PROPERTIES OF SQUEEZE CAST Al - BASE COMPOSITE MATERIALS STRENGTHENED WITH δ - ALUMINA FIBRES

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Abstract

Porous δ-alumina fibre preforms containing 15 vol.-% of fibres were infiltrated by 6061 Al alloy (1.0 wt.-% Mg, 0.6 wt.-% Si, 0.5 wt.-% Cu and 0.2 wt.-% Cr) and T6 heat treated. Bonding at interface was investigated by a Scanning Electron Microscopy and revealed relative good bonding. In order to investigate the optimum heat treatment parameters solution treatment temperatures in the range of 475-550°C, solution treatment times of 0.5 -5 hours, temperatures of cooling water of 20 ,40, 60°C, age hardening temperatures of 160 - 250°C and age hardening times of 6 - 20 hours were applied. On the basis of hardness measurements the optimum T6 heat treatment parameters were choosen resulting in the maximum hardness in the range of 133-135 HB. After 1 hour annealing at temperature range of 20 - 300°C the maximum drop of hardness was only of 23 HB reaching after 1 hour annealing at 300°C hardness of 110 HB. Tensile properties at ambient and elevated temperatures (20 - 300°C) were investigated and the maximum tensile strength of 339 MPa and yield strength of 281 MPa at ambient temperature were achieved. At elevated temperatures tensile properties of fabricated composite materials are significantly higher than of unreinforced 6061 Al alloy. Composite 6061 Al-base materials according to their high mechanical properties and relatively low density can be applied in the aircraft design.

Introduction

Composite materials on aluminium alloy matrix strengthened with ceramic fibres are characterised by very good mechanical and development properties in the relatively wide range of temperatures and their high specific stiffnes defined as E/ρ [1-3] influences on their increasing applications especially in the aircraft design.

Composite materials can be manufactured by squeeze casting of porous ceramic fibre preforms [4-6] infiltration under atmospheric pressure [7] , infiltration under gas pressure [7-9], mixing of ceramic fibres in liquid metal alloy (compocasting) [10,11] and by powder metallurgy methods [12,13].

The final processing of cast composite materials strengthened with short ceramic fibres can be realised by plastic working [3,14,15] , machining which should be applied only for the final precise working due to the relatively high production costs of composite materials [16] and extreme tool wear due to the abrasion caused by the ceramic strengthening elements [17,18] as well as by pulse laser processing [19].

Materials and Processing

Composite materials were produced by squeeze casting technique applying infiltration of the fibre preforms of 85 % porosity from SAFFIL δ-alumina fibres with liquid aluminium 6061 alloy (1.0 wt.-% Mg, 0.6 wt.-% Si, 0.5 wt.-% Cu and 0.2 wt.-% Cr).

Mechanical properties of composite materials strengthened with ceramic fibres based on heat treatable Al matrix are dependent on two major factors:

- fibre strengthening
- metal matrix properties and for heat treatable light alloys on optimum heat treatment of matrix

Fibre strengthening depends on mechanical properties of fibres and especially on their good bonding at interface which should transfer maximum loads from matrix to reinforcement.

For metal matrix properties of 6061 Al alloy especially important are heat treatment operations but especially parameters of age hardening; underaging of matrix material leads to precipitation of small number of intermetallic strengthening compounds-especially Mg_2Si, Al_2Cu and Al_2CuMg what results in relatively low mechanical properties. On the other hand overaging results in coarsening of intermetallics and significant drop of mechanical properties (FIG.1).

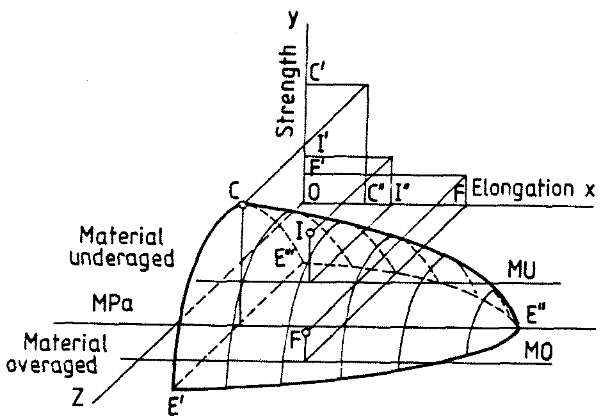

FIGURE 1. Effect of ageing parameters on properties of heat treatable light alloys

In order to define the effect of T6 heat treatment parameters (solution treatment and ageing) on hardness of 6061 Al-base composite materials they were heat treated applying the following procedure:

- solution treatment at temperatures of 475, 500, 510, 525, 550°C,
- solution treatment times of 0.5, 1, 2, 5 hours,
- temperatures of cooling water of 20, 40, 60°C,
- ageing temperatures of 160, 190, 220, 250°C,
- ageing times of 6, 12, 14, 16, 20 hours

After squeeze casting process Brinell hardness of composite materials was of 95 ±3HB and the lower hardness of 75 ±3 HB was obtained after 1 hour annealing at the temperature of 550°C and subsequent cooling of samples with the furnace. The highest hardness was reached for samples solution treated from the tempertature of 510 - 525°C (FIG.2a). Solution treatment of samples at the lower temperatures resulted in not full solution of α-phase and on the other hand annealing at higher temperature of 550°C leads to negligible melting of grain boundaries.

Effect of solution treatment time on hardness showed that the highest hardness was reached for samples solution trated during 1-2 hours (FIG.2b). Solution treatment during longer time of 5 hours caused significant grain growth finally leading to lower hardness of composite materials.

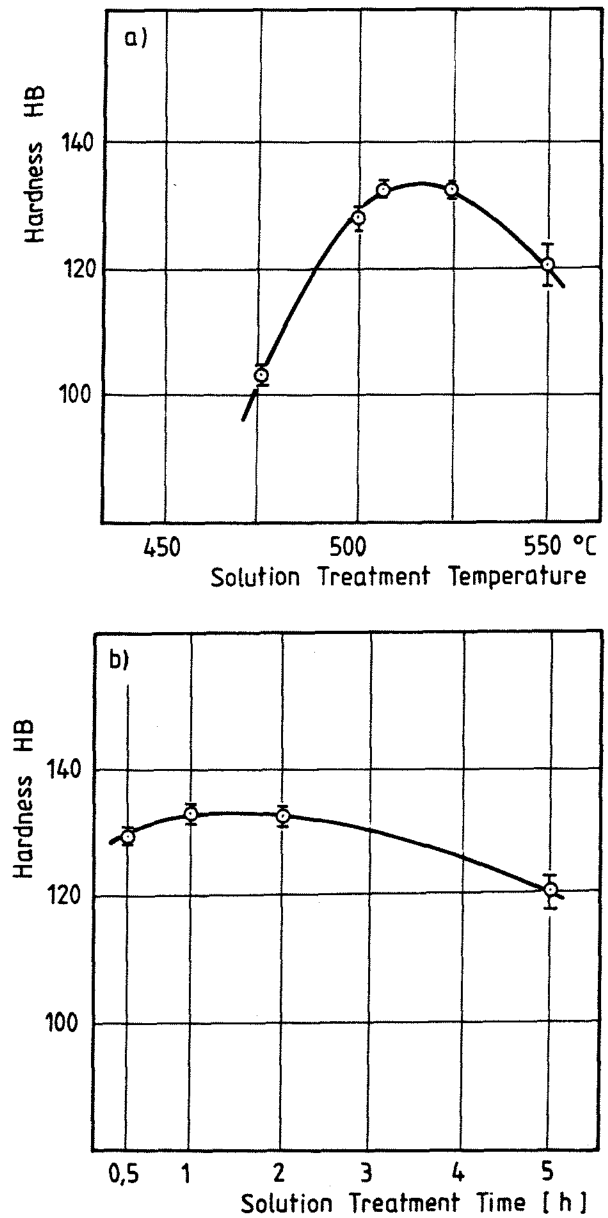

FIGURE 2. Effect of solution treatment parameters on Brinell hardness of 6061Al/15 vol.-% of δ-alumina fibres composite materials (for samples age hardened at 190°C for 14 h, cooling water temperature of 40°C),
a) effect of solution treatment temperature (for solution treatment time of 2 hours)
b) effect of solution treatment time (for solution treatment temperature of 510°C)

Samples after solution treatment were cooled in water of different temperaturs (20,40,60°C) and no influence of cooling medium temperature on hardnes was noticed.

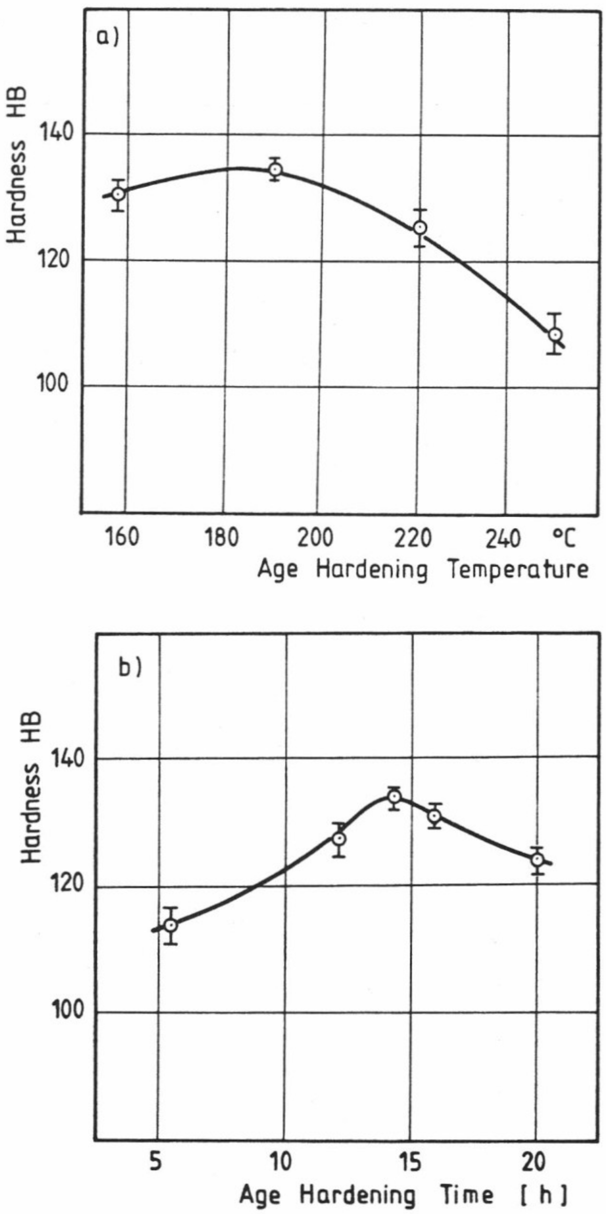

FIGURE 3.Effect of age hardening parameters on Brinell hardness of 6061Al/15 vol.-% of δ-alumina fibres composite materials (for samples solution treated at 510°C, during 2 hours,cooling water temperature of 40°C),
a) effect of age hardening temperature (for samples age hardened during 14 hours)
b) effect of age hardening time (for samples age hardened at 190°C).

The highest hardness was reached for composite materials age hardened at the temperature of 190°C and at higher age hardening temperatures there were noticed the gradual drop of hardness (FIG.3a) probably due to the growth of G - P zones. Drop of hardness was caused by application of very long age hardening times. The optimum age hardening time is 14 hours (FIG.3b).

On the base of performed investigations it can be concluded that there should be applied the following T6 heat tratment procedure in order to reach the maximum hardness of composite materials:

- solution treatment at temperature of 510°C during 2 hours,
- cooling in water of temperaure of 40°C,
- age hardening at 190°C during 14 hours.

Exprimental Results

Composite materials after production were charactерised by relatively homogeneously distributed ceramic SAFFIL fibres and structure without pores and bubbles (FIG.4).

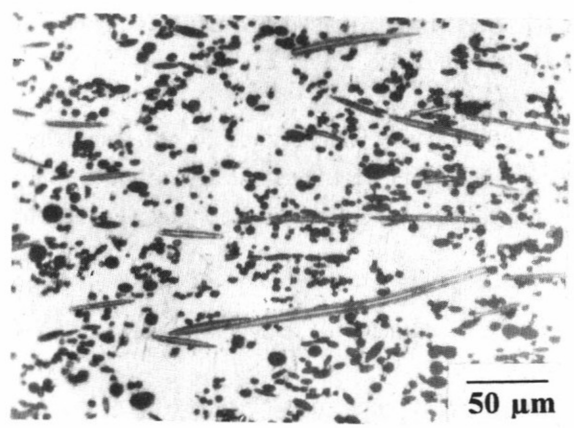

FIGURE 4. Microstructure (OM) of 6061Al/15 vol.-% of δ-alumina fibre composite material after squeeze casting.

Observations of composite materials microstructure after production by means of Scanning Electron Microscopy revealed (Fig.5) that some pores were present especially in places of fibres crossing and forming close areas where flow of liquid alloy was impossible. On the other hand bonding at fibre / matrix interface was relatively good and no bubbles or pores were found. Bonding at interface is very important factor and mechanical properties of composite materials with poor bonding can even be lower than propereties of unreinforced alloy.

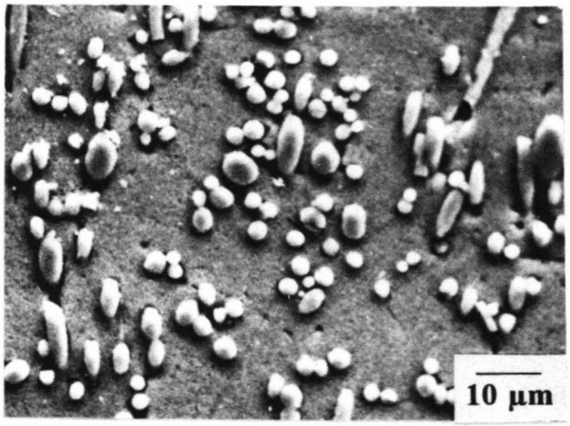

FIGURE 5. Microstructure (SEM) of 6061Al/15 vol.-% of δ-alumina fibre composite material after squeeze casting.

Hardness of produced composite materials was measured after optimum T6 heat treatment and after annealing for 1 hour at temperatures of 100, 150, 200, 250 and 300°C and is shown at FIG.6.It indicate that significant drop of hardness was noticed after annealing for 1 hour at temperatures of 250°C and it can be due to the coarsening of intermetallic Mg_2Si, Al_2Cu and Al_2CuMg compounds.

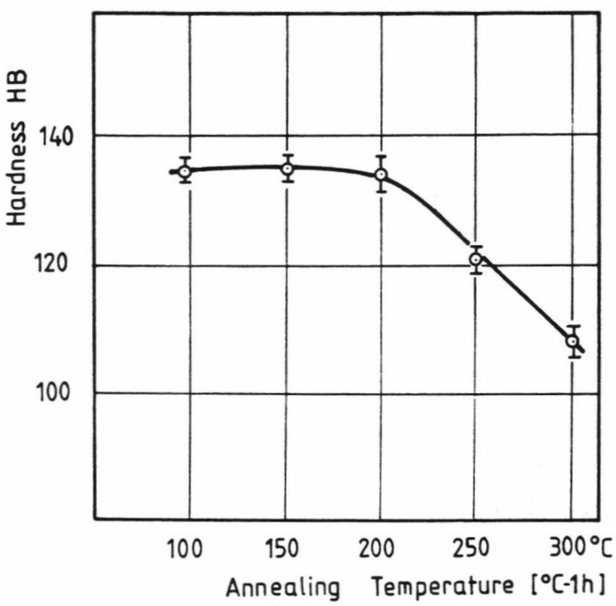

FIGURE 6. Effect of 1 hour annealing on hardness of 6061Al /15 vol.-% δ-alumina fibres composite material

Tensile and yield strength of composite materials was measured applying INSTRON tensile testing machine. Measurements were performed at ambient temperature and temperatures of 100, 150, 200, 250 and 300°C and results are shown at FIG.7 and FIG.8.

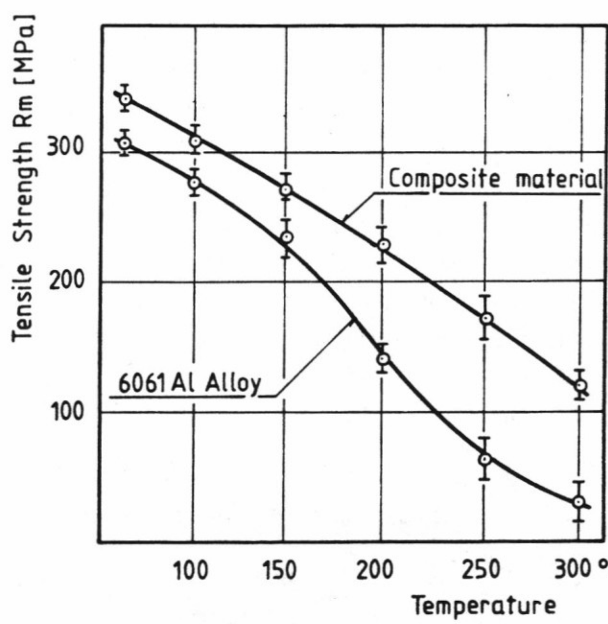

FIGURE 7. High temperature tensile strength of 6061Al/15 vol.-% of δ-alumina fibre composite material.

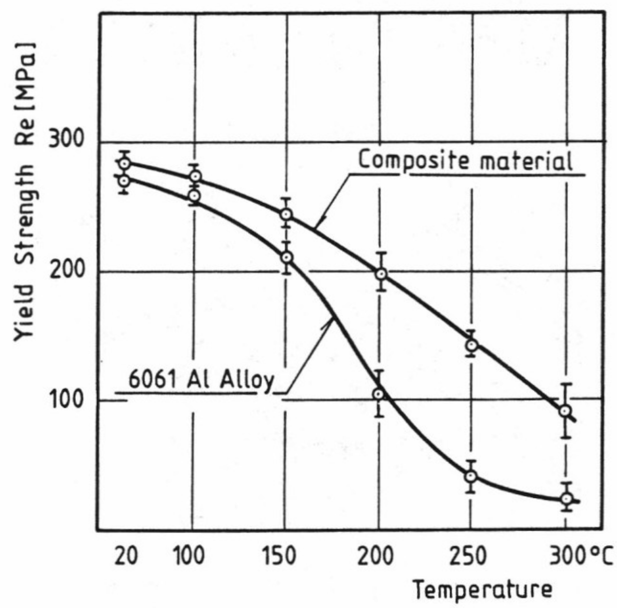

FIGURE 8. High temperature yield strength of 6061Al/15 vol.-% of δ-alumina fibre composite material.

They indicate that the maximum tensile strength of composite material at ambient temperature is 339 MPa and the significant drop of tensile strength was noticed at temperatures above 200°C what is due to the increasing plasticity of 6061 Al matrix and coarsening of intermatallic precipitates. But on the other hand at the very high testing temperatures exceeding 200°C there is significant improvement of high temperature tensile properties of fibre strengthened composite materials comparing to unreinforced 6061 Al alloy.

Observations of tensile fractures of composite materials loaded at the whole applied range of test temperatures showed that limited pull-out phenomenons of fibres from matrix were observed (FIG.9). It can be evidence of relatively good bonding at the interfaces in the range of applied testing temperatures.

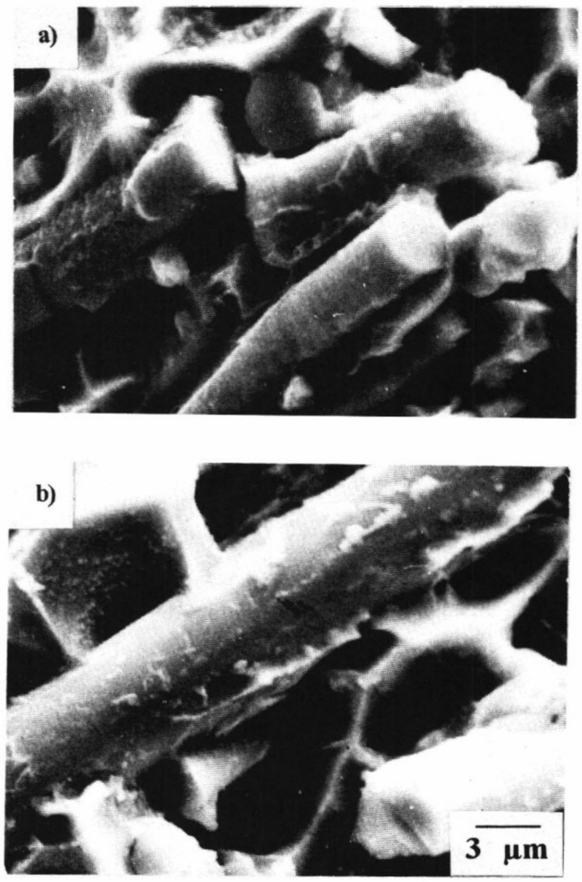

FIGURE 9. Fractures of 6061Al /15 vol.-% of δ-alumina fibre composite materials loaded at different temperatures,
a) at ambient temperature
b) at 250°C

Elongation to failure increased with increasing testing temperature; at ambient temperature elongation to failure was of 0.82% and increased to 3.17% at 300°C what was due to the increasing matrix plasticity, coagulation of strengthening intermetallic compounds and weakening of interface bonding.

Discussion of Results

Fabrication of Composite Materials

During high pressure infiltration of porous fibre preforms (squeeze casting) with liquid aluminium alloy the main goal of this process is achieving of good bonding between matrix and strengthening elements. The following factors influence on bonding at fibre/matrix interface:

- wettability between strengthening elements and matrix
- chemical reactions and occurence of reaction products at a interface
- viscosity of liquid alloy during squeeze casting process

Good wettability between matrix and strengthening elements will be obtained when the work of adhesion W_{ad} is the largest and it is calculated from the classical Dupre equation [20].

In composite materials strengthened with ceramic fibres, wettability at interface between liquid alloy and reinforcing elements can be improved during production process by means of the following methods [21-23]:

- covering of strengthening elements with metal or metal oxide layer
- modification of molten alloy with reactive elements like magnesium (Mg), calcium (Ca), titanium (Ti), zirconium (Zr) and phosphorus (P)
- thermal treatment of strengthening elements
- ultrasound treatment of liquid alloy during infiltration process

The wetting angle for Al - Al_2O_3 system depends upon temperature and in the temperature range of 670°C - 850°C is about 160° and above this temperature until 1030°C is about 70° [24]. On the other hand Laurent et al.[25] have said that the wetting angle between alumina single crystals and aluminium melt in vacuum depends upon a temperature as well and at the temperature of 700°C wetting angle θ is 101° and at 1000°C drops to θ = 86°. The relatively poor wettability of alumina by such electropositive metal like aluminium is due to the high reactivity of aluminium which is always covered with thin alumina layer. Due to the reported low wettability of alumina by aluminium the squeeze casting method for production of composite materials should be applied and other low pressure infiltration methods won't give good results.

Chemical reactions at the interfaces and forming of reaction products can lower the mutual bonding and on the other hand addition to melt of such elements like magnesium (Mg), zinc (Zn) and iron (Fe) in aluminium-alumina system can improve the bonding due to the formation of aluminates at the interface [26]. During the production process of composite materials strengthened with alumina reinforcing elements the aluminates can be formed due to the following chemical reactions[26]:

$Mg + 4/3\ Al_2O_3 = MgAl_2O_4 + 2/3\ Al$

$Mg + 2\ Al + 2\ O_2 = MgAl_2O_4$

$MgO + 2\ Al + 3/2\ O_2 = MgAl_2O_4$

$MgO + Al_2O_3 = MgAl_2O_4$

In Al - Mg basis alloys like 6061 during infiltration can be formed around strengthening fibres the magnesiu rich zones composed of $MgAl_2O_4$ spinels, magnesium oxide (MgO) and α alumina when applied reinforcing element are composed of such alumina [27].

On the other hand Wirth [28] gives reactivities in different composite systems and say that no reaction between alumina and aluminium at possible fabrication temperatures occurs (TABLE 1).

TABLE 1.Reactivity between metal matrices and reinforcing fibres [28].

Matrix	Fibres				
	B	C	SiC	Al_2O_3	ZrO_2
Mg				R>900	R
Al	R>700	R>500	R>700		R
Ti	R>900		NR<700	R>1400	R>1400

Description: R - reaction NR - no reaction

Clyne et al. [29,30] investigated grain size in composite materials with different aluminium alloys matrices like Al - Mg, Al - Cu strengthened with δ alumina fibres. They say that grain size depends on heat flow directions during solidification process. Comparing sqeeze casting processes of not reinforced alloys and reinforced alloys at the same temperature and pressure conditions they say that in composite materials grain sizes are 20 - 100 times smaller than in unreinforced alloys. Moreover in investigated alloys there were significant differencies of microhardness of matrix in regions characterized by small and large distancies between strengthening fibres. For instance in alloy Al -10% Mg strengthened with 30 vol.-% of SAFFIL δ-alumina fibres the matrix microhardness in regions characterised by the relatively small interfibre distancies was 1150 MPa and in region characterised by large interfibre distancies the microhardness was lower - namely of 950 MPa . In other composite material based on Al - 11.5% Si alloy strengthened with 24 vol.-% of δ - alumina SAFFIL fibres the measured microhardnesses were respectively of 960 and 720 MPa. Discussed variation in hardnesses can be explained by large thermal coefficients of expansion differencies and dislocation punching [31,32].

Very low viscosity of liquid aluminium of 1.30 mPa s measured at melting point [33] make easier the infiltration of fibre preforms by squeeze casting process.

Bonding

Bonding between ceramic reinforcing elements and metal matrix is of preliminary importance, because it effects on amounts of loads carried by reinforcing elements. Taking into account different kinds of regions at interface described by Taya, Dunn and Lilholt [34] and shown at FIG. 10 it can be concluded that in 6061 Al/ δ-alumina fibres composite materials precipitation free interface region with dislocation network is formed.

In fabricated composite materials relatively good mechanical bonding was ascertained because wetting of alumina by liquid 6061 aluminium alloy was improved according to relatively high temperature of 6061 Al melt. On the other hand X-ray microprobe analysis of interface did not show any reaction products at fibre/matrix interface .

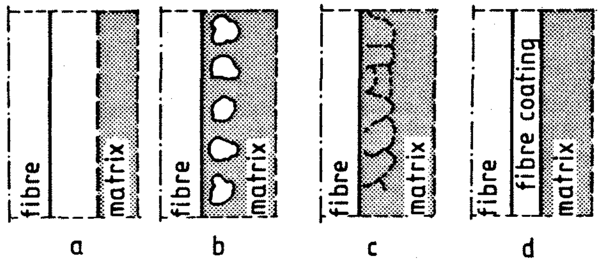

Figure 10. Character of interface regions between ceramic fibres and metal matrix [34]

a) precipitation free b) with reaction products

c) with dislocation network d) coated fibres

Heat Treatment

In performed investigations there were ascertained negligible acceleration of ageing proces comparing to unreinforced 6061 Al alloy.

Salvo et al. [35] investigated coposite materials fabricated by compocasting on the base of 6061 Al alloy strengthened with SiC and Al_2O_3 particles. They investigated ageing process at temperatures of 145, 175, 200, 220°C and by means of hardness measurements ascertained that below certain critical T_c temperature of 190°C ageing in composite materials is slower than in unreinforced 6061 Al alloy and over T_c temperature ageing rate of composite materials is higher than of unreinforced alloy. These phenomena can be explained by different precipitation mechanisms over and below critical T_c temperature. In alloy metall matrix by higher ageing temperatures nucleation of strengthening intermetallic compounds occurs heterogeneously at structure imperfections like for instance at dislocations.Nucleation and their growth will be faster in composite materials what is due to the increased dislocation density around ceramic strengthening elements (fibres, particles) generated by thermal mismatch between matrix and reinforcement. At lower ageing temperatures hardening of the matrix is rather due to the precipitation at GP zones and deceleration of ageing rate in composite materials comparing to unreinforced alloy can be due to the absence of GP zones caused by lack of quenched-in vacancies.

Ribes et al. [36] have investigated ageing process in composite materials on the base of AS7G03 (Al-7%Si-0.3%Mg) and 6061 alloys strengthened with 10 vol.-% of SiC particles fabricated by compocasting techniqe.By low ageing temperatures of 120°C alloy AS7G03 without strengthening elements ages faster than the composite material on the base of AS7G03 alloy strengthened with 10 vol.-% of SiC paricles. On the other hand by high ageing temperatures of 185°C ageing rate of composite material is much higher than of unreinforced alloy. They ascertained,that at temperature of 140°C both materials (composite material and unreinforced alloy) reach the maximum hardness after the same ageing time. When investigated ageing process of composite materials on 6061 basis and 6061 unreinforced alloy two ageing temperatures were applied: 220°C and 175°C and the ageing rate at 220°C was higher for composite material than for unreinforced alloy and at 175°C the maximum hardness of alloy was reached after the same time as of composite material. Acceleration of ageing process in composite materials at the relatively high ageing temperatures is due to the high dislocation density at the interface of reinforcing particles and matrix caused by thermal mismatch of composite components and additionally by thermal stresses caused by the same factor. Presence of high density interfaces can lower quenched-in vacancy concentration inhibiting GP zones formation at lower ageing temperatures.

Appendino et al. [37] investigated heat treatment of composite materials based on 6061 Al alloy reinforced with 14 vol.-% of SiC particles.Solution treatment was realized at temperatures of 529 and 557°C and ageing process was realized at ambient temperature and at 180°C. The artificial ageing was accelerated in the composite material what was due to the presence of zones of high dislocation density at 6061 matrix and reinforcing particles interfaces. On the other hand dislocations did not effect the kinetics of natural ageing and it can be concluded that the driving force for this process only depends on the concentration of alloying elements in the supersaturated solid solution. Hardening during natural ageing of unreinfoced 6061 alloy and composite on 6061 Al alloy base is due to the formation of primitive GP zones while in case of artificial ageing carried out at 180°C hardening is caused by formation of the needle-shaped phases and the concomitant partial precipitation of the rod-shaped phase.

Dutta et al. [38] investigated ageing process in composite materials on 6061 Al alloy basis strengthened with 10 and 30 vol.-% of SiC whiskers. They have ascertained that the optimum ageing time performed at 205°C for solution treatment performed at 530°C depends on volume content of whiskers. Accelerated ageing in composite material containing higher amount of whiskers is due to the platic deformation of matrix around strengthening fibres and with high dislocation density in these regions caused by thermal mismatch of matrix and fibres.They say [38] that this phenomenon is similar to faster age hardening of plastic deformed conventional unreinforced alloys. The additional following factors effect on accelerated ageing process in composite materials [38]:

- large cooling rate
- large thermal expansion coefficient mismatch
- large reinforcement volume fraction
- large but finite aspect ratio of fibres

Friend et al. [39] investigated ageing process in 6061 Al base composite material strengthened with 26 vol.-% of δ-alumina SAFFIL fibres. Composite material was pro - duced by squeeze casting of fibre preforms and subsequent T6 heat teeatment which consisted from solution treatment at 529°C, cooling in water and ageing during 30 minutes at temperature range 20 - 300°C.The maximum of hardness as function of ageing temperature applying ageing during 30 minutes was reached at 180°C for unreinforced alloy and for composite material. On the other hand hardness of squeeze cast composite material solution treated during 1 hour and aged during 0.5 hour at 180°C was maximum at the upper part of squeeze cast plate where the contact between molten matrix alloy and reinforcing fibres was relatively long. Hardness gradually decreased in direction of lower part of squeeze cast plate where this contact was relatively shorter. Longer solution treatment during 8 hours and ageing during 0.5 hours at 180°C make possible reaching of the same hardness on the height of squeeze cast plate. A result of interaction at the fibre/matrix interface can be removal of magnesium from matrix.This removal will be effected by variations in the liquid / fibre residence times and as result different amounts of β-Mg_2Si preci - pitates will be formed. Additionally silica used in porous fibre preforms as a binder can react with liquid matrix and effect on increasing of silicon content in metal matrix alloy.

Friend and Luxton [40] have found that the fibre content in composite materials based on 6061 Al matrix has significant effect on ageing process.Composite materials containing 5 vol.-% and 25 vol.-% of SAFFIL fibres were solution treated at 529°C during 1 hour in water and isochronally or isothermally age hardened.Isochronal ageing process was performed during 0.5 hours at the temperature range of 20 - 300°C.On the base of isochronal ageing process the optimum ageing temperature of 140°C was choosen in further isothermal ageing process. Additionally samples of composite materials were aged at ambient temperature. They say [40] that fibres in composite 6061 Al base material inhibit ageing process at ambient temperatures and hinder formation of GP zones.They ascertained that larger amount of reinforcing fibres in composite materials has larger hindering effect on formation of GP zones. Applying ageing at elevated temperatures increase of hardness depends on fibre content in composite material (smaller increase of hardness in composite materials with high reinforcing fibres content and larger increase of hardness in composite materials with small reinforcing fibres content).

Mechanical Properties

The maximum strength of 339 MPa was reached at ambient temperature and strength gradually decreased with increased testing temperature. Significant drop of strength was noticed above 200°C and it is due to increasing plasticity of 6061 Al matrix, coarsening of β-Mg_2Si precipitates and decreasing of bonding at interface. Additional factor effecting on high temperature strength can be destroying of fibres as a result of their rotation during tensile loading. On the other hand comparing strengths of 6061 Al-base composite material and unreinforced 6061 alloy there is significant improvement of tensile strength (TAB.2).

Ohori et al. [41] fabricated composite 6061 Al base materials reinforced with 17 vol.-% of SAFFIL fibres and after T6 heat treatment reached tensile strength of about 460 MPa. After extrusion the tensile strength increased to over 500 MPa.The significant drop of tensile strength was noticed at temperatures over 150 °C and at temperature of 300°C strengths of cast and extruded composite materials were of about 200 MPa.

TABLE 2. Tensile strength of composite material 6061Al / 15 vol.-% of δ-alumina fibres and relative growth in comparison with unreinforced 6061 Al alloy

Material	6061 Al	6061 Al composite	Increase
Temp. °C	Tensile strength MPa		%
20	310	339	9.4
100	278	311	11.4
150	237	268	13.1
200	140	228	62.8
250	63	170	169.8
300	30	118	293.3

On the other hand Friend [42] fabricated composite material based on Al-4.0% Zn-2.0% Mg alloy strengthened with 25 vol.-% of SAFFIL fibres and at ambient temperature no significant increase of tensile strength was noticed. Improvement of tensile strength in comparison with unreinforced Al-4.0% Zn-2.0% Mg alloy takes place at elevated temperatures.

Very important parameter of composite material is the Young's moduli effecting on possibility of practical applications. Clegg et al [43] ascertained influence of strengthening fibre amount in composite material on E-moduli when investigating composite materials containing 10, 20 and 30 vol.-% of SAFFIL fibres with the following matrices: 2024 T4, 6061 T4, Al-2.5% Mg and CP Al. There is very distrinct relationship between content of strengthening fibres and E-moduli in powder-metallurgically fabricated composite materials on 6061 Al basis strengthened with 5, 10, 15, 20 and 30 vol.-% of SAFFIL fibres [44].

Elongation to failure increases with increase of testing temperature what is due to increased plasticity of matrix and coarsening of β-Mg_2Si precipitates but this parameter is much smaller than in unreinforced 6061 Al alloy.

Applications

Metal Matrix Composites are characterised by very good mechanical and development properties at a wide range of service temperatures and especially their good fatigue and creep properties make them very useful in aircraft design. Taking into account such airframe design criteria like low vulnerability, maintainability, high reliability and extended life it can be concluded that such airframe elements like landing gears, fuselage, fittings can be designed from MMC's. Composite 6061 Al-base material strengthened with alumina fibres posses medium tensile properties and can be applied for not extremly loaded airframe elements.

Acknowledgement

This paper was prepared within the framework of research project nr. 7 S201 076 04 funded by the Komitet Badañ Naukowych (Committee for Scientific Research) in Warsaw - Poland. This project is currently performed at Politechnika Wroclawska from 1993 - 1995 and authors would like to thank for funding.

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