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THE EFFECT OF THERMAL EXPOSURE ON THE MECHANICAL PROPERTIES OF ALUMINUM-GRAPHITE COMPOSITES

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16. Abstract

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The Effect of Thermal Exposure on the Mechanical Properties of Aluminum-Graphite Composites

I. H. Khan

ABSTRACT

The mechanical properties of aluminum-graphite composites were measured at room temperature in the as-received condition, after elevated temperature exposure and after thermal cycling. The composites were fabricated by solid-state diffusion bonding of liquid-phase Al-infiltrated Thornel 50 fibers. The results showed that the maximum longitudinal tensile strength of the as-received material was 80,000 psi, which corresponds well with the rule of mixture value. The composite strength was observed to vary widely, depending on the extent of wetting of the fibers by the aluminum. The strength of the composites in the transverse direction was generally very low, due to poor interfacial bonding. Aluminum carbide (Al_4C_3) formed at the surface of the fibers at temperatures greater than 500°C. Development of the carbide was shown to be diffusion-controlled and was dependent on the time and temperature used. It was shown that the tensile strength was virtually unaffected by heat-treatment up to 500°C; beyond that temperature a drastic degradation of tensile strength occurred. The degradation could be correlated with the extent of carbide development at the interface. Thermal cycling of the composites below 500°C resulted in an observable degradation of the composite strength. Scanning electron microscopy of fractured surfaces indicated that the relatively weak interface governs the mode of failure in tension.

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INTRODUCTION

Considerable interest has been shown recently in the development of aluminum-graphite composites for aerospace applications. This is because of both the high specific strength and modulus theoretically obtainable, and the potential low cost of the composties. However, major problems encountered in the fabrication of such composites include wetting, bonding, and interfacial reaction at the fiber-matrix interface. The reaction at the interface can degrade the mechanical properties so severely as to render the material useless for practical applications. Baker *et al.*,[1] have shown in compatibility tests that carbide growth occurs at temperatures above 600°C. Jackson *et al.*[2] observed chemical interaction between Al and the fibers during stress-rupture tests at 400°C, resulting in a significant lowering of the composite strength. However, Upp *et al.*[3] in short-time high-temperature tensile tests observed no chemical reaction or degradation of the composites at 560°C. A better understanding and, hence, control of the interface reaction in the reactive composite system is, therefore, important if the full potential of aluminum-graphite as a structural material is to be realized.

A number of methods have been reported for fabricating aluminum-graphite composites from multifiber yarns and tows.[4] Among them, liquid-phase hot pressing and solid-state diffusion bonding are now commonly used. From results reported so far,[5] the solid-state diffusion bonding process appears to yield aluminum-graphite composites of the highest quality and with the most consistent mechanical properties. Composites fabricated by this method were used in the present investigation.

The mechanical properties of aluminum-graphite composites have been studied by a number of investigators. Pepper and Penty[4] prepared composites by using an infiltration/liquid phase hot pressing technique, and reported longitudinal tensile strengths of 42,300-63,850 psi, with about 26 vol. pct fibers. Using 30 vol. pct fibers, Morris[6] reported tensile strength values of 50,000-60,000 psi for composites prepared by liquid-phase hot pressing. Jackson *et al.*[2] prepared samples by vapor deposition and reported tensile strength values of 60,000-80,000 psi (with 30 vol. pct fibers). There is thus a wide range of variability in the composite strength reported by various investigators. The reasons for the lower or unpredictable values, or both, have not been defined or explored thoroughly. There is thus a need for an investigation which will define these reasons and contribute to the solution of the problems so that aluminum-graphite composites with improved and consistent properties can be developed for practical applications.

In this paper we will discuss the interface structure in aluminum-graphite composites and its effect on the composite properties, the effect of thermal cycling on the tensile properties of the composites, and observations on the mode of failure of the composites in tension.

EXPERIMENTAL TECHNIQUES

Composite Fabrication

The aluminum-graphite composites used in this investigation were fabricated by DWA Composite Specialities, Inc., using Al-infiltrated graphite fibers obtained from Aerospace Corporation and Fiber Materials, Inc. The graphite fibers used were Thornel 50, with the following properties: tensile strength 225-275 × 10^3 psi and modulus 50-55 × 10^6 psi. Thornel 50 graphite fibers consisting of a continuous yarn containing eight tows of fiber totaling approximately 11,000 individual fibers were drawn through a pre-cleaning and coating chamber and then through a molten bath of the desired aluminum alloy. The cleaning of the fiber surface was accomplished by high-temperature oxygen treatment, and the coating process served to provide an adherent coating of a mixture of Ti and B. The coating was intended to protect the fiber from chemical interaction with the molten aluminum and to promote wetting of the fiber, thus permitting complete infiltration of the yarn by the aluminum. The diameter of the resultant aluminum-graphite wire or rod was typically 0.050 in. and contained 25-30 pct graphite fiber. The typical properties of the wire ranged from ~80 to 120 ksi, depending on v/o fiber and degree of infiltration. The wire modulus was typically 22-28 × 10^6 psi. This wire was then the precursor material for further processing into bulk shapes. The aluminum alloys commonly used were 1100, 6061, and 201.

Fabrication of the aluminum-graphite precursor wire into panels was done by DWA Composite Specialties, Inc. using the solid-state diffusion bonding technique. This process resulted in a consistent and predictable product (assuming the precursor wire was consistently good). The solid-state diffusion process involved pressing of packed wires for 40 min at 3000 psi at about 560°C. The consolidated part was then trimmed and cleaned in a $HNO_3/HF/H_2O$ solution. The resulting composite had the form of an 8- by 12-in. panel which was sufficient to allow the preparation of 25 longitudinal and 5 transverse test specimens.

Test Specimens

Tensile specimens were cut from each composite panel using a high speed cutter with a line tracer. Actual dimensions of the specimens were: total specimen length, 4 in.; thickness, 0.120 in.; parallel gage section, 1.0 in.;

gage width, 0.375 in.; and radius of curvature, 0.38 in. from shoulder to gage section. Fig. 1 shows a typical tensile specimen.

All tensile testing was performed at room temperature using an MTS Systems Corp. hydraulic testing machine or similar system at an actuator velocity of 5×10^4 in./sec. For complete stress-strain curves to failure, elongations were determined from extensometer readings over the 1-in. gage length. A universal joint was used to align specimens with the center line of the loading train. Both longitudinal and transverse tensile strengths of the composite were measured.

For the purpose of detailed examination of aluminum-graphite interfaces, diffusion couples were prepared by vacuum deposition of aluminum onto carbon and graphite in an experimental ultra-high vacuum high-energy electron diffraction (HEED) system. The diffusion couples studied include Al-thin C film, Al-single crystal Gr film, and Al-crystalline bulk Gr. It was initially assumed (and subsequently confirmed) that the interface created in the diffusion couples was chemically the same as that in the aluminum-graphite composites.

Platinum marker experiments were performed to determine the diffusion behavior in the formation of the interfacial reaction product in the Al/Gr system. The specimen structures used in these experiments consisted of a polished disc of graphite on which three strips of platinum were deposited through masks. The strips were about 1 mm wide and 3.5 μ thick. Aluminum, about 8 μ thick, was then deposited onto the entire surface of the graphite specimen. The specimens were subjected to thermal exposure at 600°C for 36 hr in a high-vacuum furnace. They were then machined perpendicular to the surface by a wire saw and the transverse section examined by scanning electron microscopy.

Structural Analysis

The interface structure in the aluminum-graphite diffusion couples was examined *in situ* by high-energy electron diffraction (HEED) and scanning electron microscopy (SEM). Continuous monitoring of the HEED patterns provided information concerning the development of the interfacial reaction product as a function of time and temperature. Optical and scanning electron microscopy were used to obtain information regarding the mode of failure of the composite test specimens in tension. X-ray radiography was used to analyze macroscopic voids or defects in the composite specimens. Only specimens with no observable voids or defects were used for tensile tests.

RESULTS AND DISCUSSION

Interfacial Reaction

Electron diffraction observations with aluminum-graphite diffusion couples have shown that chemical reaction between aluminum and graphite begins at about 500°C, and that the reaction rate increases with increasing temperature. The reaction product formed at the interface was identified as Al_4C_3 with a hexagonal structure ($a_o = 3.32$ Å, $c_o = 24.89$ Å). The structure is considered to consist of hexagonal layers of Al atoms interspersed with layers of C atoms. The electron diffraction observations are illustrated in Fig. 2. The typical transmission HEED patterns from amorphous carbon and from polycrystalline aluminum deposited on the carbon are shown in Figs. 2(a) and (b), respectively. Heat treatment of the Al-C couple at 600°C resulted in the development of the pattern shown in Fig. 2(c). Analysis of the pattern shows that the aluminum carbide grows in the form of single crystalline platelets in (0001) orientation with the c-axis of the hexagonal carbide lattice perpendicular to the platelets. The platelet structure of the carbide was observed by transmission electron microscopy.

The development of the reaction zone at the interface was studied as a function of time and temperature. Fig. 3 is a scanning electron micrograph of a sectioned aluminum-graphite diffusion couple illustrating the reaction zone formed at 640°C after 75 hr. A plot of the mean reaction zone thickness against $t^{1/2}$ (Fig. 4) gives straight lines, indicating that the reaction process is diffusion-controlled. The parabolic rate constants, calculated from Fig. 4, are plotted in Fig. 5. From this plot, the activation energy for the formation of the reaction zone has been calculated and determined to be 35.17 kcal/mole. The diffusion behavior in the growth of the reaction zone was determined from platinum marker experiments. The marker experiments have shown that aluminum deposited on the platinum marker was converted to aluminum carbide. This points to the fact that carbon diffuses into aluminum to form Al_4C_3. This is expected from atomic size considerations. Carbon, which has a smaller atomic mass than aluminum is likely to diffuse into aluminum more readily. It was also observed that aluminum on the platinum marker was completely converted into Al_4C_3, while that on graphite was not. This could be due to the higher diffusivity of carbon along the Pt-Al boundaries than in aluminum. The experiments do not, however, exclude the possibility of some diffusion of aluminum into carbon.

Tensile Behavior of As-Received Composites

The room temperature mechanical properties of as-received Al-Gr composites are shown in Table I. It should be mentioned here that the fiber strength was calculated using the equation

$$\sigma_f = (\sigma_c - \sigma_m Vm)/V_f$$

where σ_f, σ_c, and σ_m are the apparent tensile strength of fibers, the strength of the composite, and the strength of the matrix, respectively; and V_m and V_f are the volume fractions of the matrix and fibers, respectively. The tensile properties of Al-Gr composites with three different aluminum matrices were investigated.

The 1100 Al-Gr composites exhibited tensile strengths that were significantly below the rule-of-mixtures value. Visual and optical microscopy observations on fractured surfaces showed that the graphite fibers were not wetted by aluminum during the fabrication process.

The 6061 Al-Gr composites, which were prepared with Al-infiltrated graphite fibers obtained from Fiber Materials, Inc., showed a consistent strength level (63,000 ± 2,500 psi). However, the same material prepared with Al-infiltrated graphite fibers manufactured by Aerospace Corporation exhibited a range of strength values from 40,000-80,000 psi. The strength levels obtained for three different panels tested were: 40,000-42,000 psi, 60,000-65,000 psi and 77,000-80,000 psi, respectively. This variation in strength from panel to panel was suspected to arise from changes in wetting conditions in the infiltration process with a resultant effect on the fiber-matrix bond strength.

The 201 Al-Gr composites generally showed consistent mechanical properties that corresponded to those calculated from the rule-of-mixtures. This fact points to the improved wetting capabilities of the 201 Al alloy with graphite fibers. The longitudinal strength and modulus are comparable to those of the best 6061 Al-Gr composites; however, the primary yield stress was observed to be consistently larger for the former owing to the higher yield strength of the 201 alloy.

The transverse strength of the Al-Gr composites was much lower than expected (Table I). The low transverse strength and failure of the fiber-matrix interface in transverse tension, together with the large amounts of fiber pull-out observed, imply a weak interface and therefore a low interfacial bond strength. The ratio $\sigma_{trans.}/\sigma_{long.}$ is about 0.06, indicating that splitting and hence notch-insensitive behavior is to be expected in notched tensile tests.[7] Further, the weak interface and large pull-outs are factors that are expected to confer an appreciable degree of toughness on the material investigated.

Composite Microstructure

The microstructure of a polished section of a longitudinal composite specimen is illustrated in Fig. 6(a). Al-infiltrated precursor wires with Al matrix are clearly visible. No bond lines between wires can be seen. An enlarged view of the precursor wire (Fig. 6(b)) shows fairly uniform distribution of the fibers. The white background represents the distribution of the Al matrix. Fig. 6(c) shows the microstructure of the fibers when the polished surface was etched. Here the fibers look fairly smooth, indicating that they have not reacted with aluminum during the fabrication process, especially during the fabrication of the precursor wires.

The SEM observation of the microstructure of the fracture surface of an as-received longitudinal specimen is illustrated in Fig. 7(a). Fiber pull-outs can be seen, with evidence of ductile behavior of the matrix in the form of necking. Fig. 7(b) shows a typical fracture surface when the composite specimen was heat treated at 600°C for 24 hr. The surface shows extensive fiber pull-outs. The carbide overgrowth on the fibers can be clearly seen, indicating that the graphite-carbide bonding is stronger than Al-carbide bonding. A view of a section of the surface reveals development and propagation of cracks at the boundary between the Al matrix and the fibers (Fig. 7(c)). Observations of an etched area near the fracture surface on a longitudinal surface of the specimen show random broken fibers at the broken edge. However, no broken fibers were observed away from the fracture surface.

Fig. 8 illustrates the microstructures of graphite fibers before and after chemical interaction at 600°C. The specimens were prepared by dissolving the Al matrix just prior to SEM examination. As can be seen in Fig. 8(a), prior to high-temperature exposure, the surfaces of the fibers are smooth, indicating no chemical reaction at the fiber surface during the fabrication process. However, chemical reaction at 600°C resulted in surface damage, as shown in Fig. 8(b). The micrograph indicates that the carbide growth on the fibers is irregular. Carbide particles nucleate on the fiber surface and increase in size and density with increasing time and temperature until they form a continuous layer at the interface. This layer can be extremely irregular, due to the relatively large size and random orientation of the particles. As discussed earlier, the reaction kinetics study shows that the carbide growth is a diffusion-controlled process. It may be suggested that the carbide layer acts as a diffusion barrier; this in turn implies that the growth of the isolated particles proceeds mainly by surface diffusion. The surface reaction is likely to create random notching of the fiber surface, thereby causing drastic degradation of the fiber strength and hence of the composite strength.

A typical SEM fractograph of a transverse composite specimen is shown in Fig. 9. The specimen is seen to fail at the fiber-matrix interface. The surfaces of the graphite fibers are clearly visible with little or no aluminum matrix material adhering to them. This observation explains the poor transverse strength observed for the composites.

The matrix microstructure as observed by scanning microscopy of a fractured specimen is illustrated in Fig. 10. The fact that the cracks were observed in both longitudinal and transverse fractured specimens suggests that they developed during the fabrication process. These appear to be caused by the stresses and strains that develop because of thermal mismatch between aluminum and graphite.

Effect of Chemical Interaction

The effect of prior high-temperature exposure on the room temperature tensile strength of 6061 Al-Gr composites was determined over the temperature range from 20° to 640°C. The results are summarized in Fig. 11. As can be seen, thermal exposure has little effect on the tensile strength of the composites up to 500°C; at 550°C and higher, a drastic degradation in the strength occurs. This degradation in the composite strength can be correlated with the chemical reaction at the matrix-fiber interface. As discussed earlier, the chemical reaction between aluminum and graphite begins at about 500°C, and the reaction rate increases with increasing temperature. The interfacial reaction results in the development of Al_4C_3 at the surface of the graphite fibers. The sharp drop in the strength is probably then due to the increased carbide growth at the interface. Similar effects have been reported by Metcalfe[8] in titanium-boron composites, and by Pattnaik and Lawley[9] in aluminum-steel composites, both of which exhibited a reaction zone at the metal-fiber interface. Sutton and Feingold[10] have suggested that an appropriate thickness of the reaction zone at the interface may improve the interfacial bond strength and consequently the strength of the composites. Umakoshi *et al.*[11] have observed such an effect in tungsten fiber-copper alloy composites. However, no such effect was observed in aluminum-graphite composites.

The stress-strain behavior of the composites at room temperature and after thermal treatment is illustrated in Fig. 12. The significant reduction in composite strength observed for the 600°C curve is undoubtedly due to the fiber-matrix interaction and the consequent formation of brittle intermetallic Al_4C_3 at the interface. The primary and secondary moduli are seen to be virtually unchanged after the high-temperature treatment, implying that the fiber modulus is unaffected despite the observed surface damage and consequent degradation in fiber strength.

Effect of Thermal Cycling

In the longitudinal direction of the Al-Gr composites, there is considerable mismatch of thermal expansion coefficients between the Al matrix and the graphite fibers ($\alpha_{Al} = 23 \times 10^{-6}/°C$, $\alpha_{thornel} = 0.54 \times 10^{-6}/°C$. It is likely that during thermal cycling, the mismatch would lead to failure of the bond at the matrix-fiber interface, and thus to a degradation of the composite properties.

The composite specimens were thermally cycled between room temperature and 500°C. The specimens were heated in a vacuum furnace for 15 min at 500°C and then rapidly cooled by quenching in water at room temperature. The results are illustrated in Fig. 13. The figure shows that observable degradation (~18 pct) occurs during the first 10 cycles, beyond which no appreciable degradation is observed. The fracture behavior was observed to be the same as that of the specimens that were not thermally cycled. Thermal cycling appears to weaken the fiber-matrix bond strength, and the stresses and strains that might develop due to the thermal mismatch probably concentrate at the interface, causing the bond to fail. This result is in contradiction to the observations of Pepper *et al.*,[12] who reported no degradation of the composite strength due to thermal cycling.

Since thermal cycling was performed at temperatures between 20° and 500°C, chemical reaction at the interface, which could lead to degradation of the composite strength, was not possible.

Mode of Failures

Optical and scanning electron microscopy of fractured and etched composite specimens have provided information on the mode of deformation and failure of the composites in uniaxial tension. The mode of failure of the composites observed was a function of the extent of interfacial reaction. As shown in Fig. 14(a), the failure of the composite is preceded by a shear crack. This crack is likely to initiate at or near the specimen shoulder and propagate along the matrix-fiber interface before the specimen fails. The crack initiation occurs at the shoulder because the stress concentration at this point is maximum. Extensive fiber pull-out occurs during tension (Fig. 7). The ductile behavior of the matrix can be seen in the form of necking. As evidenced by fiber pull-outs, the weak interface appears to govern the mode of failure in tension.

Temperature exposure above 500°C (for example, at 600°C) accentuates the chemical reaction at the matrix-fiber interface, and the composite strength degrades drastically (Table II). Overall fracture morphology is similar to that of the untreated specimen. However, severe cracks develop at the boundary between the matrix and the fibers (Fig. 7(c)). The cracks develop because of decreased interfacial bond strength resulting from chemical reaction. The composite material is then essentially brittle, resulting in a composite brittle failure (Fig. 14(c)). Fig. 14(b) is a case in between, which was observed after temperature exposure at 550°C for 24 hr. Reduction in strain to failure of the composites compared with the as-received condition is consistent with the brittle nature of the fracture surface (Fig. 14(c)).

CONCLUSIONS

The following conclusions can be drawn from the present investigation:

1) Aluminum-graphite composites prepared by solid-state diffusion bonding exhibit tensile properties that correspond well with those predicted by the rule of mixtures. The tensile properties could vary widely, however, depending upon the infiltration and wetting of the aluminum matrix.

2) Chemical reaction occurs at the aluminum-graphite interfaces at temperatures above 500°C, and the reaction product formed is aluminum carbide (Al_4C_3) with a hexagonal structure. The extent of the carbide development is time and temperature dependent, showing characteristics of a diffusion-controlled process of carbide growth.

3) The carbide growth on the graphite fibers causes surface damage, resulting in degradation of the fiber strength and hence the composite strength.

4) The tensile strength of the composites is not affected by thermal exposure up to 500°C; above 500°C a serious degradation of tensile strength occurs. This degradation is related to the extent of the carbide growth at the matrix-fiber interface. The retention of room temperature strength and toughness up to 500°C points to the superiority of the Al-Gr composites over

conventional high-strength Al alloys in high-temperature applications. However, the poor transverse strength of the composites is seen as a major hindrance to their practical use at this time. Improvement in transverse properties must necessarily stem from an improvement in interfacial bond strength or from incorporation of graphite fibers at an angle to the loading direction, analogous to angle-ply epoxy composites.

5) Thermal cycling of the composites between 20° and 500°C causes degradation of the interfacial bond strength and hence observable degradation of the composite strength.

6) In the as-received condition, the failure in tension is preceded by shear cracks. However, a brittle failure occurs when the composite specimen is subjected to thermal exposure at and above 600°C. The weak interface seems to govern the mode of failure in tension.

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Table I. Tensile Properties of As-Received Al-Gr Composites

Matrix	1100	6061*	201
Longitudinal strength σ_{c_1}, psi	13,000 ± 3,000	63,000 ± 2,500	77,000 ± 2,000
Longitudinal modulus E_1, psi	524,000	21.16×10^6	19.65×10^6
Longitudinal modulus E_2, psi	—	14.28×10^6	14.50×10^6
Transverse strength σ_{c_2}, psi	—	2,200-5,500	—
Apparent fiber strength σ_f, psi	—	184,700	236,500
Yield stress, psi	3,300	8,000	24,700
Fiber volume, pct	30	27	27

*Composite material prepared with Al-infiltrated fiber from Fiber Materials, Inc.

Table II. Effect of High Temperature Exposure on Tensile Strength and Modulus of 6061 Al-Gr Composites

Composite Condition	Modulus, psi (primary)	Tensile Strength, psi (longitudinal)
As-received	21.16×10^6	65,000
600°C for 1 1/2 hr	20.51×10^6	53,000
7 hr	20.51×10^6	35,000
24 hr	20.22×10^6	29,500
48 hr	—	29,000
96 hr	—	25,000
114 hr	20.20×10^6	25,000

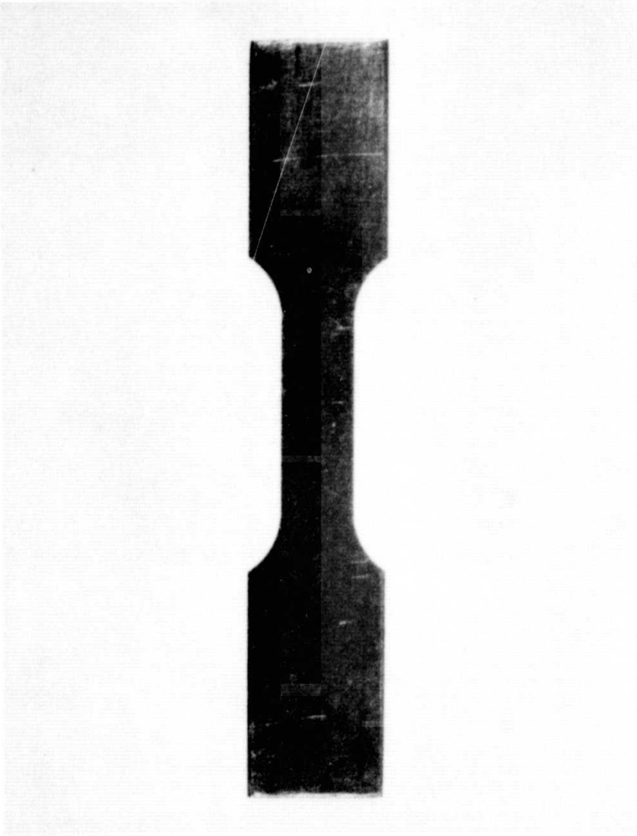

Fig. 1 - Typical aluminum-graphite composite tensile specimen.

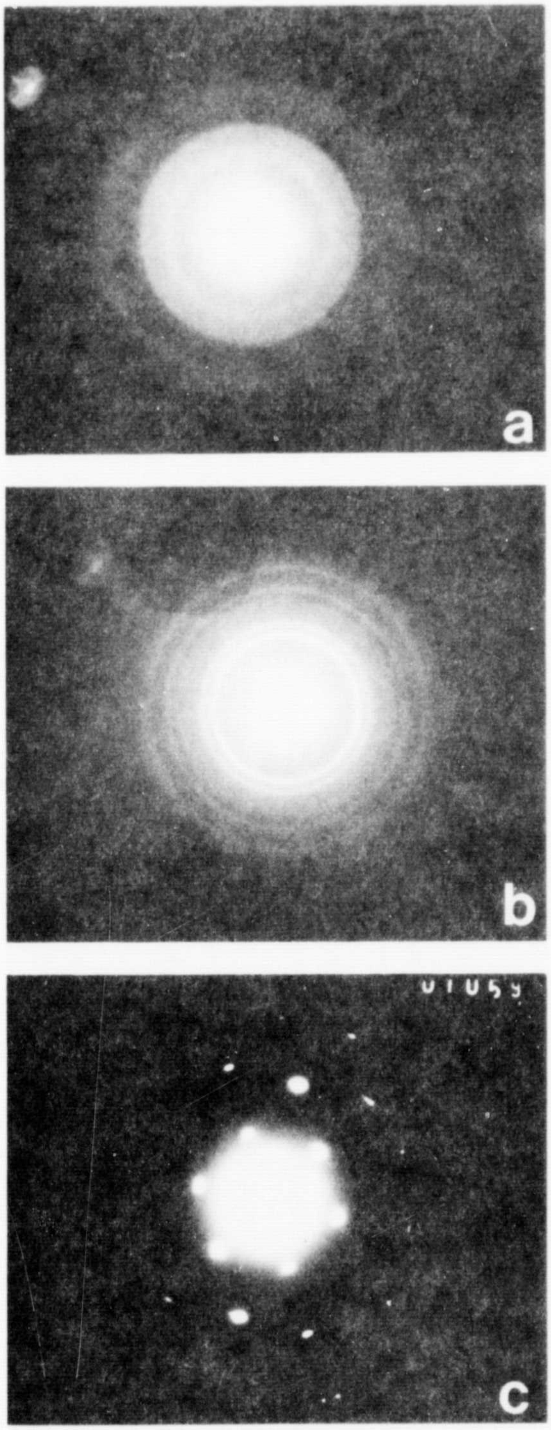

Fig. 2 - Transmission electron diffraction patterns from (a) amorphous carbon film, (b) Al (~600 Å thick) deposited on C, and (c) same as (b) but heated to 600°C for 2 hr in vacuum.

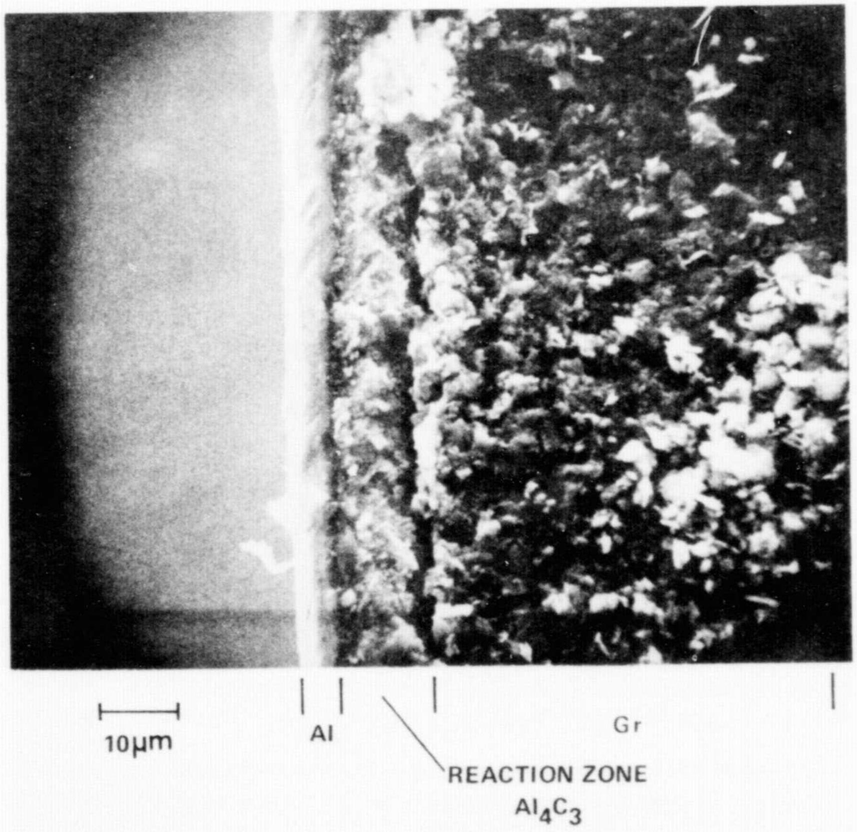

Fig. 3 - Scanning electron micrograph showing the reaction zone formed in Al-Gr systems at 640°C for 75 hr.

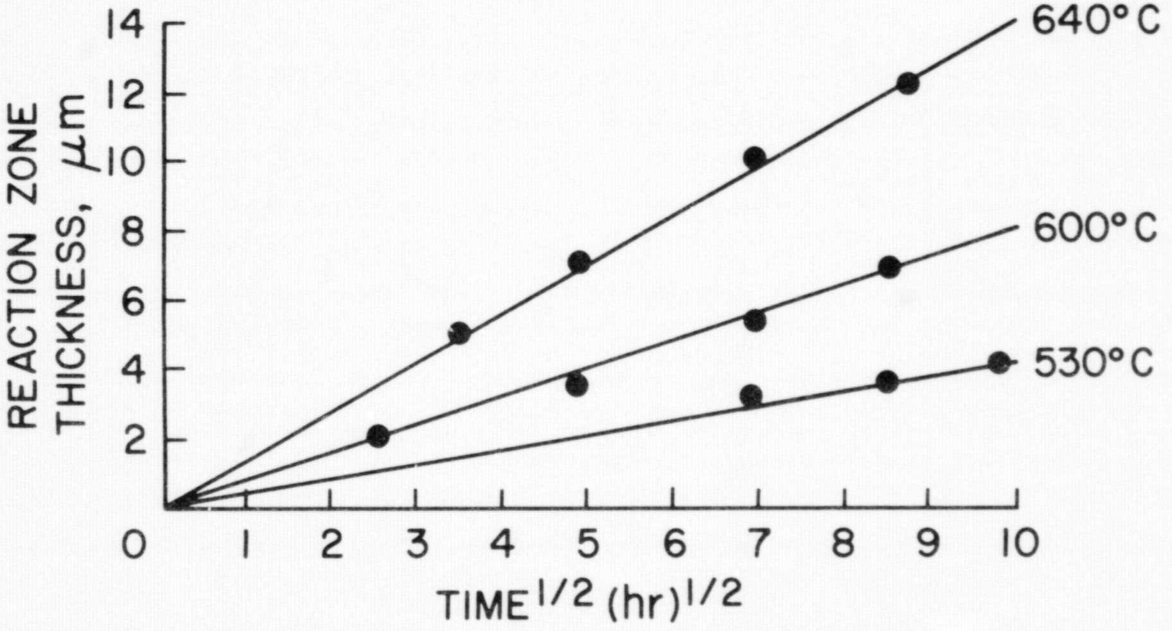

Fig. 4 - Thickness of the reaction zone as a function of temperature and time for Al-Gr systems.

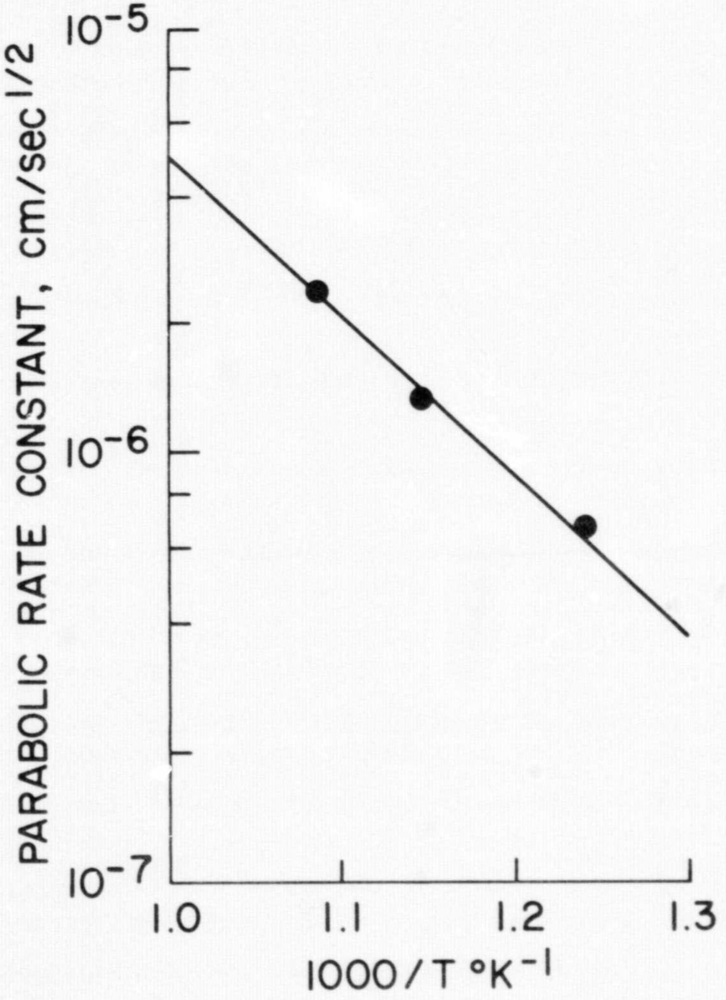

Fig. 5 - Temperature dependence of the rate constants for reaction zone growth in Al-Gr systems.

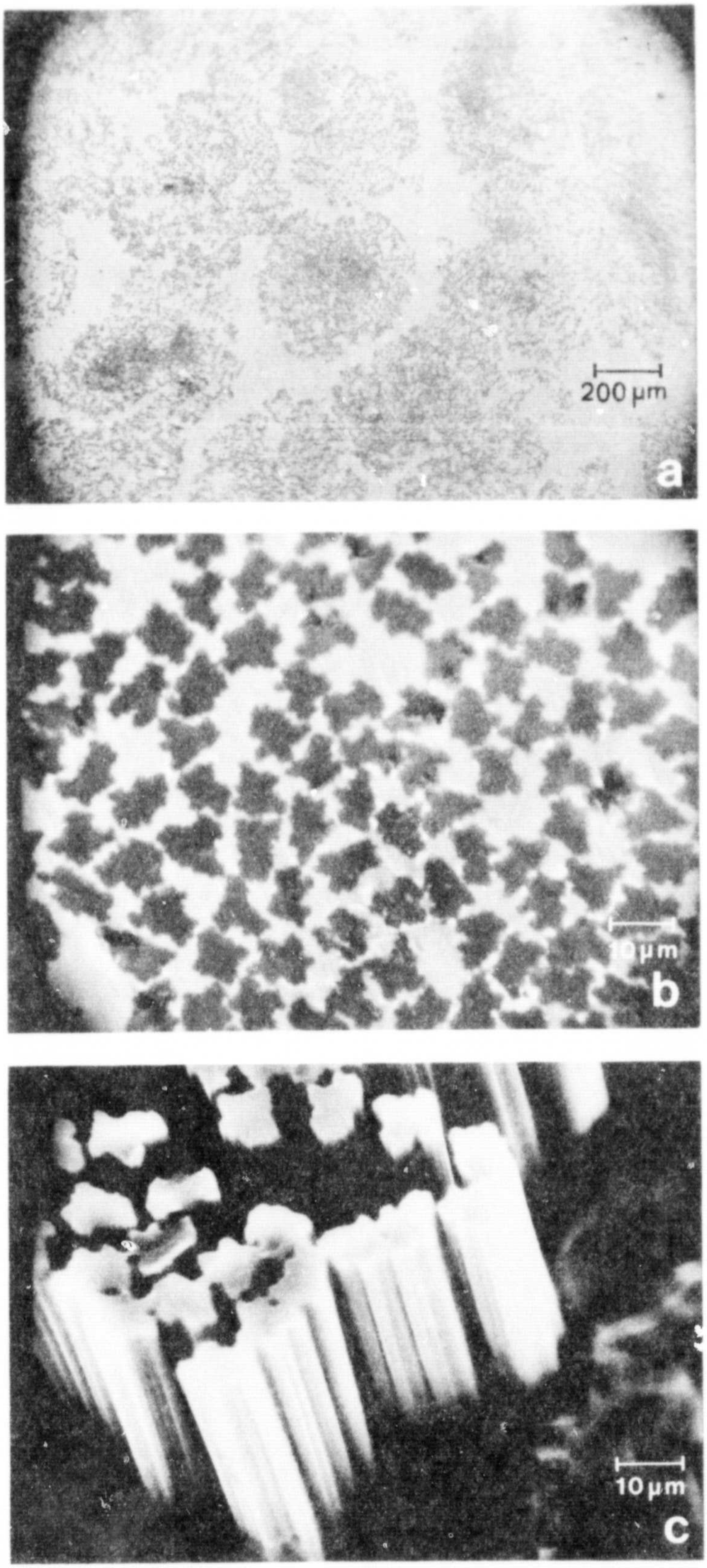

Fig. 6 - Microstructures of a longitudinal Al-Gr composite specimen: (a) polished transverse section, (b) enlarged view of a section of (a) illustrating fiber distribution, and (c) the polished surface after etching.

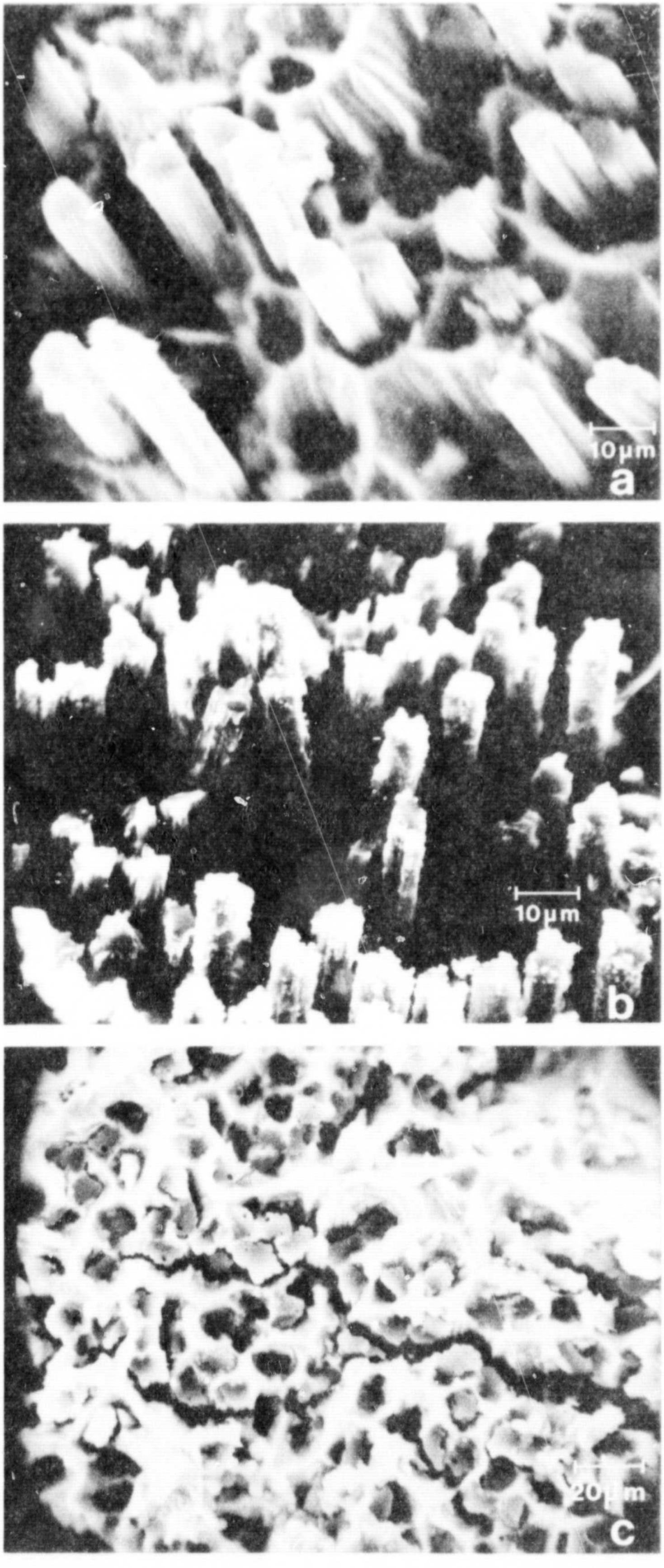

Fig. 7 - Microstructures of the fracture surface of a longitudinal Al-Gr composite: (a) the fracture surface of an as-received specimen, (b) the fracture surface of a specimen heat-treated at 600°C for 24 hr, (c) an area of (b) showing crack propagation.

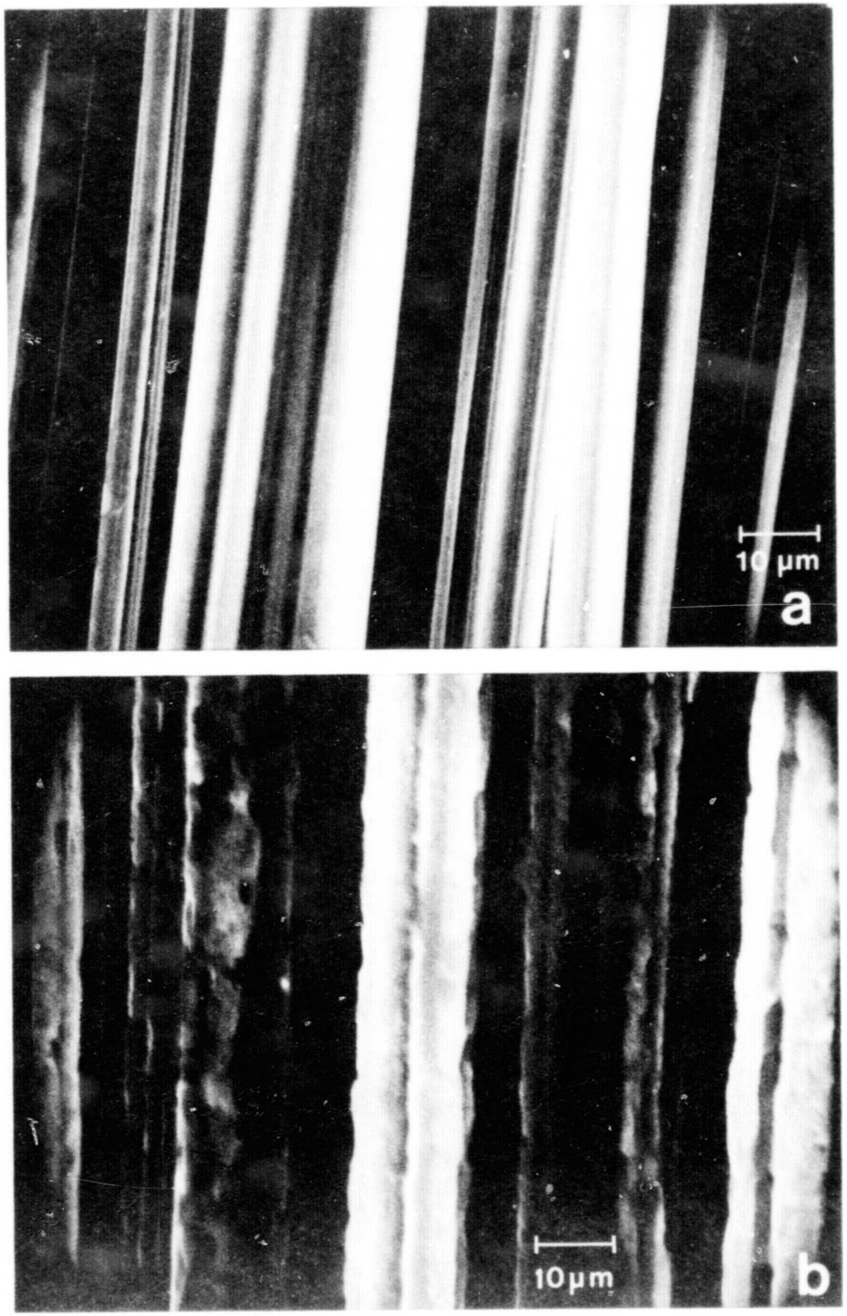

Fig. 8 - Microstructures of graphite fibers (a) before and (b) after chemical reaction at 600°C for 24 hr. (Specimens prepared by etching of the Al matrix.)

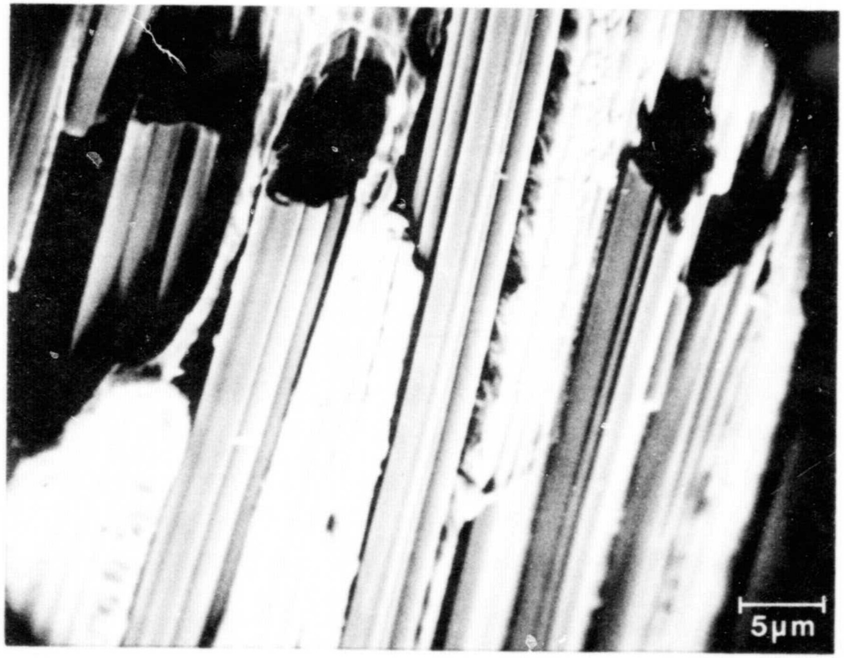

Fig. 9 - Scanning electron micrograph of the fracture surface of a transverse Al-Gr composite specimen.

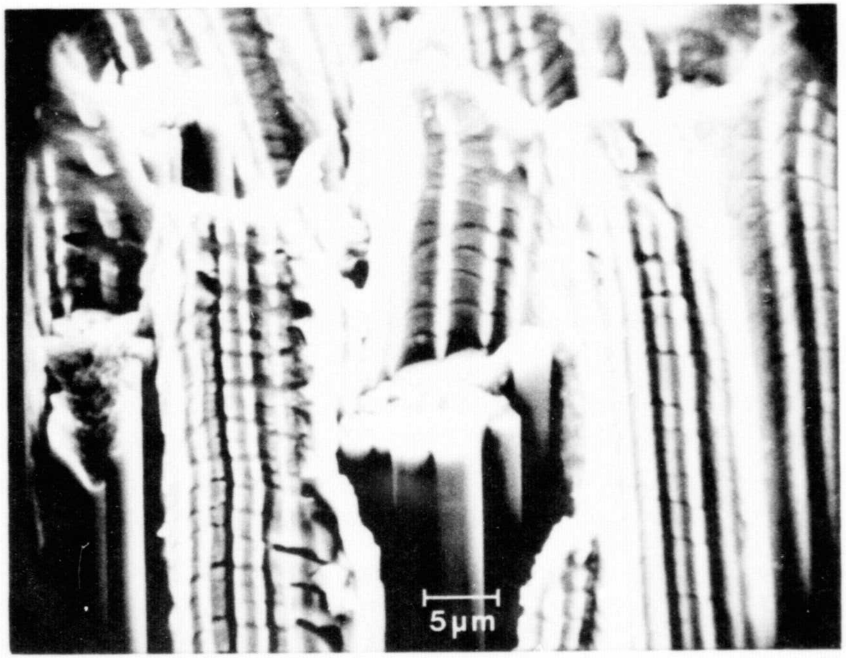

Fig. 10 - The fracture surface of a longitudinal aluminum-graphite specimen, exhibiting Al matrix microstructure.

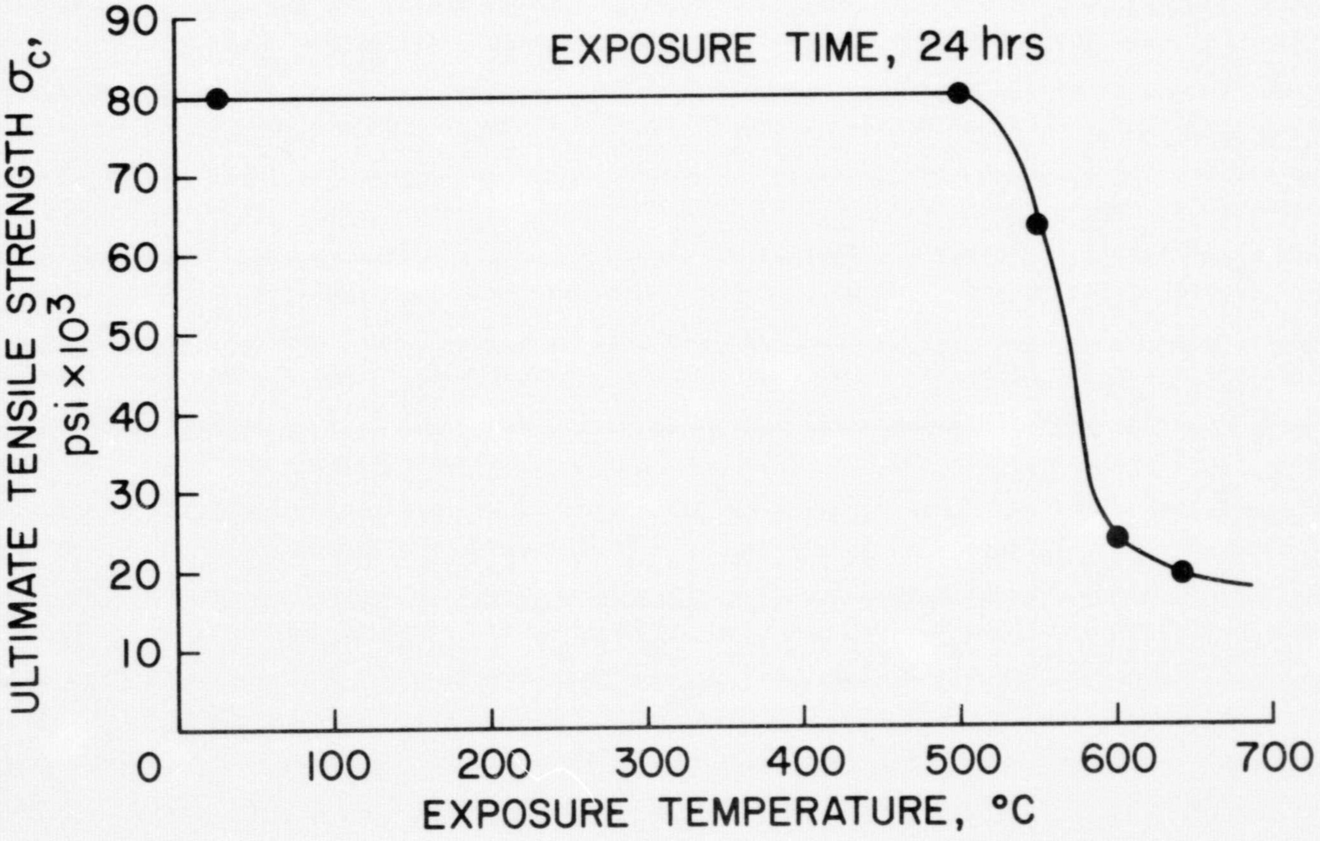

Fig. 11 - The tensile strength of 6061 Al-Gr composites as a function of temperature.

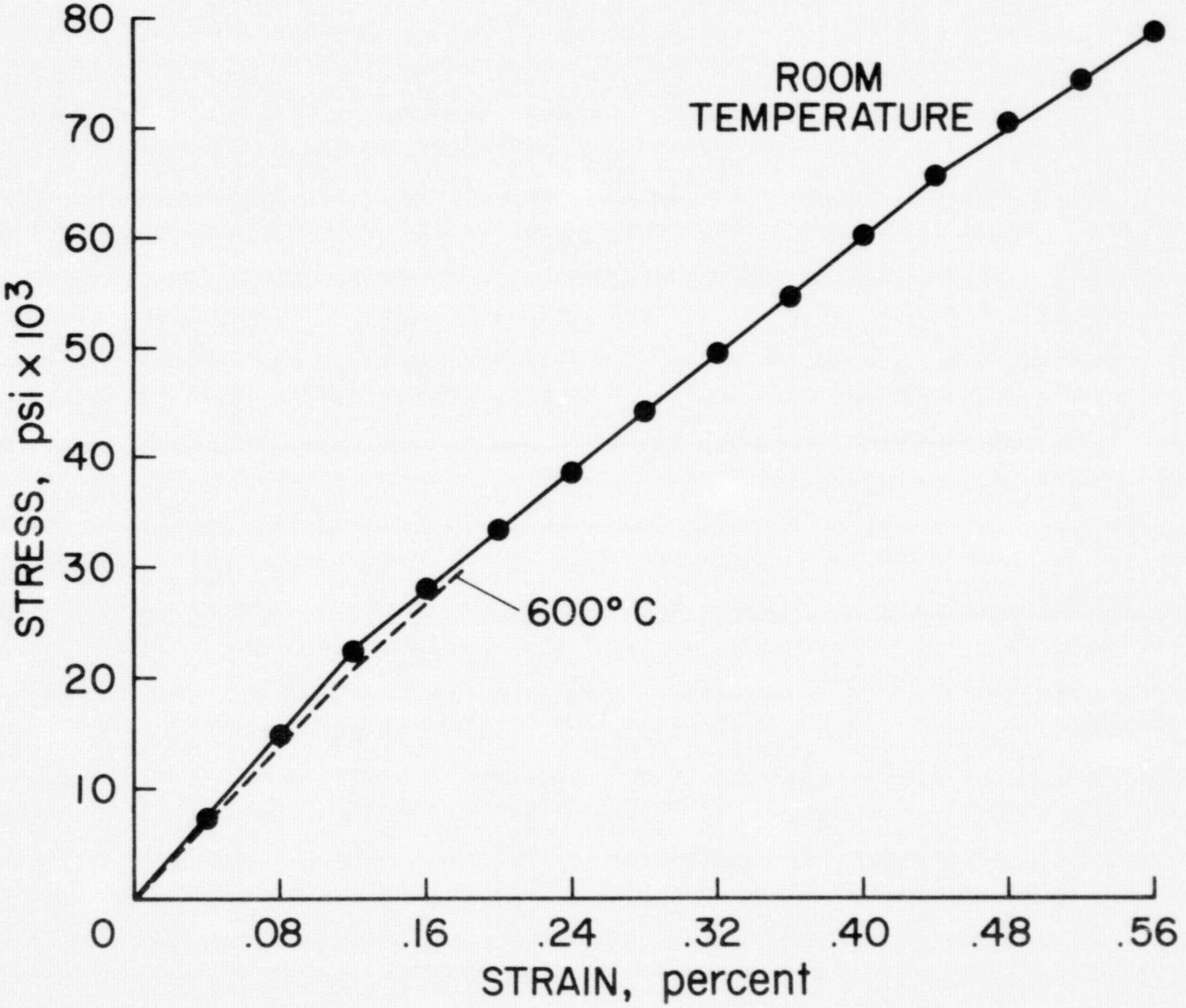

Fig. 12 - Representative tensile stress-strain curves for Al-Gr composites.

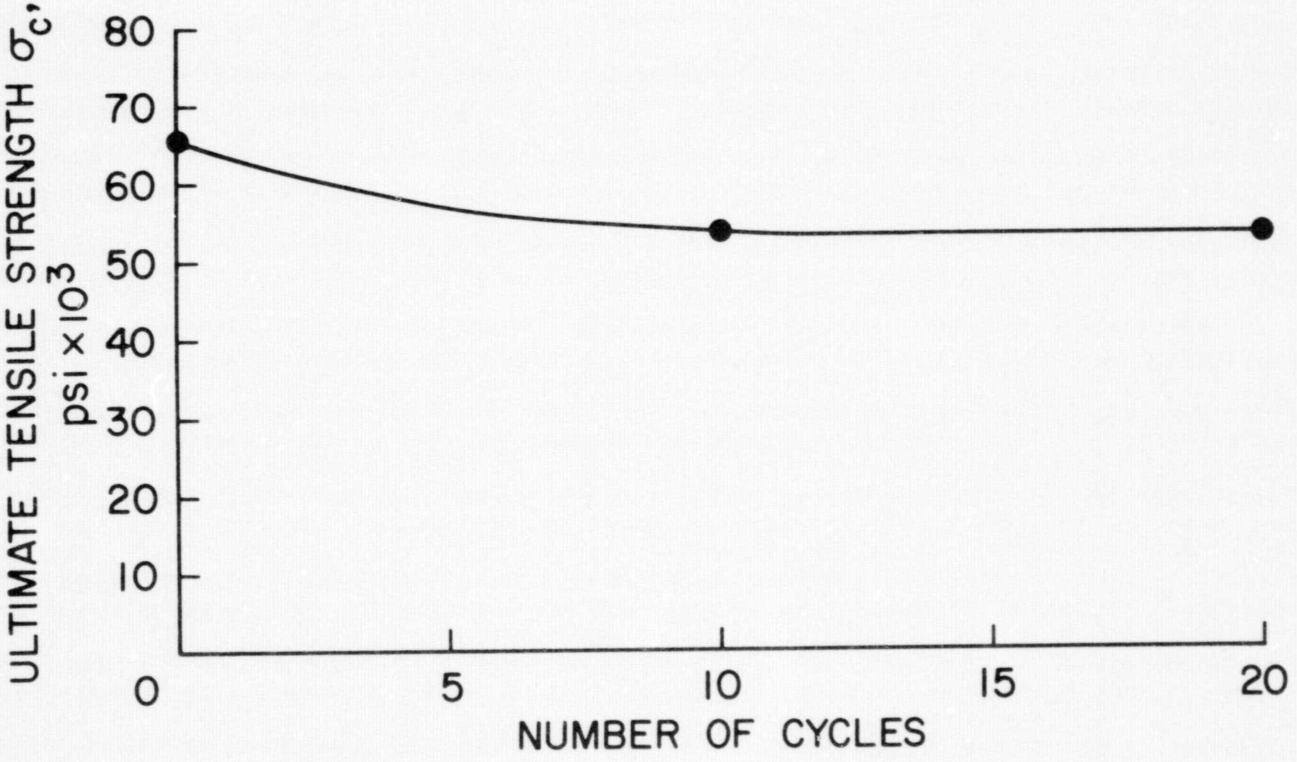

Fig. 13 - The effect of thermal cycling (20° - 500°C) on the tensile strength of Al-Gr composites.

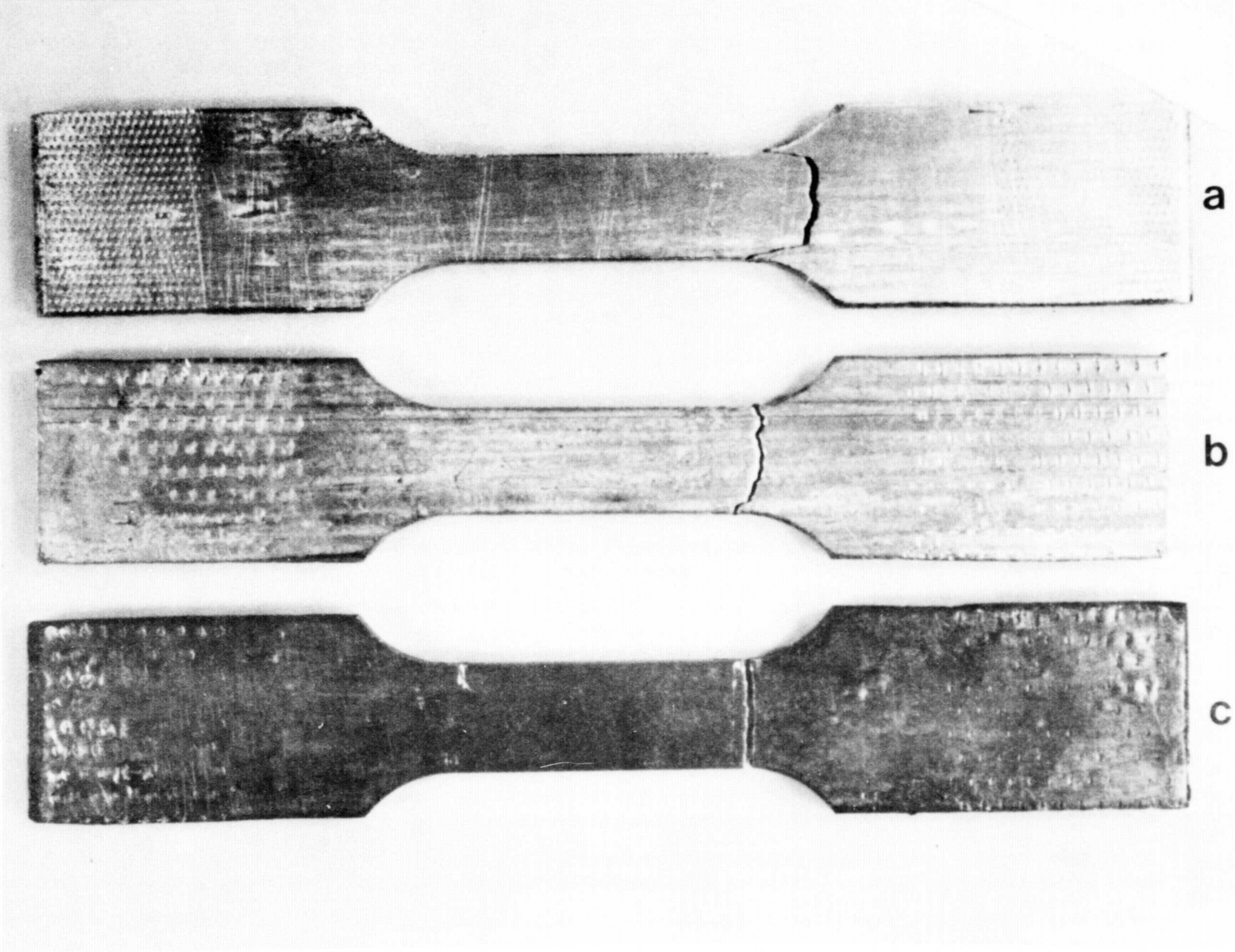

Fig. 14 - Optical micrographs of Al-Gr composite specimens showing nature of failure: (a) at room temperature (as-received specimen), (b) heat-treated at 550°C for 24 hr, and (c) heat-treated at 600°C for 24 hr.