Military Technical College
Kobry El-Kobbah
Cairo, Egypt

10th International Conference
On Aerospace Sciences&
Aviation Technology

BONDING AND PROCESSING TECHNIQUE OF LAMINATED COMPOSITE MATERIALS

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ABSTRACT

An optimum composite technology by diffusion bonding was developed to obtain aluminum-laminated composites. Pure aluminum and duralumin laminates that have distinct properties were bonded together without the use of an intermediate binding layers or using pure Cu or Brass as intermediate layers by applying the hot pressing technique. The effect of hot pressing parameters (P,T & t) were studied from the microstructural point of view. Also, the micro chemical analysis was carried out to follow the evolution of diffusion of the different elements in the interdiffusion zone under different parameters to predict the phases that may be formed through it. The mechanical properties of the produced laminated composites, before and after age hardening, were studied.

The results obtained from the microstructural analysis revealed that, binding of both laminates without using a binding material can be achieved at an optimum conditions of P=54MPa, T=500°C & t=5hrs. Excessive diffusion of copper atoms from the duralumin laminate towards the pure aluminum was recorded and attained an average depth of 135μm. This bleeding of copper atoms leads to a substantial decrease of the mechanical properties of the duralumin side. On the contrary the application of an age hardening cycle leads to an increase of hardness from 90 to 240 VHN in the duralumin laminate.

To retain the level of the copper atoms in the duralumin and in the same time to secure enough diffusion that allows sufficient bonding between both laminates a copper layer of 10μm thickness was used as a binding material.

Using of brass as a binding material provided a better binding effect where zinc enhances copper atoms to penetrate in the brass intermediate layer towards the pure laminate.

KEY WORD

Aluminum, Duralumin, Laminate, Diffusion bonding, Hot pressing, Brass, Copper

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INTRODUCTION

Among many forming processes, diffusion bonding is one of the advanced techniques for binding materials that have distinct properties that are used in the manufacturing of various parts and components for aerospace applications.
Even with an intermediate binding material or without, it is possible to obtain sufficient joint strengths under a proper compaction pressure, binding temperature and after holding for a sufficient time to allow a good area of contact.
Diffusion bonding of titanium has been used to produce aerospace components using copper of 100μm thickness as an intermediate layer as described by Norris [1] at a temperature of 938^0C for 1hrs holding time in a vacuum atmosphere In addition, C.F.Yang [2] described the diffusion of different constituents in the Al-Zn-Mg alloy using Zn as interlayer by electroplating at a temperature of 515^0C for 4hrs holding time and under pressure of 1MPa and under a vacuum of 10^{-4} to achieve a minimum shear strength of 35MPa.
Diffusion bonding was also attempted for Al_2O_3 and Si_3N_4 ceramics by Shimada-M [3] using high-pressure technology reached up to 3Gpa and 500^0C. Moreover, using of ductile metal interlayers as Al,Ag,Cu and Ni to binding Al_2O_3 ceramic is carried out by Nicholas-M-G[4]
W.D.Macdonald and T.W.Eagar [5] described diffusion bonding of titanium alloy using zinc interlayer at a temperature of 800^0C for 4hrs holding time under vacuum, where the produced joint attained one third of the parent metal strength. Zhao[6] demonstrated the diffusion bonding of SiC/2024Al composites by means of pure aluminum foil interlayer under a temperature of 570^0C for 1hrs holding time and pressure of 16MPa. Diffusion bonding of an Al-Cu alloy using metallic interlayers such as an Al-Li alloy and pure silver, has investigated by Escalera[7].
In this work, the hot pressing technique was applied to produce aluminum laminated composite materials by diffusion bonding of pure aluminum and duralumin laminates with or without using intermediate binding materials. In addition, the mechanical properties of the produced aluminum laminated composite samples before and after age hardening were studied. This was accomplished using various testing, such as the tensile test, Vickers micro hardness test and the microstructure using scanning electron microscope.

EXPERIMENTAL WORK AND MATERIALS USED

This issue is devoted to introduce and explain the experimental procedure for bonding of commercial pure aluminum and duralumin laminates having different properties with or without using an intermediate binding material to produce aluminum laminated composite by applying the hot pressing technique
Various binding materials were used between the two laminates after the surface of both laminates were carefully ground using different emery papers up to the grade 600. Pure copper or brass (35%Zn) of 30μm thickness were used by vacuum deposition on the surface of the duralumin laminate.
The desired arrangement of both laminates during processing is shown in Fig.1a. The sample is then put in the central position of a special die as shown in Fig.1b.
A hydraulic press of max load capacity of 50KN was used to apply a pressure of 54MPa on the sample and then put in a muffle furnace to secure the desired temperatures ($T=450^0C$, 500^0C). The adopted holding time was t=1,3,5hrs.

In order to attain the utmost properties of the duralumin laminate an age hardening treatment consists of solution treatment at 520^{0}C , quenching to room temperature following by an aging process at 200^{0}C.
Microstructure of the produced composite samples at different hot pressing parameters were carried out using Scanning electron microscope (SEM) type Remma-202 equipped by WDX & EDX facilities.
In addition, the specimen was machined using a wire cutting machine to attain the prescribed dimension of the used test specimens as shown in Fig.1c.
The shear strength was measured by applying a tensile load on specimens having an overlapped area of 30*30mm^2 using an electro hydraulic testing machine. Moreover, the Vickers microhardness at the interface of the produced laminated composite was measured on an optical microscope equipped by microhardness measuring facilities.

RESULTS AND DISCUSSION

Different groups of Al-laminated composite test samples were prepared at different hot pressing parameters to study the effect of these parameters on the structure and characteristics of the bonding interface. The results of hot pressing in the studied temperature range (450°C to 500°C), for different holding times and at an initial constant pressure of 54 MPa are presented in figures from (2a-h).
Figs. (2a-d) illustrate the diffusion-bonding interface between pure aluminum and duralumin sheets obtained after different holding times and under a bonding temperature of 450°C. We can state that, in the specimen subjected to short holding times a clearly visible separation all the long of the interface has occurred, with the presence of relatively large voids of an average size from 3 up to 5µm, while those obtained after longer times up to 5hrs a net improvement of interface nature was obtained and the absence of any clear separation or voids.
Figs. (2e-h) illustrate that separation is much less visible for the produced composite sample obtained after 1 hr holding time and under a binding temperature of 500°C relative to those obtained under the same holding time at 450°C. Prolonged holding time to 5hrs demonstrates, an interface nearly uniform and nearly free of any delamination and void existence.
On the other hand, WDX analysis was applied on the prepared sample at a temperature of 450°C. Specimen subjected to short holding time at this temperature proved that copper diffusion towards pure aluminum can be considered negligible while it increases from 65µm to 85µm for prolonged holding times. Moreover, the diffused copper atoms were able to form second phase particles in the pure aluminum laminate as shown in Figs.(3a-c). Table (1) illustrates the thickness and the average Al-atomic% of each indicated area along the interdiffusion zone which reveals the nature of the expected present phases along the interface.
Figs.(3d-f) illustrate that the WDX analysis of a diffusion bonded sample, at a temperature of 500°C for 1hr holding time, has a limited diffusion and penetration of copper across the interface. While increasing holding time from 3hrs up to 5hrs indicates that copper diffusion increased from 75µm to 135µm into the pure aluminum. Table (2) demonstrates the thickness and the average Al-atomic% of each indicated area across the interface in the pure aluminum laminate
The effect of holding time at the temperatures 450°C and 500°C on the interdiffusion layer thickness is shown in Fig. (4a). This reveals that, at 450°C by increasing the holding time up to 5hrs the interdiffusion zone is increased to 85 µm while at a higher

temperature of 500°C the interdiffusion thickness increased to 135 μm approximately for the same interval of holding time.
Fig. (4b). demonstrates the shear strength of the produced aluminum-laminated composites versus the holding time. It shows the deep influence of diffusion on the shear value of the prepared sample at a temperature of 500°C for 5hrs holding time that attain the base metal strength (pure aluminum).
Table (3) Summarizes the Al-laminated composite interface characteristic and the average shear values, beside to the location at which fracture occurs.
The evolution of the indentations size after aging is clearly demonstrated fig.(5). Moreover, the obtained Vickers micro hardness values across the interface before and after heat treatment were also plotted against the distance from the interface and presented in Fig. (6). This can easily show the influence of the age hardening treatment on the measured mechanical properties.
This confirms that the micro hardness values of commercially pure aluminum don't show any significant difference of the hardness values before and after age hardening beyond an average depth of 130μm. while, the micro hardness values in pure aluminum from this limit to the original interface show a sensible increase of hardness of about 180 VHN and this can be attributed to the diffusion of copper across the interface. On the other hand, duralumin demonstrates a highly significant increase of hardness from 90 to 240 VHN after heat treatment.
Figs (7a-c) illustrates the diffusion bonded interface obtained after different holding times and under a bonding temperature of 450°C between both laminates using copper as a binding material. It reveals a pronounced continuous separation after short holding time all the long of the interface of an average length 40-50μm approximately. Increasing holding time demonstrates a nearly uniform interface from any voids or delamination.
Figs (7d-f) illustrates a clear separation and debonding along the interface at short holding times and at a temperature of 500°C. This separation is sensibly less than the interface prepared at the same holding time and under a temperature of 450°C. prolonged holding time to 5hrs demonstrates an interface nearly without any delamination and void existence , in addition, we cannot observe any more the intermediate copper layer which proves that it is diffused completely.
The results of the WDX analysis are presented in figs.(8a-c) which demonstrates a negligible diffusion of copper towards pure laminate for a sample prepared at binding temperature of 450°C and after short holding times. Moreover, the prolonging holding time to 5hrs the penetration depth can attain 125μm approximately in the pure laminate and is able to form second phase precipitates. Table 4 illustrates the thickness and the average Al-atomic% of each indicated areas across the interdiffusion zone.
Figs.(8d-f) presents the results of the WDX analysis for the prepared samples at a temperature of 500°C for different holding times. These results illustrate a small penetration of copper atoms to an average depth of 25 μm approximately under short holding times which is increased to 100 μm and 170 μm after 3 and 5hrs holding times respectively. Besides, copper atoms were able to form second phase particles on the pure laminate. In addition, table 5 demonstrates the thickness and the average Al-atomic% of each indicated areas across the interface in the pure aluminum laminate with the probable present phases.
The effect of holding time at temperature 450°C and 500°C on the interdiffusion layer thickness is shown in Fig.(9a) which reveals that, at temperature 450°C by increasing

the holding time up to 5hrs the interdiffusion zone is increased to 125 µm. At a higher temperature of 500°C the interdiffusion thickness increased to 170 µm approximately for the same interval of holding time. So its clear that, due to the presence of copper as a binding material and for prolonged holding time copper was enhanced to diffuse and penetrate to a higher depth than that observed in the case of bonding of laminates without using any binding materials.

This allows, from one hand, that duralumin laminate reserves its composition and consequently mechanical properties and, from the other hand, diffusion bonding between laminates is enhanced. This supports our target of binding of dissimilar materials having distinct properties.

Fig (9b) shows the effect of time at both temperatures on the average shear strength of aluminum-laminated composites. The results at 1 and 3 hrs holding times indicated limited effect on the diffusion bonding process and also the average shear strength. This can be related to the thickness of the interdiffusion zone at this holding time which is not suitable to achieve a complete bonding. Prolonged holding times up to 5hrs at the same temperatures provides a satisfactory bonding. This can be attributed to the enhancement of copper diffusion for the prolonged holding times and consequently the deep penetration of copper.

Table (6) Summarizes the Al-laminated composite interface characteristics and the average shear values.

Fig. (10a-b) demonstrates the indentation size evolution between pure aluminum and duralumin laminate after aging at binding temperatures 450°C and 500°C. A significant increase in the micro-hardness values were obtained. A hardness of 175VHN. in the pure aluminum laminate was reached at an average depth of 120-130µm approximately at 450°C as shown in Fig.(11), and this can be attributed to the diffusion of copper from the interface to the pure aluminum laminate. On the other hand, increasing the bonding temperature to 500°C results in a pronounced increase of micro hardness values (210VHN) for an average penetration depth of 160-170µm in the pure laminate.

Figs.(12a-b) demonstrates the diffusion bonded interface between pure aluminum and duralumin using brass as an intermediate binding material. This reveals significant continuous separations all the long of the interface of an average thickness 25-30µm approximately at short holding times. Moreover, the delamination effect is reduced and produced a nearly uniform interdiffusion zone with the absence of any separation along the interface but voids were occurred of an average size 5-8µm which is much less than those observed in case of using copper as binding material only. This is, in addition, to the presence of some minor cracks at a prolonged holding times.

Figs.(12c-d) illustrates a pronounced clear evolution of the interface and the existence of some minor voids of an average size 1-2µm approximately in addition to the presence of the binding layer of an average thickness 0.25-0.5µm approximately at a short holding time. Besides, the interface obtained after 3hrs holding time is nearly without any delamination and void existence and the brass intermediate layer is diffused completely into the basic laminates.

Figs.(13a-b) presents, the WDX microanalysis for a sample prepared at a binding temperature of 450°C and after 1hrs holding time. This analysis showed a negligible diffusion of copper while zinc diffusion toward pure aluminum laminate is very fast and complete.

Figs.(14a-b) indicate that at prolonged holding times copper is much more enhanced and penetrates up to 140μm approximately toward the pure laminate and were able to form second phase precipitates.

The WDX microanalysis for a prepared sample at a temperature of 500°C after 1hr holding time is shown in Fig.(14C). We can note that small penetration of copper atoms into the pure laminate reached an average depth of 40μm. Which is higher than that observed at 450°C for the same holding time. In addition, Fig(14d) indicates that, after 3hrs holding time copper has a penetration depth of 180μm into the pure aluminum laminate and was able to form second phase particles on the pure aluminum side that reached to approximately 140μm depth.

In addition, tables (7-8) illustrates the depth of penetration and the average Al-atomic%, Cu-atomic% and Zn-atomic% of each indicated areas across the interdiffusion zone at a temperature of 450°C for 5hrs holding time and a temperature of 500°C for 3hrs holding time respectively. This reveals that zinc diffuses faster to the outer surface than copper. The expected present phases across the interface towards the pure aluminum laminate to an average depth 200μm approximately are $Al+CuAl_2$.

The effect of holding time at temperatures 450°C and 500°C on the interdiffusion layer thickness is shown in Fig.(15a) which reveals that, at a temperature of 450°C by increasing the holding time up to 5hrs the interdiffusion zone is also increased to 140 μm. At a higher temperature of 500°C the interdiffusion thickness increased to 220 μm approximately for the same interval of holding time.

So it is clear that, using brass as a binding material will enhance copper diffusion rather than using copper alone as a binding material due to the presence of zinc in brass which have a deep influence of copper diffusion and allows it to penetrate to a higher depth than that observed in the case of binding of laminates using pure copper as a binding material. On the other hand, sufficient high binding effect between the two laminates can be achieved. This supports our target of bonding of dissimilar materials having distinct properties.

Fig. (15b) illustrates the effect of holding time on the average shear strength of aluminum-laminated composite. It is clear that, at 1 and 3 hrs holding times at a binding temperature of 450°C a limited effect on the diffusion bonding process and consequently the average shear strength were observed. On the contrary at a prolonged holding times up to 5hrs a satisfactory bonding was obtained. On the other hand, at a binding temperature of 500°C and after 1hr holding time the diffusion is still limited and consequently, the shear strength demonstrates low values. Moreover, prolonged holding time of 3hrs revealed a pronounced influence in increasing the average shear strength values.

Table (9) Summarizes the Al-laminated composites interdiffusion zone characteristics and the average shear values, beside to the location of fracture.

Vickers micro hardness measurements obtained on the specimens treated for 5hrs at 450°C and 3hrs at 500°C are illustrated in Figs. (16a-b).

Fig.(17) indicates that the values of Vickers hardness in the duralumin when brass is used as a binding material attain 290VHN which are higher than those obtained when using pure copper instead. This can be explained by the effect of zinc in promoting hardness by the solid solution hardening mechanism. Moreover, the values of hardness obtained after binding for 3hrs at 500°C are higher than those measured on specimens treated for 5hrs at 450°C for the same penetration depth over 120μm. In addition for the same conditions when using pure copper as a binding element we notice that the penetration depth of copper in the pure aluminum laminate is lower than when using brass as a binding element. This can be attributed that the solubility of copper in aluminum is increased by zinc additions. At 450°C the solubility of copper is only 2.8% when there is no zinc while it increase up to 3.6%Cu at a level of 9%Zn as explained by Sietz [8].

CONCLUSIONS

1- Hot pressing technique can be used to produce aluminum laminated composite materials by diffusion bonding. The hot pressing parameters control to a great extent the final mechanical and structural properties of these composites.

2- Adequate bonding between a laminate of pure aluminum and another of duralumin without using any intermediate binding material can be obtained by diffusion bonding at 500°C for 5hrs holding time. Prolonged treatment at this temperature beyond 5hrs can cause severe bleeding of copper from duralumin toward pure laminate and consequently degradation of its mechanical properties.

3- The mechanical and structure characteristic of aluminum laminated composite produced by the hot pressing technique were improved by using a layer of pure copper as a binding material between the two laminates which is a direct result of the enhancement of binding by the diffusion of copper and the saving of the copper required level in the duralumin laminate.

4- Optimum binding strength was obtained by using brass as a binding material. Moreover the values of hardness in the duralumin was increased by about 25% relative to those obtained in the case when a pure copper layer was only used

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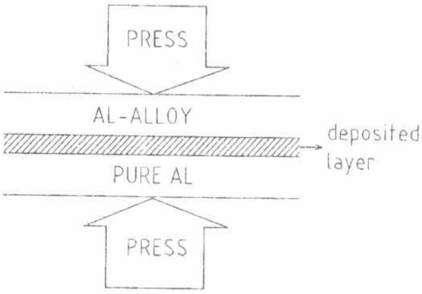

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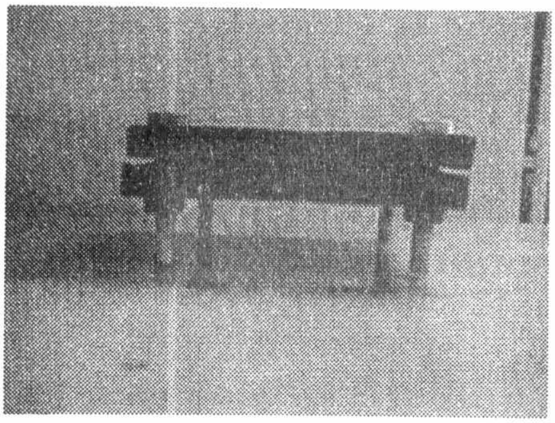

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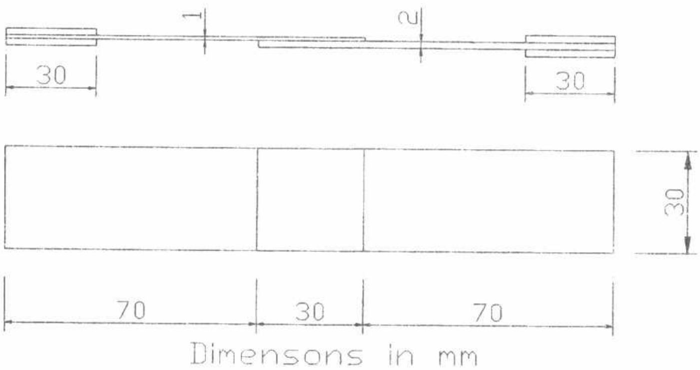


(c)

Fig.1 : a- Schematic drawing of the hot pressing technique
B-The used custom die for hot pressing.
c- Standard flat tensile test specimen

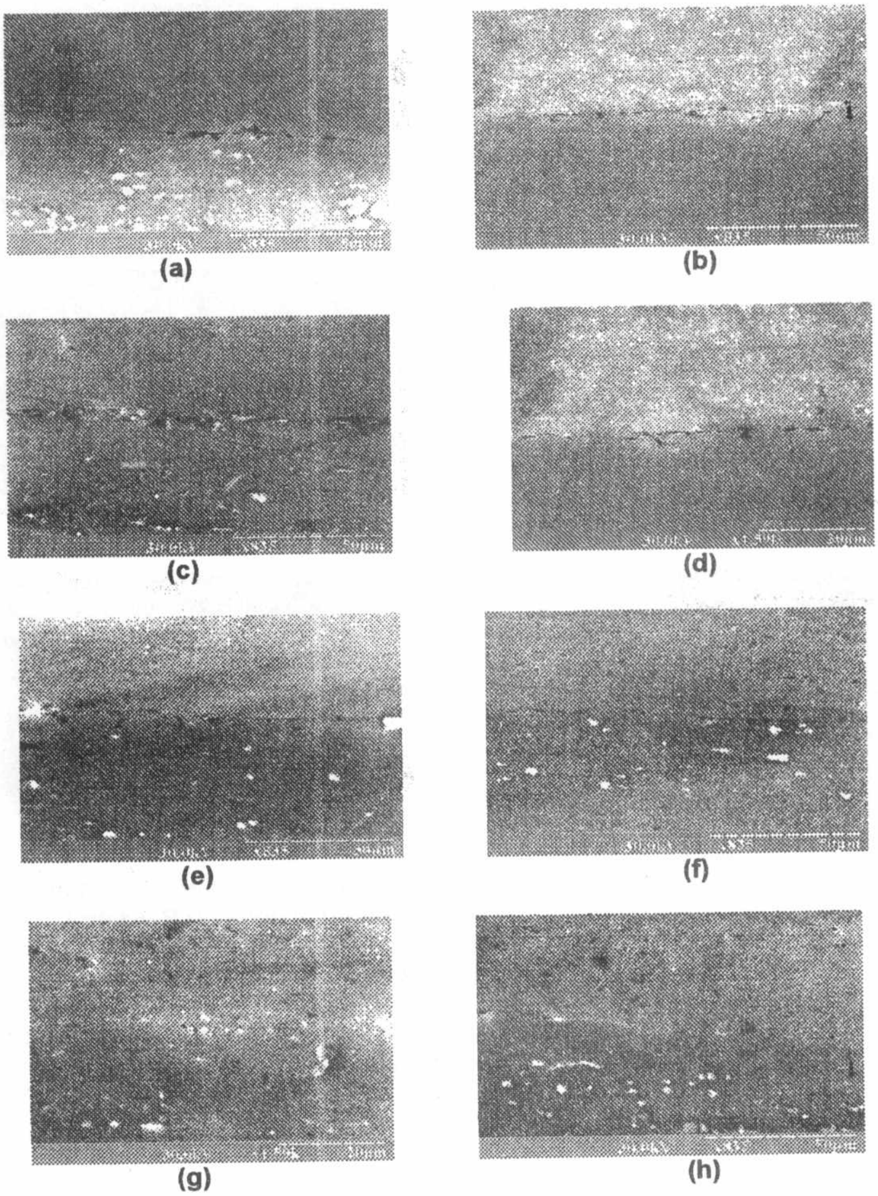
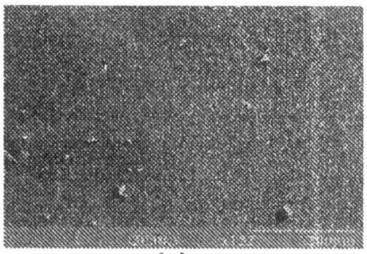


Fig.2 Scanning Electron Micrograph of Al-laminated composite without using any filling material produced at P=54MPa:
(a-d)-T=450°C, and t=1,3 and 5hr respectively
(e-h)-T=500°C, and t=1,3 and 5hr respectively



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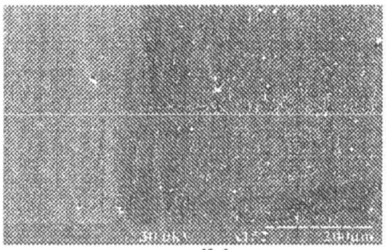

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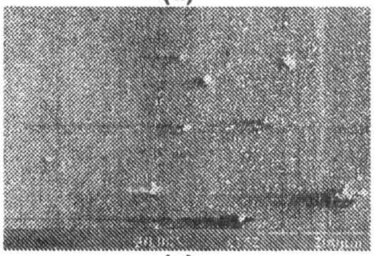

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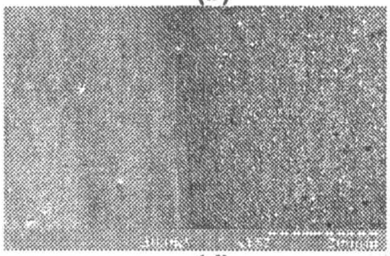

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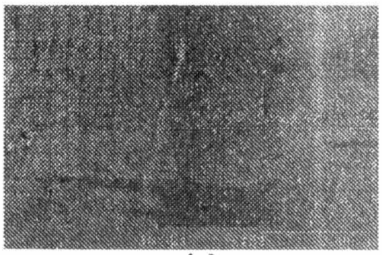

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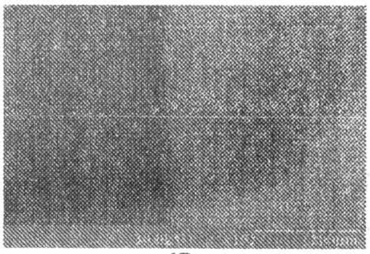


(f)

Fig.٣ WDX analysis showing line distribution of copper through the interface for samples that prepared without filling material at P=54MPa (a-c)-T=450°C, and t=1,3 and 5hr respectively (d-f)-T=500°C, and t=1,3 and 5hr respectively

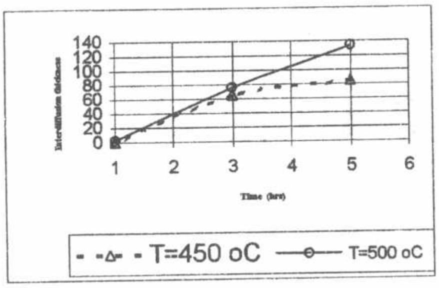
Table (1-2) Expected Al-Cu phases formed through the interdiffusion zone at T=450°C, t=5hr and T=500°C, t=5hr respectively

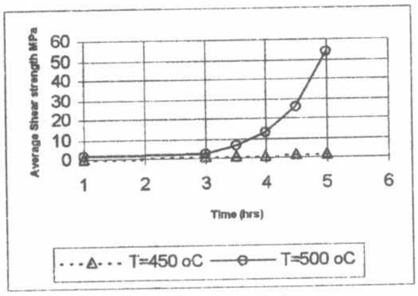
Thickness (µm)	Average Al%	Expected Phase
10	15-20%	α_{Cu}
25	30-35%	γ_2
	45-50%	η_2
75	65-70%	θ
90	100%	α_{Al}

(1)

Thickness (µm)	Al%	Expected Phase
25	10-15%	α_{Cu}
65	31-37%	γ_2
100	40-45%	ξ_2
135	64-68%	θ
155	100%	α_{Al}

(2)





(a) (b)

Fig.4. Samples prepared without bonding material at P=54MPa showing a-Interface layer thickness at different holding times b-Average shear strength of Al-MMC samples at different temperature.

Table (3) Al-laminated composite without bonding material showing the interface characteristics and the mechanical properties at P=54MPa

Bonding Parameter		Interdiffusion Thickness (μm)	Average Shear Strength (MPa)	Fracture Location
T(°C)	T(hrs)			
450	1	---	---	--
450	3	65	1.1	BI
450	5	85	1.3	BI
500	1	---	---	---
500	3	75	2.1	BI
500	5	135	54	BM

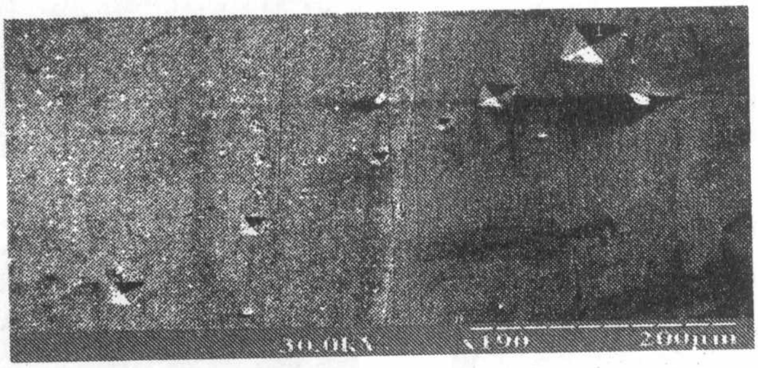


Fig. 5 SEM of Al-laminated composite without filling material showing Vicker Micro hardness after age hardening for a prepared sample at P=54MPa, T=500°C and t=5hrs

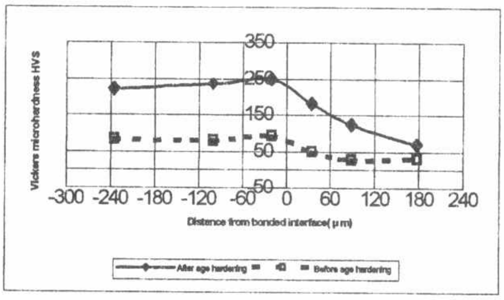
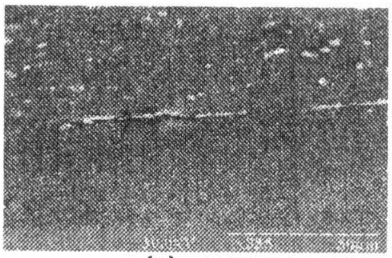


Fig.6. Vickers Micro hardness properties from the interface for a prepared sample without bonding material before and after aging at P= 54MPa, T=500°C and t=5hrs



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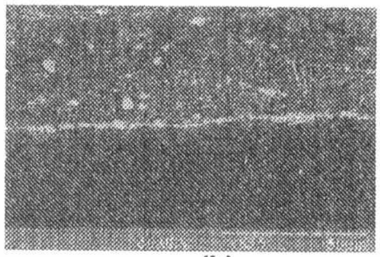

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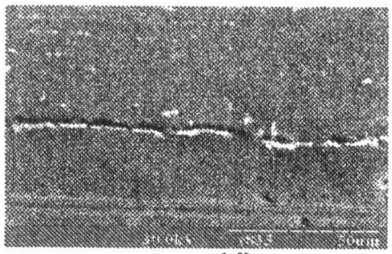

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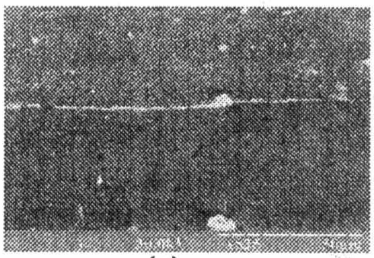

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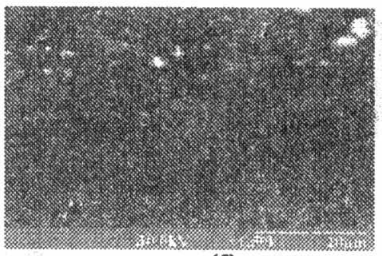
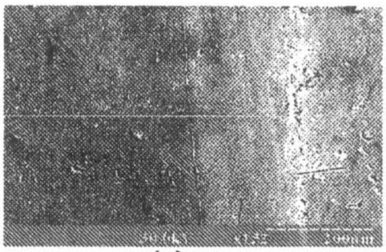


(f)

Fig.7. Scanning Electron Micrograph of Al-laminated composite using copper as a binding material produced at P=54MPa:
(a-c)-T=450°C, and t=1,3 and 5hr (d-f)-T=500°C, and t=1,3 and 5hr



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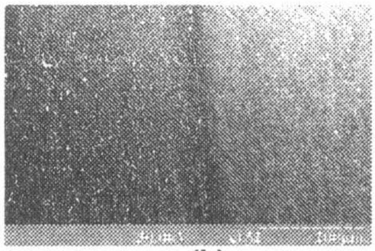

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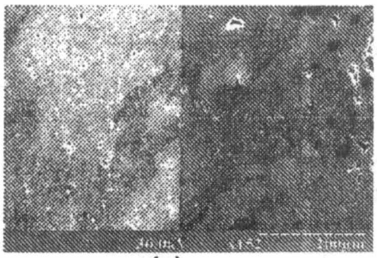
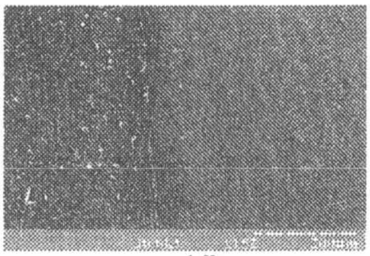
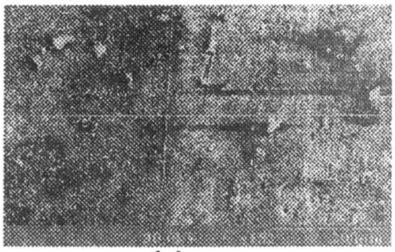


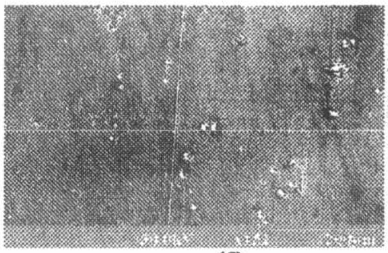
(c)



(d)



(e)



(f)

Fig.8. WDX analysis showing line distribution of Cu through the interface for samples that prepared using copper as filling material at P=54 MPa (a-c)-T=450°C, and t=1,3 and 5hr (d-f)-T=500°C, and t=1,3 and 5hr

Table (4-5) Expected Al-Cu phases formed through the interdiffusion zone at T=450°C, t=5hr and T=500°C, t=5hr respectively

Thickness (μm)	Al%	Expected Phase
25	15-20%	α_{Cu}
50	30-35%	γ_2
70	45-49%	η_2
100	65-69%	θ
130	100%	α_{Al}

(4)

Thickness (μm)	Al%	Expected Phase
40	10-15%	α_{Cu}
80	31-37%	γ_2
110	40-45%	ξ_2
150	64-68%	θ
180	100%	α_{Al}

(5)

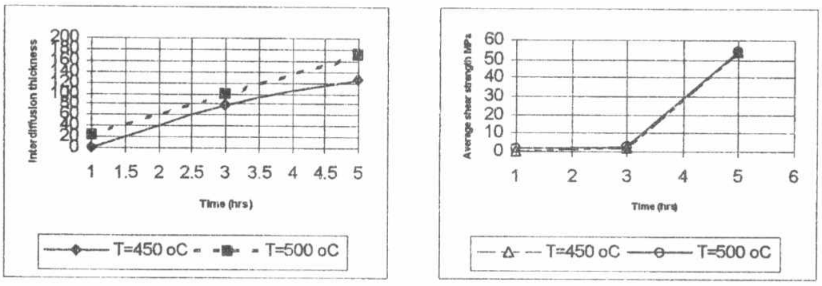
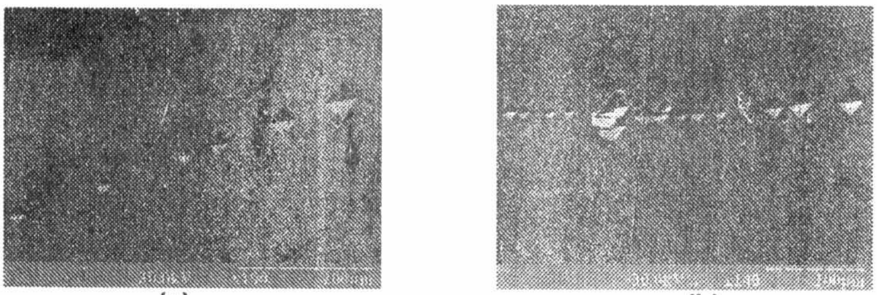


Fig.9. Samples prepared using Cu as binding material at P=54MPa showing a-Interface layer thickness at different holding times b-Average shear strength of Al-MMC samples at different temperature.

Table 6. Al-laminated composite using copper as bonding material showing interface characteristics and the mechanical properties at P=54MPa

Bonding Parameter		Interdiffusion Thickness (μm)	Average Shear Strength (MPa)	Fracture Location
T(°C)	T(hrs)			
450	1	---	---	--
450	3	80	1.4	BI
450	5	125	54	BM
500	1	25	1.9	BI
500	3	100	2.4	BI
500	5	170	54	BM



(a) (b)

Fig.10. SEM of Al-laminated composite using copper as binding material showing Vickers Micro hardness after age hardening, for a prepared sample under P=54Mpa , t=5hrs. and at a-T=450°C b-T=500 °C

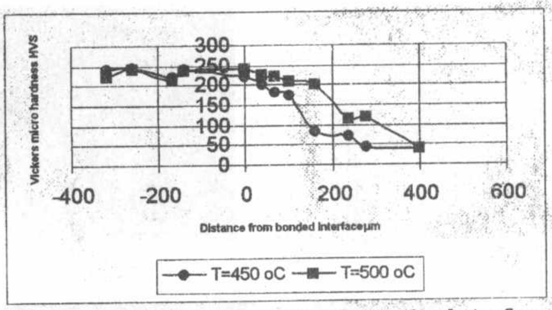


Fig.11. Vickers Micro hardness properties from the interface for a prepared sample after aging at P=54 MPa,T=450°C and 500°C at t=5hrs

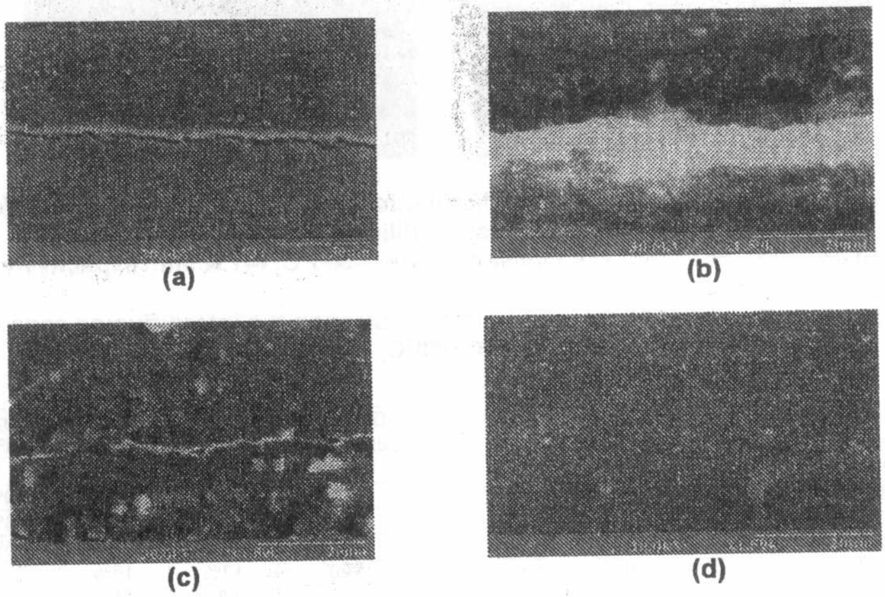


Fig.12. SEM of Al-laminated composite using brass as a binding material at (a-b)-T=450°C, and t=3hr and 5hr (c-d)-T=500°C, and t=1hr and 3hr

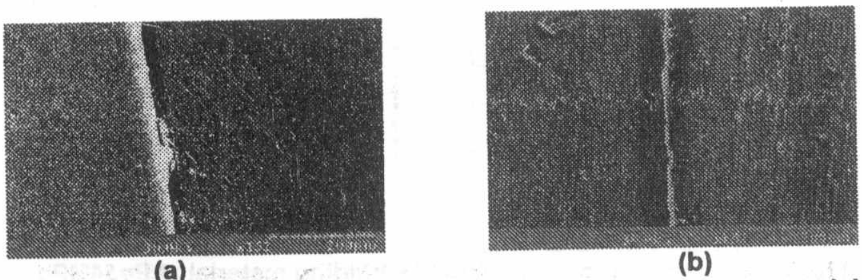
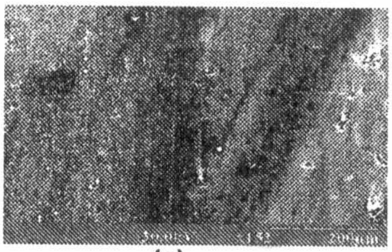
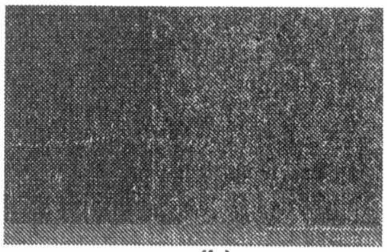


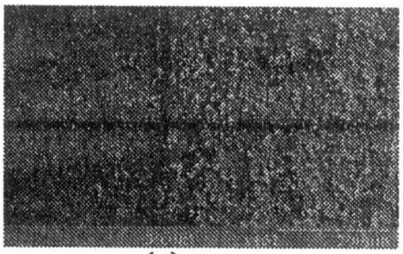
Fig.13. WDX analysis through the interface using brass as filling material at P=54 MPa ,T=450°C, and t=1hr showing line distribution of a- Cu & b-Zn



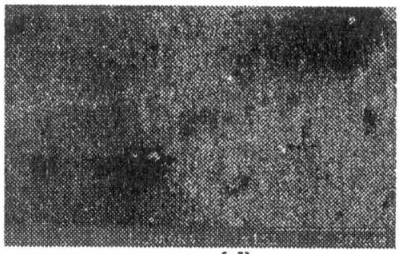
(a)



(b)



(c)



(d)

Fig.14. WDX analysis showing line distribution of Cu through the interface for prepared sample using brass as filling material at P=54MPa
(a-b)-T=450°C, t=3 & 5hr respectively (c-d)-T=500°C, t=1 & 3hr respectively

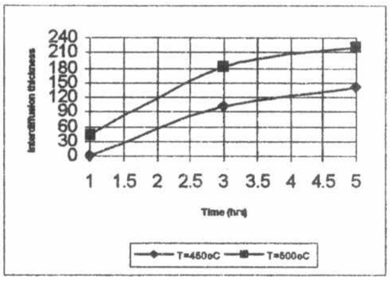
Table (7,8) Expected Al-Cu phases formed through the interdiffusion zone at T=450°C, t=5hr and T=500°C, t=3hr respectively

interface depth (μm)	Average Cu%	Average Zn%	Average Al%
40	40%	5%	55%
70	36%	8%	56%
90	25%	12%	63%
130	10%	14%	76%
160	0%	21%	89%

(7)

Depth of penetration (μm)	Average Cu%	Average Zn%	Average Al%
65	46%	4%	50%
90	39%	8%	53%
135	23%	12%	65%
190	13%	16%	71%
240	0%	21%	89%

(8)



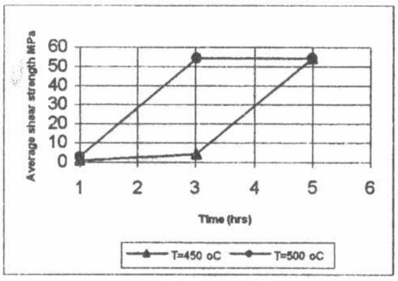


Fig.15. Samples prepared using brass as binding material at P=54MPa
a-Interface layer thickness at different holding times
b-Average shear strength of Al-MMC samples at different temperature.

Table 9 Interdiffusion zone characteristics and the mechanical properties of Al-laminated composite using brass as a binding material.

Bonding Parameter		Interdiffusion Thickness (µm)	Average Shear Strength (MPa)	Fracture Location
T(°C)	T(hrs)			
450	1	---	---	--
450	3	100	2.2	BI
450	5	140	54	BM
500	1	40	1.8	BI
500	3	180	54	BM
500	5	220	54	BM

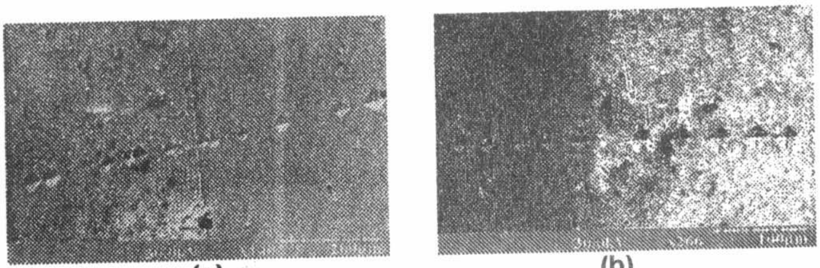

(a) (b)

Fig.16. Scanning electron micrograph of Al-laminated composite using brass as bonding material showing Vickers Micro hardness after age hardening, for a prepared sample at P=54Mpa a-T=450°C& t=5hrs b-T=500 °C& t=3hrs

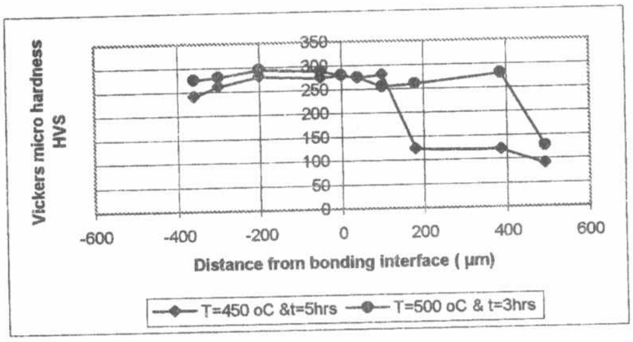


Fig.17. Vickers Micro hardness values from the interface for a prepared sample using brass as a binding material after aging at P=54MPa, T=450°C after 5hrs and 500°C after 3 hrs holding time