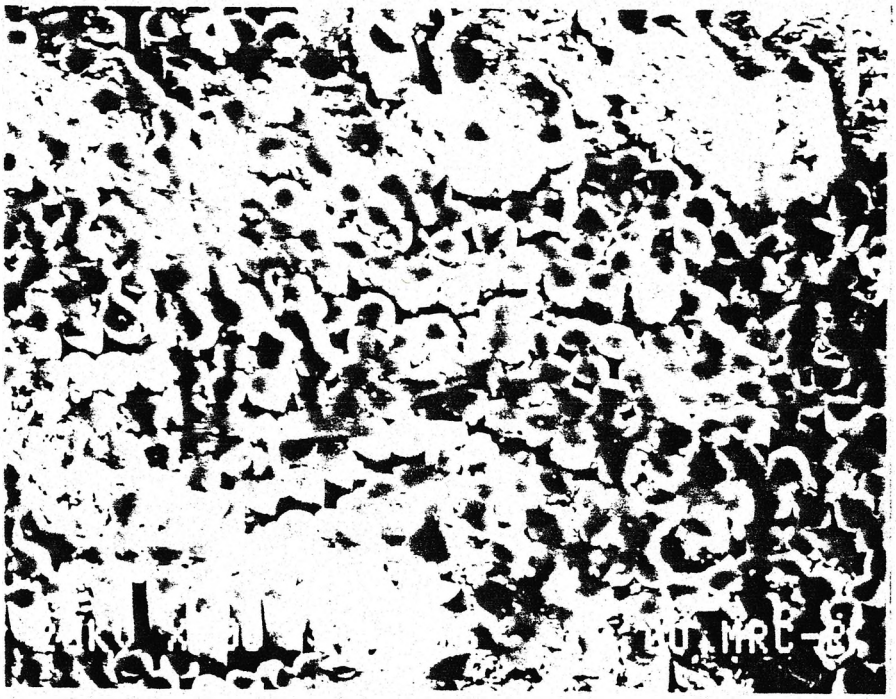


Fig.20 SEM fractograph of a Tyranno fiber reinforced mullitic matrix composite, fired at 1350°C, showing crack parallel to the fibers

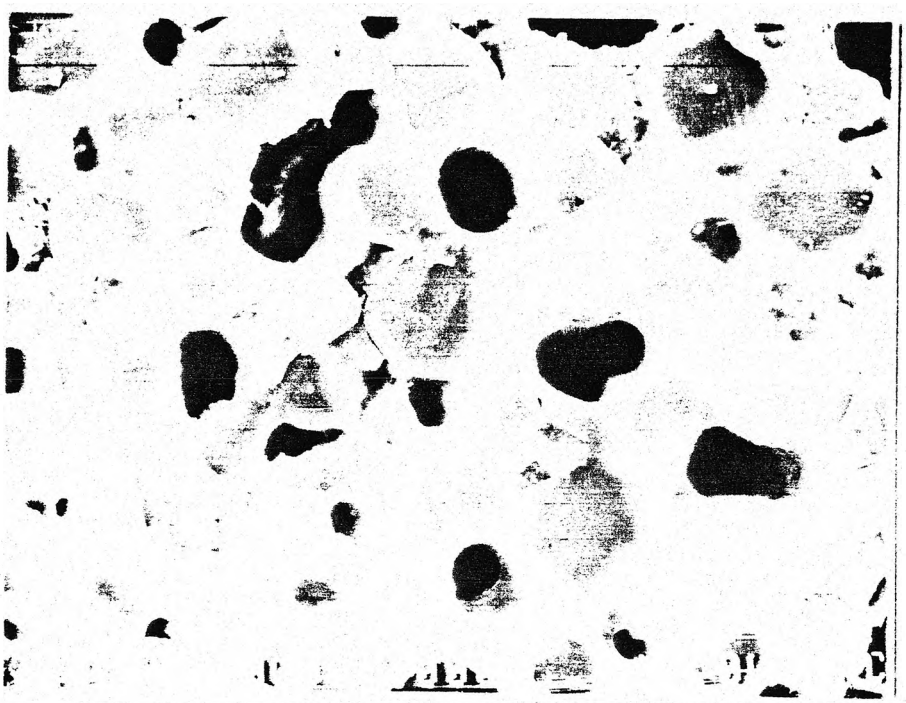


Fig.21 SEM fractograph of a unidirectional alligned Nicalon fiber ceramic matrix composite, fired at 1450°C, showing fiber debonding and fiber pull out

VITA

Wen-Chiang Tu was born on August 8, 1961 in Tainan, Taiwan, Republic of China. He Received his primary and secondary education in Tainan, Taiwan. In June of 1983 he received his Bachelor of Science in Materials Science and Engineering from National Tsing Hua University in Shin-Chu, Taiwan. He joined the R. O. C Army as a second lieutenant in July, 1983 and retired from the Army in July, 1985. Immediately thereafter he joined the Department of Materials Science and Engineering of National Tsing Hua University as a research assistant. In August of 1986 he entered the University of Missouri-Rolla to pursue his Master of Science degree in Ceramic Engineering. He is a member of the American Ceramic Society.