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Short carbon fiber-aluminium matrix composite material prepared by extrusion of powder mixtures

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ABSTRACT

Unidirectional short carbon fiber/aluminum matrix composites have been prepared by hot extrusion of fiber/powder mixtures. Effects of processing parameters on the fiber length and the interfacial reaction between fibers and aluminium have been studied. Composites with different fiber volume fraction (up to 15 vol. %) were tested on their longitudinal stiffness and tensile strength at room as well as elevated temperatures to investigate the strengthening effect caused by addition of fibers into the matrix material. An experiment of annealing of the composites was done to study the influence of annealing time and temperature on the interfacial bonding strength and on the amount of reaction product (Al_4C_3) at the interface.

INTRODUCTION

Carbon fibers (CF) possess highest specific stiffness compared with other ceramic reinforcements. In addition, they are commercially available at relative low cost and they are not health hazardous. These properties makes them attractive as a reinforcement for light metals such as aluminum and its alloys. However, interfacial reaction at higher temperatures requires the protection of the fibers, especially when the composite is prepared using liquid matrix [1], which makes the fabrication of composites difficult and expensive. In order to develop a cost effective manufacturing technique, the ideal solution would be the elimination of the need for a fiber coating via minimization of the interfacial reaction by reducing the contact time between fibers and metal at extremely high temperature. This demand can be accomplished by extrusion of fiber/powder mixtures because it involves a solid state process and allows very high compacting rates. Moreover, the extrusion provides a method for direct production of various long profiles in which the longitudinal properties are dominant. The reinforcing fibers can be easily aligned in this direction. However, the major drawback of powder metallurgical techniques is extreme fiber damage during processing resulting in significant reduction of reinforcing efficiency [2,3]. The aim of this study is to investigate the structural and mechanical properties of short CF/Al-matrix composites prepared by powder metallurgical techniques which have been optimized to minimize the fiber shortening and the interfacial reaction between the constituents.

EXPERIMENTAL

Commercially pure Al (ECKA AS011/S) and age hardenable 6061-aluminum alloy powders (mean size $\sim 32 \mu\text{m}$) were employed as a matrix material. Powder and short carbon fibers Courtaulds Grafil HMS (density $\sim 1.87 \text{ g/cm}^3$, ultimate tensile stress $\sim 3 \text{ GPa}$, Young's modulus $\sim 390 \text{ GPa}$, diameter $\sim 6.5 \mu\text{m}$, mean length $\sim 1 \text{ mm}$) were wet mixed, then unidirectionally oriented and hot pressed into billets perpendicularly to the fiber orientation, in Ar-atmosphere. The billets were hot extruded at a ram speed of 3 cm/s using a round die (extrusion ratio 1:16, die entrance angle 90°). Some of the aligned fiber-powder mixtures were

canned into Al-cans and hot extruded without pre-densification as loose mixtures. The extruded 6061-matrix composites were heat treated under T6 conditions (solutioning at 530°C, water quenching and annealing at 177°C for 8 hours). The fiber length in the extruded composite was determined from the micrograph of the filter cake after first dissolving the matrix in HCl. Approximately 200-300 fibers were measured for each sample. Tensile strength measurement was performed on an Instron™ testing machine at a cross-head speed of 0.1 cm min⁻¹. The shoulder-type specimen was held at the test temperature with the accuracy of ±2K for 10 min. In order to stabilize the matrix structure at the testing temperature, each specimen was annealed for 24 hours in an external furnace at this temperature, then furnace cooled and immediately prior to testing again reheated. An electrical extensometer with a gauge length of 25 mm was connected to the specimen during tensile testing at room temperature. Young's modulus was determined from the linear part of the stress-strain diagram. A linear curve was obtained by reversing the cross-head motion at the strain of 0.1%. The amount of aluminum carbide was computed from the H₂:CH₄ ratio determined by gas chromatography after dissolving the composite in NaOH.

RESULTS AND DISCUSSION

Structure of the extruded composite

Fibers in extruded bars are relatively uniformly distributed and well aligned unidirectionally into the extrusion direction (Fig. 1). Satisfactory fiber distribution has been obtained for fiber volume fractions up to 0.15. Higher fiber fractions resulted in improper embedding of the fibers in the matrix.

Fiber length. Fiber shortening during composite preparation is the major shortcoming of the applied fabrication method. Fibers long initially about 1 mm (mean aspect ratio ~150-160) have been fragmented into short lengths in following processing sequence:

- i) almost no shortening during wet blending, orienting and drying (l/d 140-150)
- ii) moderate shortening during hot pressing, depending on pressing temperature and pressure (l/d 80-120)
- iii) extreme shortening during hot extrusion (l/d 5-50)

Detrimental fiber damage occurs during onset of extrusion due to a significant level of shear stresses arising in the extrusion die [4]. With increasing temperature the matrix shear stress decreases resulting thus in less fiber breakage (Fig. 2). More intensive fiber damage during extrusion of hot pressed billets, compared to those using loose mixtures, can be attributed to the embrittlement of the fiber-matrix interface in the billets caused by chemical reaction between Al and C-fibers during hot pressing and heating before extrusion. Composites with a fiber aspect ratio up to 50 (Fig. 3) can be prepared using extrusion temperatures in the semi-solid range (6061-matrix).

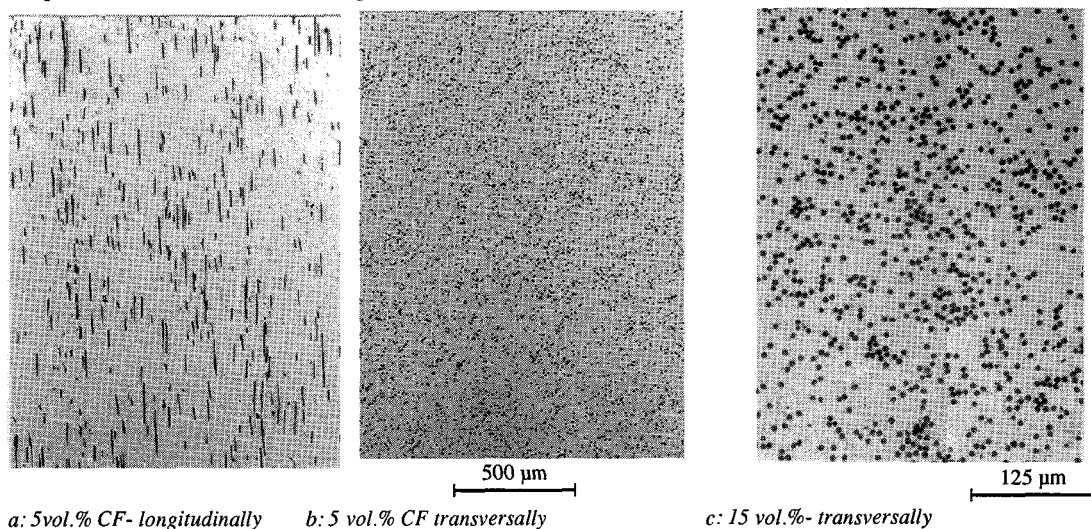


Fig.1 Cross sections of extruded Al-matrix composites

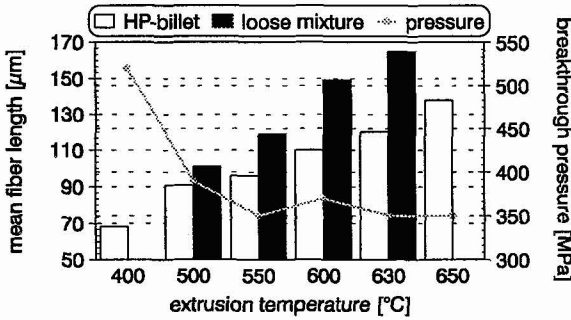


Fig. 2 Mean fiber length and extrusion breakthrough pressure at different extrusion temperatures

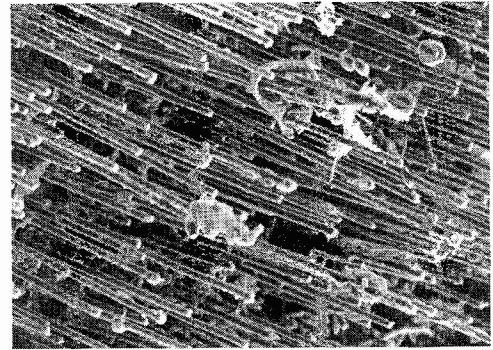


Fig.3 SEM of extruded 6061-matrix/ 10 vol.% CF composite (deep etched)

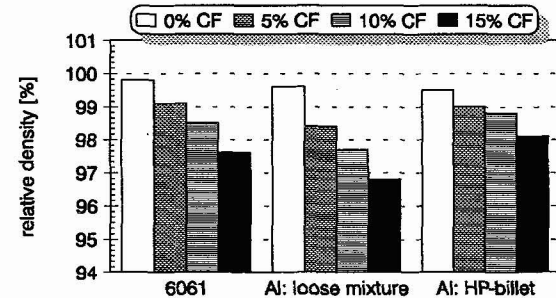


Fig.4 Relative density of extruded composites

Relative density of the composites (ratio of the experimentally achieved density to the theoretical one) decreases with increasing fiber volume fraction (Fig. 4). The effect of the matrix can also be noticed. Partially liquid 6061-matrix possesses more fluidity than solid Al-matrix and allows thus better embedding of fibers which results in higher final density. Composites extruded from hot pressed billets exhibit slightly higher densities than those extruded from loose mixtures. This can be explained as follows: The true densification of the composite is completed after

elimination of free interparticle (fiber) surfaces. This process is driven by diffusion and is to a certain degree realized during hot pressing. Assuming that the diffusion controlled increase of density during extrusion is the same for both hot pressed and loose mixture billets, the final density of composites extruded from hot pressed billets should be higher.

Interfacial reaction.

Aluminum carbide amounts determined in composites prepared by different methods are listed in Table 1. Almost no interfacial reaction has been observed in composites extruded from loose mixtures. The chemical reaction between fiber and matrix during heating of loose mixture has been precluded by the presence of inactive alumina on the powder surface. Suitable conditions for interfacial reaction are created only after application of sufficient deformation on mixture, e.g. during upsetting stage of extrusion. As the extrusion is relatively fast it cannot provide sufficient time for diffusion and significant carbide growth. This leads to an assumption that the interfacial reaction in composites extruded from hot pressed billets takes place predominantly during hot pressing and heating of the billets to extrusion temperature. Higher carbide quantities in 6061 matrix composites were caused by the presence of liquid phase in this matrix material during extrusion. Liquid phase dissolves a certain amount of carbon and provides better conditions for diffusion and hence more intensive interfacial reaction than the solid phase.

Table 1 Aluminum carbide in short carbon fiber reinforced composites
 extrusion: temperature-T
 hot pressing : temperature-T, time-30 min, pressure-16,6 MPa.

composition	fabrication	T [°C]	Al ₄ C ₃ -amount [wt.%]
Al+5 vol.% CF	hot pressed	600	0.25
Al+5 vol.% CF	hot pressed+extruded	630	0.42
Al+5 vol.% CF	extruded from loose mixture	635	0.05
Al+10 vol.% CF	extruded from loose mixture	640	0.11
6061-5 vol.% CF	hot pressed	600	0.54
6061-5 vol.% CF	hot pressed+extruded	640	0.56
6061-5 vol.% CF	extruded from loose mixture	635	0.18

Mechanical properties

Young's modulus of composites determined parallel to the fiber alignment is shown in Fig. 5. As expected it increases with increasing fiber volume fraction. Despite shorter fiber lengths the longitudinal stiffness of composites extruded from hot pressed billets is higher than that of composites extruded from loose mixtures. This effect can be attributed to the improved fiber-matrix bonding due to the higher degree of interfacial reaction in these composites. The experimentally measured moduli attain almost the theoretical values computed according to the "Halpin-Tsai" equation [5] considering also the effect of residual porosity.

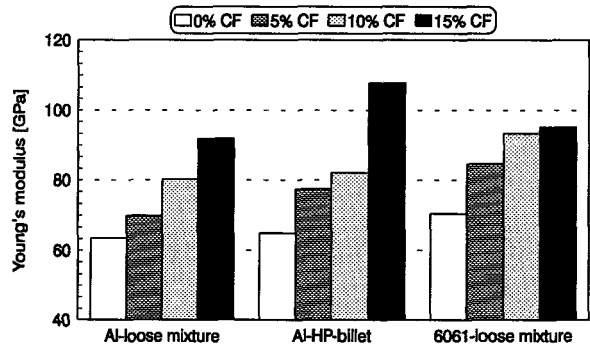


Fig.5 Longitudinal Young's modulus of extruded composites for different fiber volume fraction

Ultimate tensile stress at room and elevated temperature of composites extruded from loose mixtures is summarized in Table 2. The strength of Al-matrix composites increases with increasing fiber volume fraction V_f , although the reinforcing efficiency is low. Insufficient fiber strengthening is due to the weak bonding between fibers and matrix. According to previous calculations [6] the average interfacial shear stress does not exceed the value of 25 MPa. Since this value is considerably lower than the matrix shear yield stress τ_y , the load transferred from the matrix onto the fiber remains almost unchanged up to 300°C. It results in increasing efficiency of reinforcement with increasing temperature, because the matrix strength decreases. At higher temperatures τ_y significantly decreases, becomes smaller than the interfacial bonding strength and begins to control the load transfer. This leads to the slight reduction of reinforcing efficiency. Moreover, the interfacial reaction between fiber and matrix takes place at temperatures above 400°C, resulting in substantial reduction of the fiber strength. This has been confirmed by measurement of lengths of fibers extracted from Al-matrix/10 vol.%CF composite after tensile testing. Fiber lengths in composites tested at temperatures up to 400°C were about the same as those measured before testing (mean lengths ~130-150 μm), while the fibers in composites tested at 500°C were notably fragmented (mean length ~71 μm). Weak interfacial bonding leads to high critical fiber length and thus in high minimal fiber volume fraction V_{min} (see [7]). As can be estimated in Fig. 6, V_{min} for Al-matrix composites belongs to the range of (0.1-0.15) at 20°C, (0.05-0.1) at 100°C and (0-0.05) at >100°C. Slight strengthening observed at the composite in which $V_f < V_{min}$ can be attributed to the changes in matrix structure [8].

Table 2 Ultimate tensile stress [MPa] of extruded short carbon fiber composites at different testing temperatures (average of at least 3 measurements)

matrix	Al						6061				
	20°C	100°C	200°C	300°C	400°C	500°C	20°C	100°C	200°C	300°C	400°C
0 vol.% CF	136	118	90	60	41	35	343	320	204	60	36
5 vol.% CF	146	130	110	85	61	38	325	304	220	63	45
10 vol.% CF	153	150	128	101	69	41	318	309	230	102	59
15 vol.% CF	166	155	132	106	79	46	288	277	215	116	81

Since the degree of interfacial reaction in 6061-matrix composites extruded from loose mixtures is similar to that in Al-matrix ones, the interfacial bonding strength in these composites is approximately equal. It means that the substitution of Al by the age hardened Al-alloy-matrix, which possesses high yield stress, cannot increase the load transferred from matrix into the fibers. As the age hardened 6061-alloy exhibits much higher strength than Al, the fiber volume fraction of 0.15 does not exceed the minimal value needed for fiber strengthening at room temperature. Similar behavior can be expected at 100°C, since the matrix

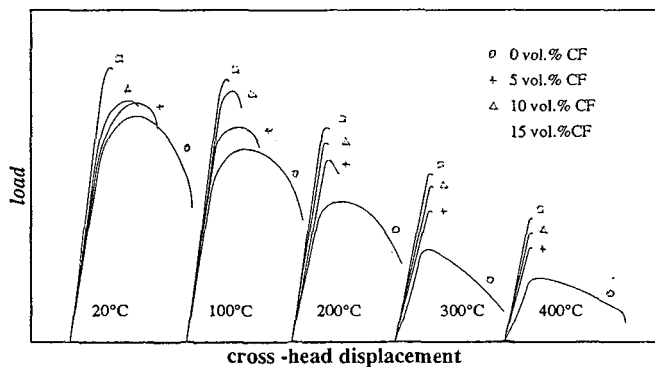


Fig. 6 Load-displacement curves recorded during tensile testing of Al-matrix/CF composites at different temperatures

strength reduction is not very large. However, the significant matrix weakening takes place above 200°C, due to overaging and recovery. It results in the reduction of V_{min} assuming that the load transferred by fibers remains unchanged. At higher temperatures (>300°C) practically no difference in the strength of composites with both matrices exists. In contrast to Al-matrix composites the addition of fibers up to V_{min} do not contribute to the strength of age hardened 6061-alloy (T6). Even a slight strength reduction can be observed (after normalizing by matrix volume

fraction). This reduction can be attributed to the dislocation density gradient [9], residual stresses and to the residual porosity.

The weak fiber/matrix bonding in composites extruded from loose mixtures can be improved by controlled interfacial reaction. An annealing experiment was done with Al-matrix/10 vol.%CF composites extruded from loose mixture, in order to verify this assumption. The experiment was performed as follows; the composites were annealed at 600°C in air for different times, then furnace cooled and tensile tested. After tensile test the fragmented fibers were extracted from the fracture surface by dissolving the matrix in HCl, and measured in order to determine the critical fiber length. Comparison of the fiber lengths after tensile test with initial lengths (before testing) has revealed that the fibers in unannealed or in for short time annealed composites were not fragmented during tensile testing. It confirms the assumption that the fibers had not attained their critical lengths. However, significant fragmentation of fibers was observed in composites annealed for longer time (Fig. 7) The fibers were fragmented into short lengths, between $l_c/2$ and l_c . Critical fiber length l_c was then obtained from the mean length of fragments l_m ($l_c = 4l_m/3$). The results are summarized in Table 3.

Table 3 UTS, elongation A_5 , aluminum carbide amount Al_4C_3 and critical fiber aspect ratio l_c/d in Al-matrix/10 vol.% CF composites extruded from loose mixtures and annealed at 600°C for different time extrusion temperature-650°C; l_m/d - fiber aspect ratio in composites after tensile test (initial $l/d = 20$)

annealing time [min]	UTS [MPa]	A_5 [%]	Al_4C_3 [wt.%]	l_m/d	l_c/d
0	152	12.5	0.110	21.0	>21.0
60	177	9.5	0.335	20.3	>20.3
120	171	11.3	0.465	-	-
300	167	10.2	0.773	11.5	15.3
600	167	13.1	1.051	8.5	11.3
1140	168	13.3	1.171	6.3	8.4

The critical fiber length decreases with increasing annealing time at 600°C, due to improving of interfacial bonding strength. The strength of composite should then increase. However, this was not observed. Considerable reduction of fiber strength caused by interfacial reaction was probably the main reason for this discrepancy (reduction of fiber strength leads also to the reduction of critical fiber length). Masson *et al.*[10] have reported the strength of HM-35 carbon fibers coated with aluminum being 1762±352 MPa after annealing at 650°C for 60 min (initial fiber strength was 4275±793 MPa). It means that the positive effect of fiber/matrix bonding improvement due to annealing has been diminished by reduction of the fiber strength, resulting in low tensile stress born in the fibers. Fiber strength is maximum in the first stage of annealing. However, insufficient bonding in this case leads to high critical length. This length could not be achieved by extrusion. The effect of increasing bonding strength, after annealing time up to 60 min, exceeds the negative influence of fiber weakening at the same time, which results in slight strength

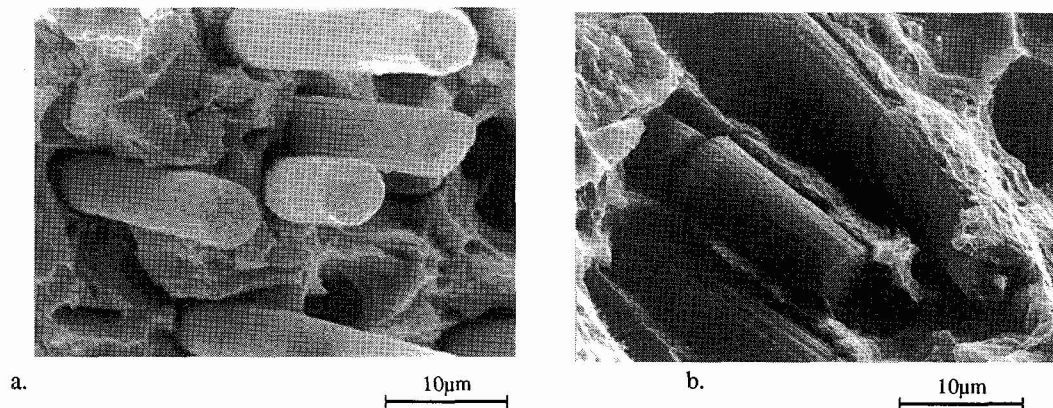


Fig. 7 Fracture surfaces of extruded Al-matrix/CF composites before (a) and after annealing at 600 °C for 10 hours (b)

improvement. However, after this period significant fiber weakening takes place, the effect of fiber strengthening becomes less pronounced and the strength of the composite is controlled mostly by the matrix strength.

It should be noted that the interfacial shear stress τ is not constant along the fiber length. It is likely that the interfacial bonding strength in annealed composites is higher than average τ . The fiber can be stressed up to its strength, then it will break with simultaneous reduction of the average axial tensile stress. During further loading the fiber debonding occurs, if the fiber length is shorter than critical one. The roughness of the debonded fiber surface increases with annealing time (Fig. 7), causing higher frictional stresses on the interface during further deformation and thus resulting in an increase of the fiber axial tensile stress. If this stress attains the fiber strength, the fiber fails again. Following this mechanism, the fiber can be continuously fragmented into short lengths if the matrix is able to withstand increasing stress after fiber fracture (e.g. if $V_f < V_{min}$).

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