

Military Technical College
Kobry El-Kobbah
Cairo, Egypt



10th International Conference
On Aerospace Sciences &
Aviation Technology

MECHANICAL PROPERTIES AND FRACTURE MECHANISMS OF AL-METAL MATRIX COMPOSITES REINFORCED WITH STAINLESS STEEL FIBERS

SHAMEKH^{*} M. E., RABEEH^{**} B. M., SALLAM^{***} M. T.

ABSTRACT

This work investigates the hot pressing technique, to produce Al-metal matrix composites (AL-MMC) reinforced with stainless steel fibers. The effect of hot pressing parameters, temperature, holding time, and pressure, was studied from microstructural point of view. Moreover microchemical analysis was carried out for the interdiffusion zone to predict the intermetallic compounds formed through it. Finally, the mechanical properties of the composites and the fracture mechanisms under continuous/incremental monotonic loading were studied. The results revealed that the Al-MMC samples that were prepared at T=640 °C, t=0.25 hr, and P=65 MPa gave an optimum thin continuous and uniform interdiffusion zone and hence the optimum mechanical properties. This interdiffusion zone consists of different distinct phase layers of different intermetallic compounds. Failure analysis proved that damage is initiated early after loading at outer plies and propagates through inner plies until catastrophic failure occurs. The governing damage failure modes are matrix cracking, fiber debonding, and delamination.

KEYWORDS

Composite, Hot Pressing, Interdiffusion zone, Intermetallic compounds, Monotonic, Debonding, Delamination.

^{*}Graduate Student, Dpt.of Material Science and Technology, M.T.C.

^{**} Assistant Prof. , Abha univeristy, Saudi Arabian Kingdom

^{***} Prof. , Dpt.of Material Science and Technology, Military Technical College

INTRODUCTION

During the last two decades, composite materials have been used in many applications, especially in the aerospace and automotive industries. Most of the commercial work on MMCs has focused on aluminum as the matrix metal.

The combination of light weight, environmental resistance, and useful mechanical properties has made aluminum alloys very popular; these properties also make aluminum well suited for use as a matrix metal [1]. Applications of Al-MMCs include drive shafts, fan blades, and shrouds, springs, bumpers, interior panels, tyres, brake shoes, clutch plates [2].

Metal-Matrix Composites are gaining increasing attention for structural applications. However, the database relating to their processing techniques, mechanical properties and microstructural characterization remains limited especially in the developed countries. Also, whereas most composites are heterogeneous and anisotropic, the relationships between forces and deformations are much more complicated for anisotropic composites than they are for conventional isotropic materials, and this can lead to unexpected behavior [3].

Also fracture mechanisms of these composites under different loading types are not completely understood. In this work, a method of producing metal matrix composite materials, which is the hot pressing technique, is used in producing composite specimens with aluminum as the matrix, and stainless steel as the fiber. Several factors affect this process and the resulting mechanical properties such as hot pressing parameters, fiber volume fraction, and composite architecture are studied. This was accomplished using various testing, such as the tensile test and scanning electron microscopy [4]. The specimens were loaded under monotonic loading in incremental steps to various fractions of the ultimate tensile strength [σ_u] measured previously to study the fracture mechanisms of unidirectional [0]₅ Al-MMCs reinforced with stainless steel fibers.

EXPERIMENTAL WORK

The samples of AL-MMC material reinforced with stainless steel fibers were produced using the Hot Pressing technique based on matrix of commercially pure aluminum foils (0.5 mm thickness) and fibers of common austenitic stainless steels 0.2-mm diameter class under the designation of the series 304 (18/8). The chemical composition and physical and mechanical properties of these aluminum foils and fibers are given in tables 1, 2, 3, and 4 respectively. To achieve the preparation of the composite samples using the Hot Pressing technique, a custom die specially made was designed to hold the desired arrangement of fiber/matrix materials during processing as shown in Fig. (1). In this technique, five layers of aluminum foils were stacked along with fibers in a unidirectional architecture ([0]₅) in the specific fiber volume fraction as shown in Fig. (2). Different samples were prepared at constant fiber volume fraction ($V_f = 7.6\%$) at different temperatures from 500 to 640°C with 20°C step, different holding times ($t = 0.25, 0.5, 1, \text{ and } 2 \text{ hr.}$), and different pressures ($P = 13, 39, \text{ and } 65 \text{ MPa}$). Microstructure of these AL-MMC specimens was examined using SEM. Also, EDX and WDX analyses were applied on the best visually recorded cases where thin continuous and uniform interface was obtained. Standard flat tensile test specimens, under the designation: D 3552-77 [7] are used. Figure (3)

provides the dimensions of the used specimens. The first set of tensile specimens ([0]5 specimens prepared at constant $V_f=7.6\%$) was loaded continuously to failure. The second set of tensile specimens was prepared at the optimal hot pressing parameters, which gave the best mechanical properties at different fiber volume fractions. The third set of tensile tests was conducted on smooth specimens ([0]5) prepared at the optimal hot pressing parameters which gave the best mechanical properties and at $V_f=13.2\%$ to study the deformation and cracking phenomena associated with damage under uniaxial loading. The specimens were loaded in incremental steps, to various fractions (0.1, 0.2.... till 1.0) of the ultimate tensile stress $[\sigma_u]$ determined from the first set of tests. Surfaces of the incrementally deformed and fractured tensile specimens were examined in a scanning electron microscope (SEM) to determine the predominant fracture mode and to characterize the microscopic mechanisms governing tensile fracture.

RESULTS AND DISCUSSIONS

Microstructural Analysis

Wide range of temperatures from 500 to 640°C was tested. However, the temperatures below 600°C revealed a clear delamination and less filling with no clear interdiffusion zone so, the temperature was tested at a narrow range from 600°C to 640°C with a 20°C step. The results of hot pressing in this temperature range from 600°C to 640°C, for different holding times and pressures revealed that there is a complete bonding between the matrix layers without delamination and voids. Enough diffusion occurred between these layers till these matrix layers became a unit continuous block of material. There is a similarity of the formed interdiffusion zone (interphase) between the matrix and fibers. An optimum thin continuous and uniform interdiffusion zone can be obtained in the Al-MMCs reinforced with stainless steel fibers when they are prepared at $T=640^\circ\text{C}$, $t=0.25\text{ hr}$, and $P=65\text{ MPa}$ as shown in Fig. (4). It is clear that by increasing the temperature, the interphase thickness and its continuity are increased at constant holding time and pressure. Also, the same effect of the holding time was obtained but with different ratios. The pressure satisfies the enhancement of the diffusion process which is reflected on increasing the continuity of the interdiffusion zone and its thickness.

X-Ray Microchemical Analysis Using SEM

The results of EDX analysis for Al-MMC samples reinforced with stainless steel fibers prepared at $T=640^\circ\text{C}$, $t=0.25\text{ hr}$, and $P=65\text{ MPa}$ are presented at Fig. (5) showing line distributions of the different elements through the interphase layer.

The detailed structure of the interdiffusion zone, in the case of Al-MMCs, which were obtained after 0.25 hr, was difficult to be resolved by the SEM. That is because the formed intermetallic compounds layers are very thin and when using a probe of size $3\mu\text{m}$ in the EDX analysis, more than one layer of these intermetallic compounds can be detected in the same time, so the variation between these layers is very difficult. By increasing the holding time, the thickness of the interphase layer is increased and so is the thickness of each intermetallic compound. Moreover, a complex interdiffusion zone containing distinct phase layers was produced so, WDX and further EDX analyses were then used to detect these structures in greater details especially after 2 hr where a concentric ring structure developed around the fiber. As

shown in figures (6) and (7), it can be noted that the interdiffusion zone consists of different distinct phase layers of different intermetallic compounds. From the Al-Fe phase diagram, it can be stated that there are different intermetallic compounds formed from iron and aluminum. These intermetallic compounds and the Al-atomic % of each stoichiometric compound are presented in Table (5).

A comparison between the concentrations of Al at different regions through the interdiffusion zone obtained by WDX analysis and the average Al-atomic % of each stoichiometric iron-aluminate intermetallic compound shown in Table (5) was carried out. It is found that the probable formed intermetallic compounds through the interdiffusion zone can be predicted as shown in Table (6).

Mechanical Properties of The Al-MMC

The mechanical properties such as tensile strength (σ_{ut}), proof stress ($\sigma_{0.2}$), and ductility ($\delta\%$) of the unidirectional laminate composites $[0]_5$ at constant volume fraction ($V_f=7.6\%$) and different hot pressing parameters such as temperature, pressure, and holding time will be presented and discussed in the following paragraphs. Tables (7, 8, and 9) summarize the fiber/matrix interface characteristics and the tensile test results of the yield strength, ultimate strength, and ductility for AL-MMCs which were prepared at different temperatures and holding times and under constant pressures of 13, 39, and 65MPa respectively. The effect of the hot pressing temperature and holding time on the tensile strength of these samples are presented in figures (8, 9, and 10). It can be noted that by increasing the holding time, the ultimate strength, proof stress, and ductility are decreased but with different percentages. For example, increasing the holding time from 0.25hr to 0.5hr at a constant temperature of 640°C and pressure of 65 MPa results in a decrease by about 10% in the ultimate strength and by about 13% in the proof stress while ductility is decreased by about 12.6%. On the other hand, when the holding time is increased from 0.5 hr to 1 hr, the ultimate strength is decreased by only about 1.9% and the proof stress by about 3.85% while ductility is decreased by about 11.7%. Moreover, increasing the holding time from 1 hr to 2 hr results in a decrease by about 1.4% in the ultimate strength and by about 1.1% in the proof stress while ductility is decreased by about 11.3%. This can be attributed to the strong dependence of the interphase layer thickness on holding time. Also the effect of temperature was studied. It was shown that increasing the temperature from 600°C to 620°C at a constant holding time of 0.25 hr increases the ultimate strength by about 2.53% and the proof stress by only 1% while ductility is decreased by about 5.1%. On the other hand, by increasing the temperature from 620°C to 640°C, the ultimate strength is increased by about 3.9% and the proof stress by 2% while ductility is decreased by about 12.8%. That is because AL-MMCs that are prepared at $T=640^\circ\text{C}$ and $t=0.25$ hr secure the optimum morphology and thickness of the interphase layer that ensure the strong bonding between the fiber and matrix so, they have a higher strength but lower ductility than that are prepared at $T=620^\circ\text{C}$ and the same time. On the other hand, it is found that increasing the temperature from 600°C to 620°C at a constant holding time of 0.5 hr decreases the ultimate strength by about 1% and the proof stress by only 2% while ductility is decreased by about 13.5%. Nearly the same behavior occurs at 1 hr and 2 hr but with different percentages. From these results, the effect of the hot pressing pressure can be visualized. For example the ultimate strength is increased by about 1.1% and the proof stress by 2.5% while ductility is

decreased by about 4.8% when the value of pressure is changed from 13 MPa to 39 MPa at a constant temperature of 600°C and holding time of 2 hr. On the other hand, when the pressure is increased from 39MPa to 65MPa, the ultimate strength is increased by about 1% and the proof stress by 1.3% while ductility is decreased by about 2.8%. In fact the difference in the thickness of the interphase layer is very small but the main effect lies in the continuity of the interphase in the three cases.

Mechanical Properties of AL-MMCs at Different Fiber Volume Fractions

Stress-Strain diagrams of AL-MMC samples prepared at $T=640^{\circ}\text{C}$, $t=0.25\text{hr}$, and $P=65\text{MPa}$ for different fiber volume fractions are shown in Fig. (11) and the mechanical properties are presented in Table 10. An important parameter controlling the properties of a fiber reinforced composite material is the fiber volume fraction. It is found that by increasing the fiber volume fraction (V_f), the ultimate tensile strength (UTS) and the proof strength are increased while ductility is decreased. That is because increasing the fiber volume fraction means the increase of the load-carrying components (fibers) so, the strength is increased but also means the increase the formation of brittle intermetallic compounds of iron and aluminum through the interdiffusion zone between the fiber and matrix ,hence, ductility is decreased.

Fracture Mechanisms Under Monotonic Loading

When the specimens were loaded in incremental steps, to various fractions (0.1,...till 1) of the ultimate tensile stress [σ_u], damage phenomena associated with incremental loading were symmetrically determined from ex-situ examination of the gauge section of the incrementally deformed test specimens in a scanning electron microscope. Samples for SEM observations obtained from the deformed specimens by sectioning parallel to the fracture surface. When the test specimens were monotonically loaded at a load level corresponding to $0.2 \sigma_u$, it was observed that low percentage of debonding along the fiber/matrix interface was occurred in the outer plies, while no clear damage evidence in the inner ply as shown in Fig. (12).

With progressive loading to a level corresponding to $0.4 \sigma_u$, the percentage of debonding along the fiber/matrix interface was increased in the outer plies till a complete separation between the fiber and matrix was occurred. On the other hand, the inner plies were subjected to low percentage of debonding along the fiber/matrix interface as shown in Fig. (13). This mode of fracture is a brittle because it occurs mainly in the brittle intermetallic compounds (through the interdiffusion zone) and non-linear behavior of stress-strain diagrams was observed due to this debonding. Continued increase in uniaxial load to level corresponding to $0.5 \sigma_u$, matrix crack initiation was observed to occur in the outer plies. This fracture mode is a ductile one because it occurs mainly in Al matrix which is a ductile material. On the other hand, the percentage of debonding or separation size between the fiber and matrix in the inner plies was increased as shown in Fig. (14).

At a load level of $0.7 \sigma_u$, matrix cracks were nucleated by an extension of the interfacial cracks into the matrix but the crack size of one of them was increased. This was occurred initially in the outer plies, while nucleation of matrix cracks with complete debonding was occurred in the inner plies as shown in Fig. (15).

At a load level of $0.9 \sigma_u$, the initiation of matrix crack growth preceded final fracture through growth and eventual coalescence of the matrix cracks while, evolution of matrix cracks in the inner ply was observed as shown in Fig. (16). Moreover another fracture mode was observed called delamination which consists of the separation of

matrix layers from one another, after that fiber fracture was occurred. Finally catastrophic failure occurred by ductile dimpled fracture in the aluminum matrix. There was also evidence of fiber pull-out with the interfaces between the fibers and the matrix showing visible signs of degradation or damage during the pull-out process as shown in Fig. (17).

It can be noted that the damage modes in the inner ply occurs later than that occurs in the outer plies which are more stressed than the inner plies.

CONCLUSIONS

1. The production of Al-MMC by hot pressing technique is influenced by varying the process parameters, adequate bonding between the matrix layers without delamination can be obtained under a hot pressing temperature of 600 °C.
2. An optimum thin continuous and uniform interdiffusion zone can be obtained in the Al-MMCs when they are prepared at T=640 °C, t=0.25 h, and P=65 MPa.
3. It was found that the interdiffusion zone consists of different distinct phase layers of different intermetallic compounds.
4. There are a strong dependence between the resulting mechanical properties and the interdiffusion zone characteristics. AL-MMCs that have been prepared at T=640°C, t=0.25 hr, and P=65 MPa gave optimum mechanical properties.
5. Failure analysis proved that damage is initiated early after loading at outer plies and propagates through inner plies until catastrophic failure occurs. The governing damage failure modes are matrix cracking, fiber debonding, and delamination.

REFERENCES

- [1]. Joseph R. Davis, "Metals Handbook; Properties and Selection: Nonferrous Alloys and Special-Purpose Materials" Tenth edition vol: 2, ASM, 1990.
 - [2]. D. Charles, "Addressing the challenge of aircraft component design and manufacture from MMCs", J. Aerospace Engg. , p: 1-13, 1992.
 - [3]. Ronald F. Gibson; " Principles of Composite Material Mechanics ", McGraw-Hill. Inc, 1996.
 - [4]. B.M.Rabeeh, and H.M.Eltaher, "Characterization of different hot-pressed aluminum composite architectures", Proceeding of the 8th International Aerospace and Science Aviation Technology Conference, ASAT Conf. 4-6 May, Cairo, Egypt, 1999,.
 - [5]. J. R. Davis, "ASM Specially Handbook: Stainless Steels," ASM International Materials Park, Ohio, 1996.
 - [6]. P.K. MALLICK " Composites Engineering Handbook " Marcel Dekker, Inc. 1997.
 - [7]. Manual book of ASTM Standards, "Standard Test Method for Tensile Properties of Fiber-Reinforced Metal Matrix Composites", vol: 1-3, July 1989.
-

Table 1. Chemical Composition of the used commercial pure aluminum foils [1].

Al purity	Si	Fe	Cu	V	Mn	Mg	Ti
Min. 99.5%	Max. 0.25%	Max. 0.4%	Max. 0.05%	Max. 0.05%	Max. 0.05%	Max. 0.05%	Max. 0.03%

Table 2. Physical and mechanical properties of the matrix [1].

Elastic Modulus (GPa)	Yield Strength (MPa)	Tensile Strength (MPa)	Ductility (%)	Density (g/cm ³)	Coeff. Of thermal expansion (K ⁻¹)
70	28	76	39	2.7	28.1

Table 3. Chemical composition of the used austenitic stainless steel fiber [5].

AISI	% C	% Cr	% Ni	% S	% Si	% P	% Mn	% Fe
304	0.08	20.0	8.0	0.03	1.0	0.045	2.0	R

Table 4. Physical and mechanical properties of the fiber [6].

Elastic Modulus (GPa)	Tensile Strength (MPa)	Density (g/cm ³)	Coeff. Of thermal expansion (K ⁻¹)
198	1400	8.0	18.0

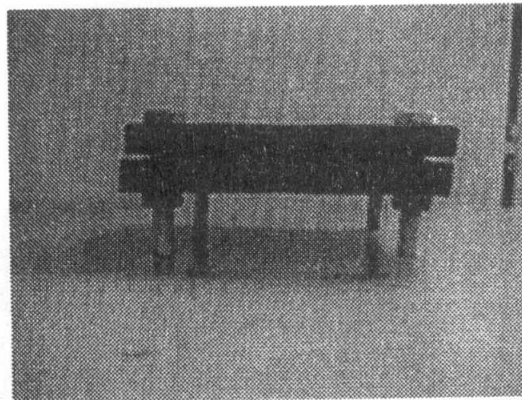


Fig.1. The used custom die for hot pressing.

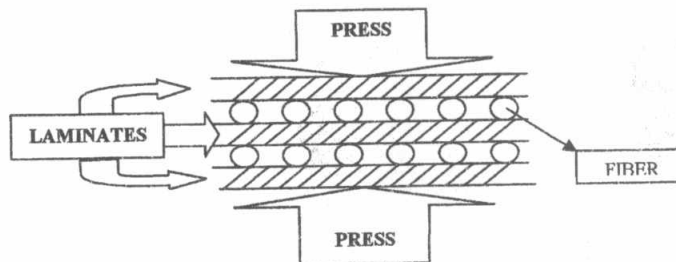


Fig.2. Schematic drawing of the hot pressing technique.

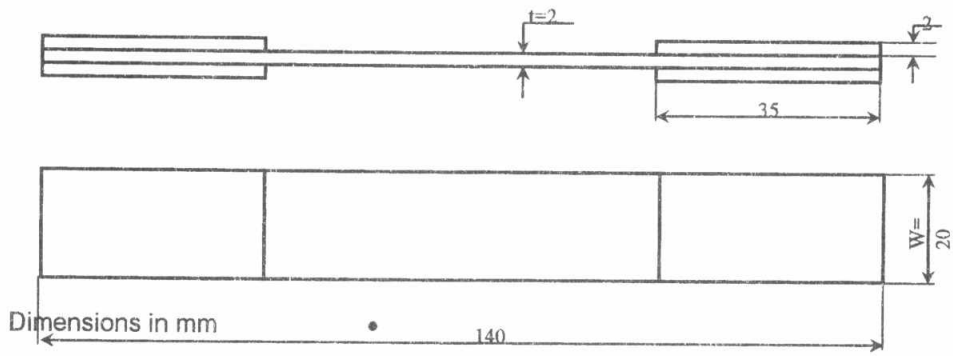


Fig.3. Standard flat tensile test specimen [7].

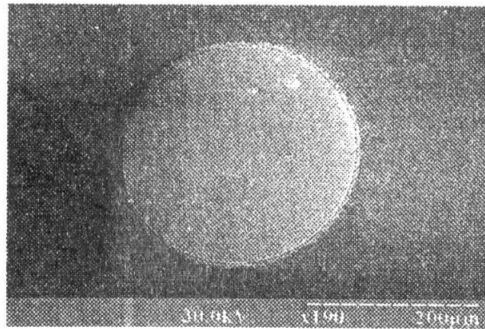


Fig.4. SEM of AL-MMC sample reinforced with stainless steel fibers at T=640°C, t=0.25hr, and P=65MPa.

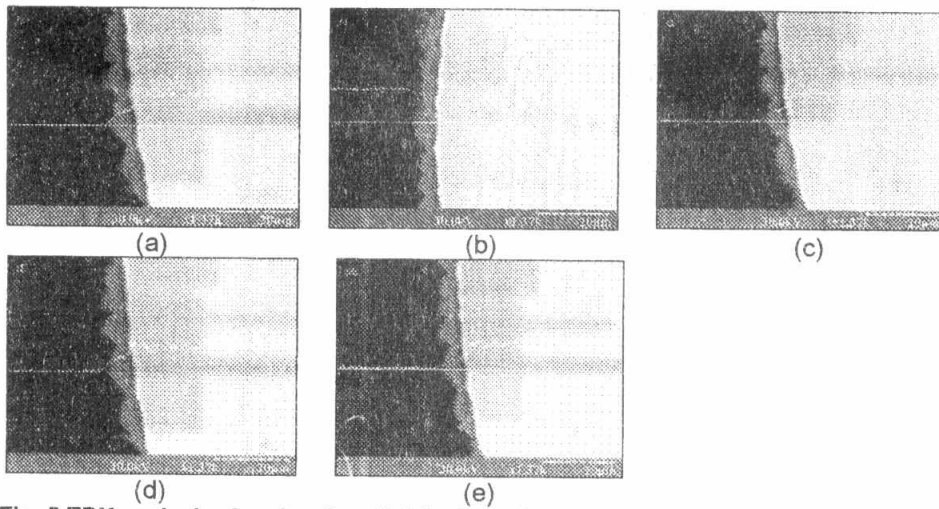


Fig. 5 EDX analysis showing line distributions through the interphase layer which were prepared at T=640 °C, t=0.25hr, and P=65 MPa (a) Fe, (b) Al, (c) Cr, (d) Ni, and (e) Mn.

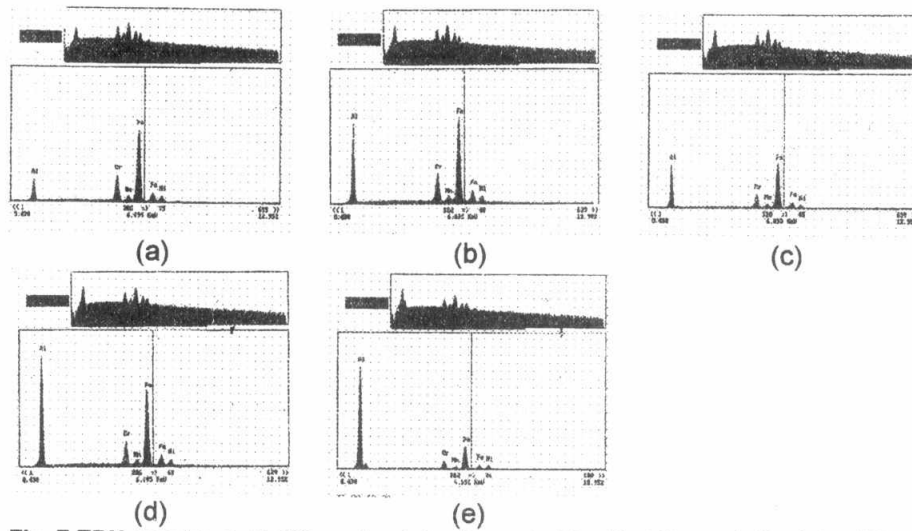


Fig. 7 EDX spectrum of different points shown in Fig. (6 a) through the interdiffusion zone (a) 1, (b) 2, (c) 3, (d) 4, and (e) 5 respectively.

Table 6. The predicted iron-aluminate intermetallic compounds formed through the interdiffusion zone.

Layer	Thickness (μm)	Al %	Predicted Intermetallic compounds
A	4	3-31	Fe ₃ Al
B	4.5	31-42.23	FeAl
C	4.5	42.23-53.462	FeAl ₂
D	13.5	53.46-57.48	Fe ₂ Al ₅
E	4.5	57.48-60.73	FeAl ₃
F	4.5	60.73-68	Fe ₂ Al ₇

Table 7. Fiber/matrix interface characteristics and the mechanical properties at 13MPa

Hot pressing parameters			Interphase characteristics		Mechanical properties		
P (MPa)	T (°C)	T (hr)	Mean thick. (μm)	Average cont. (%)	σ _{ut} (MPa)	σ _{0.2} (MPa)	δ (%)
13	600	0.5	5	Localized	151.7	108.3	23
		1	8	50	146.3	103.5	20
		2	15	60	143.5	99.5	18.9
	620	0.5	7.5	40	150	108	21
		1	15	75	144	100	18.5
		2	23	95	141.7	98	17.4
	640	0.5	13.5	65	146.5	102	19
		1	25	93	141.4	97	16.6
		2	33	95	140.5	95.33	16.14

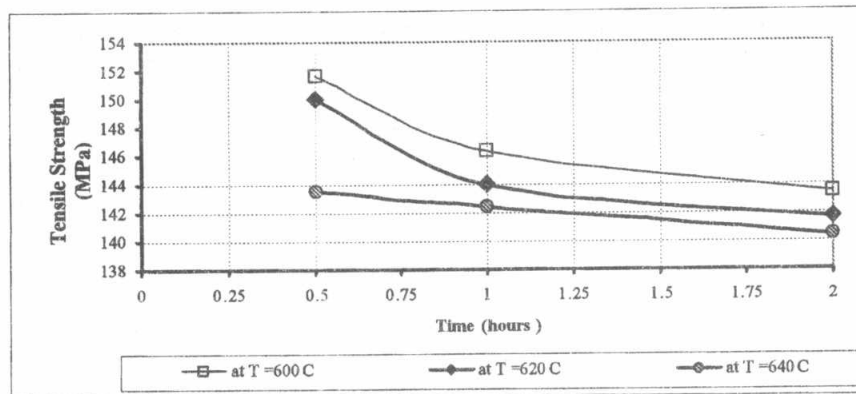


Fig.8. Strength of Al-MMC samples prepared by hot pressing at a constant pressure of 13MPa and different temperatures and holding times.

Table 8. Fiber/matrix interface characteristics and the mechanical properties at 39MPa

Hot pressing parameters			Interphase characteristics		Mechanical properties		
P (MPa)	T (°C)	T (hr)	Mean thick. (μm)	Average cont. (%)	σ_{ut} (MPa)	$\sigma_{0.2}$ (MPa)	δ (%)
39	600	0.5	6	20.3	153.5	111	21.5
		1	9	66	149.3	107.9	19
		2	15.5	87.5	145	102	18
	620	0.5	7	66	151.5	108	20
		1	15.5	94.5	144.5	102.31	17.5
		2	25	98	143	98	16
	640	0.5	18	95	144.5	102.3	18
		1	28	Continuous	143	100.6	15.3
		2	35	Continuous	142	96.46	14

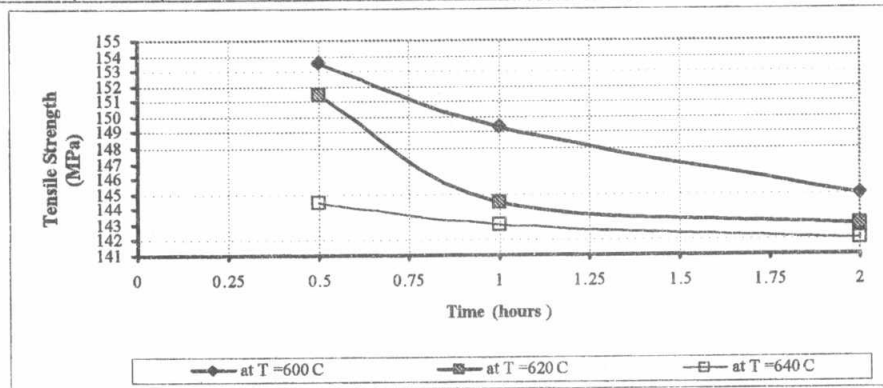


Fig. 9. Strength of Al-MMC samples prepared by hot pressing at constant pressure of 39MPa and different temperatures and holding times.

Table 9. Fiber/matrix interface characteristics and the mechanical properties at 65MPa

Hot pressing parameters			Interphase characteristics		Mechanical properties		
P (MPa)	T (°C)	T (hr)	Mean thick. (μm)	Average cont. (%)	σ_{ut} (MPa)	$\sigma_{0.2}$ (MPa)	δ (%)
65	600	0.25	Not clear interphase layer		150.2	110.9	23.5
		0.5	7.8	35.6	149.5	107.4	22
		1	10	84.7	147.5	105.6	19.5
		2	14	Continuous	146	103.3	17.5
	620	0.25	4	22.2	154	111.5	22.3
		0.5	13	97.2	148	105.2	19
		1	17.5	Continuous	146.4	100	16.5
		2	27	Continuous	144.5	98.5	15.5
	640	0.25	6	Continuous	160	113.8	19.44
		0.5	20	Continuous	143.7	98.8	17
		1	31	Continuous	142.08	95.03	15
		2	38.5	Continuous	139	94	13.3

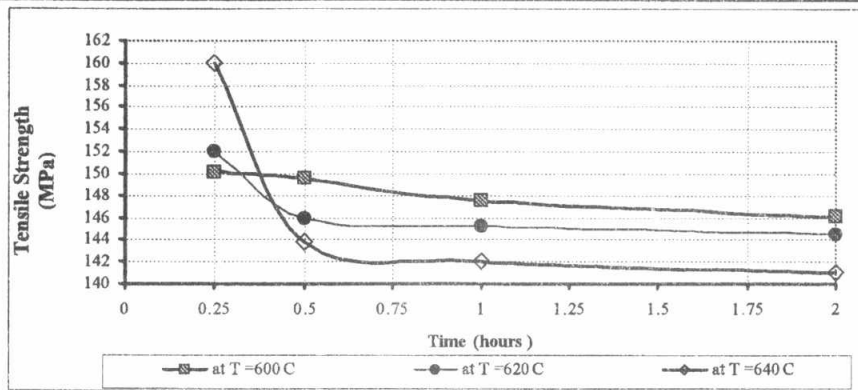


Fig. 10 Strength of Al-MMC samples prepared by hot pressing at different temperatures and holding times and constant pressure (P=65MPa).

Table 10 Mechanical properties of test samples prepared at T=640°C, t=0.25hr, P=65MPa, and at different fiber volume fractions.

Test samples produced at different V_f (%)	Mechanical Properties			
	E (GPa)	σ_{ut} (MPa)	$\sigma_{0.2}$ (MPa)	δ (%)
Monolithic Matrix ($V_f=0\%$)	70	60	34	22.4
$V_f=7.6\%$	78	160	113.78	19.44
$V_f=13.2\%$	85	200	145	12.3
$V_f=14\%$	87	210	147	10.5
$V_f=16.4\%$	90	230	180	7.6

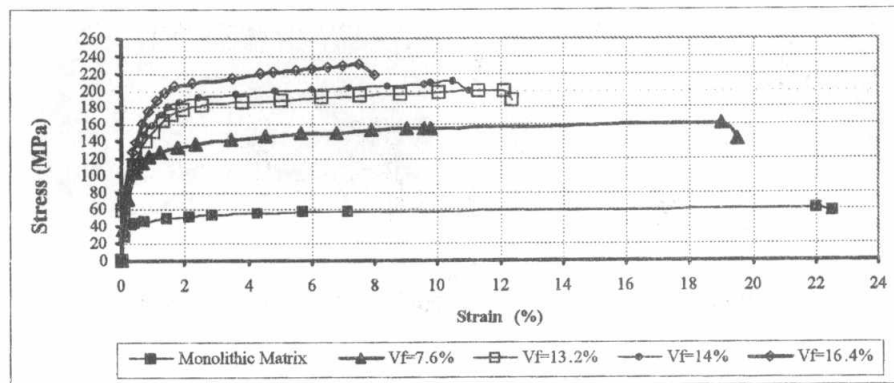


Fig. 11 Stress-strain diagrams of Al-MMC samples that prepared at T=640°C, t=0.25h, and P=65MPa and at different fiber volume fraction.

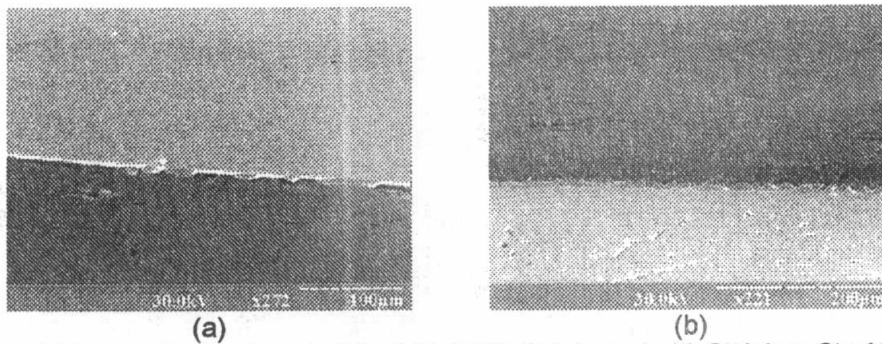


Fig. 12 Damage Mechanisms in [0]₅ of AL-MMCs Reinforced with Stainless Steel Fibers Monotonically loaded to 0.2σ_{UT} at Room Temperature:
 (a) Debonding in the outer plies.
 (b) There is no clear damage evidence in the inner plies.

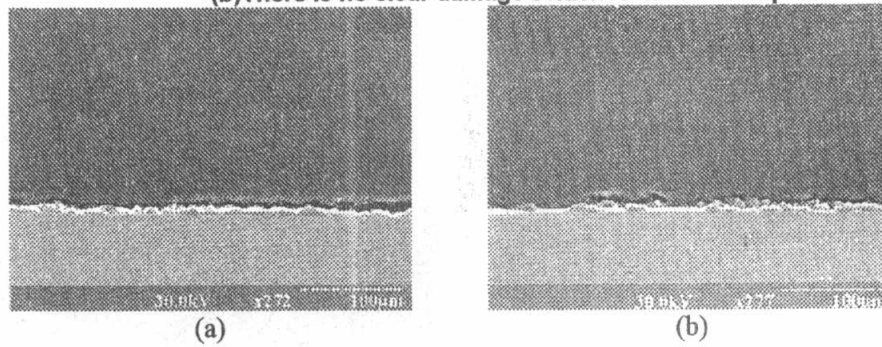


Fig.13. Damage mechanisms in [0]₅ AL-MMCs Reinforced with Stainless Steel Fibers Monotonically loaded to 0.4σ_{UT} at Room Temperature:
 (a) Further debonding in the outer ply.
 (b) Low Debonding in the inner ply.

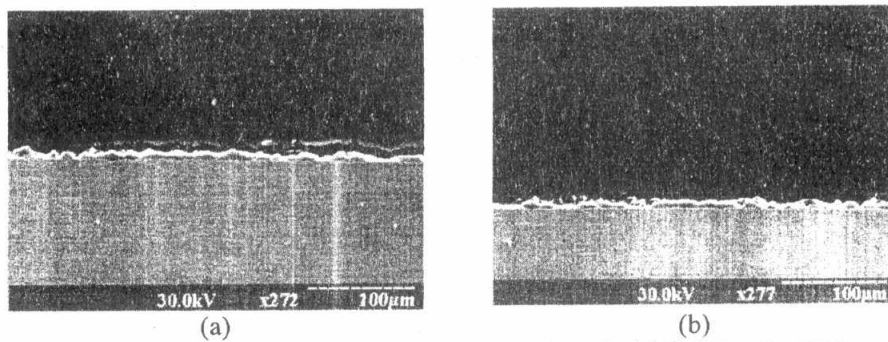


Fig.14. Damage Mechanisms in [0]₅ AL-MMCs Reinforced with Stainless Steel Fibers Monotonically loaded to 0.5 σ_{UT} at Room Temperature:

- (a) Further debonding and matrix crack initiation in the outer ply.
- (b) Further debonding in the inner ply.

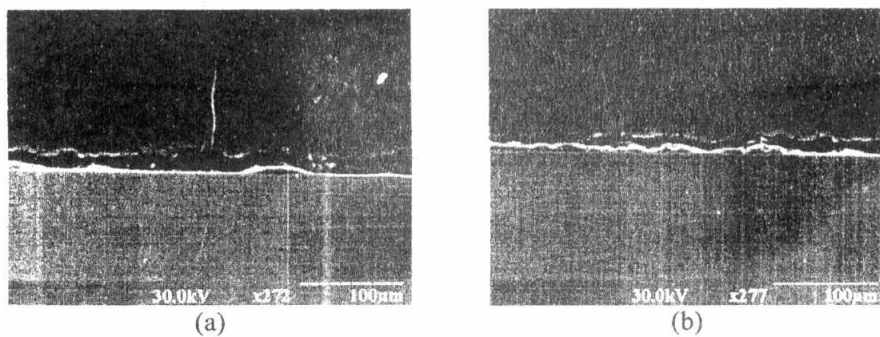


Fig.15. Damage mechanisms in [0]₅ AL-MMCs reinforced with stainless steel fibers monotonically loaded to 0.7 σ_{UT} at room temperature:

- (a) Matrix crack from interfaces with debonding in the outer plies.
- (b) Nucleation of matrix crack from interfaces with debonding in the inner ply.

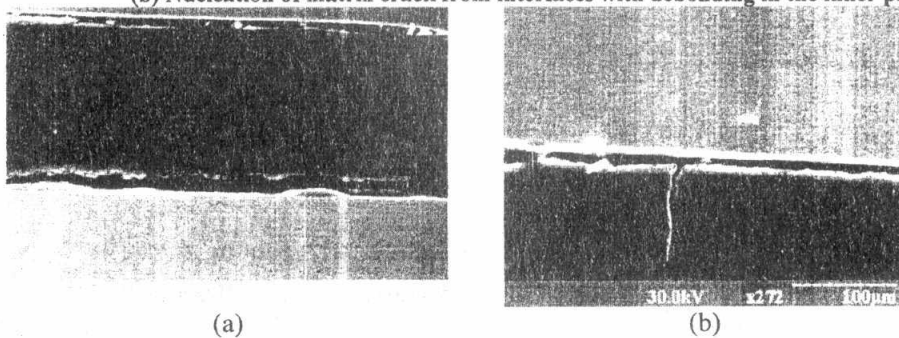
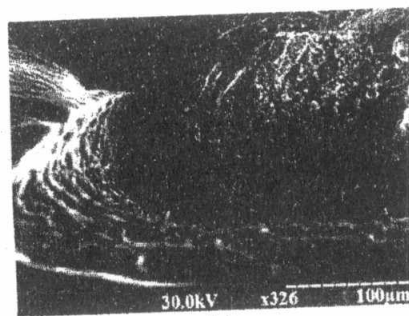
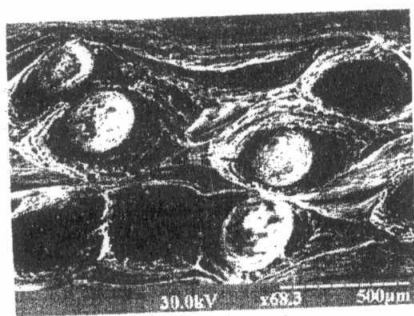


Fig. 16 Damage Mechanisms in [0]₅ AL-MMCs Reinforced with Stainless Steel Fibers Monotonically Loaded to 0.9 σ_{UT} at Room Temperature:

- (a) Matrix crack coalescence in the outer plies.
- (b) Evolution of matrix cracks in the inner ply.



(a)
Fig. 17 Typical fracture surface morphologies of [0]_s AL-MMC sample at Room Temperature showing evidence of:
(a) Fiber pull-out from the matrix with partially delamination between the matrix layers.
(b) Ductile dimpled matrix failure.