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DEVELOPMENT OF AL-AL₂O₃ MMCS BY DIMOX & DEVELOPED COMPOSITES CHARACTERIZATION

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ABSTRACT

Amongst several metals, Aluminium (Al) is attractive due to its ductility, malleability, good conductivity, light weight, good strength, availability in abundance (8% of earth crust is aluminium). Alumina (Al2O3) is the most cost effective and widely used material in the family of engineering ceramics for preparing Al based metal matrix composites. In the present work, the investigators have attempted to develop aluminium based composites prepared by direct oxidation of aluminium under ambient conditions using stir casting technique. The process parameters have been varied in limited range and their effect on microstructure and mechanical properties of the composites is discussed

KEYWORDS: Metal Matrix Composites, DIMOX, Stir Casting.

INTRODUCTION

Aluminium based metal matrix composites are finding versatile application in industries. Composites are tailor made material. These materials contain two major components; the matrix and; the reinforcement. Matrix component is more in quantity (as per the definition of MMCs, greater or equal to 5% by wt) and reinforcement is a contrasting phase distributed in matrix in order to reinforce the matrix. The reinforcement does not make solid solution with the matrix rather; it is physically distributed all around the matrix and results in its reinforcement. Usually the matrix and reinforcement material have contrasting physical and mechanical properties.

Aluminium is a soft, durable, lightweight, and malleable metal with appearance ranging from silvery to dull gray, depending on the surface roughness. It is nonmagnetic. It is also insoluble in alcohol, though it can be soluble in water in certain forms. The yield strength of pure Al is 7–11 MPa, while its alloys have yield strengths ranging from 200 MPa to 600 MPa [1]. Aluminium has about one-third the density and stiffness of steel. It is easily machined, cast, drawn and extruded. A thin layer of aluminium can be deposited onto a flat surface by physical vapors deposition or (very infrequently) chemical vapors deposition or other chemical means to form optical coating sand mirrors. When so deposited, a fresh, its pure film serves as a good reflector (approximately 92%) of visible light and an excellent reflector (as much as 98%) of medium and far infrared radiation.

Liquid state fabrication of Metal Matrix Composites involves incorporation of dispersed phase into a molten matrix metal, followed by its solidification. In order to provide high level of mechanical properties of the composite, good interfacial bonding (wetting) between the dispersed phase and the

liquid matrix should be obtained. Wetting improvement may be achieved by coating the dispersed phase particles. Proper coating not only reduces interfacial energy, but also prevents chemical interaction between the dispersed phase and the matrix. The method of liquid state fabrication of Metal Matrix Composites is Stir Casting [2].

Several researchers have also prepared composites by direct oxidation of matrix material. This technique is commercially known as DIMOX [3]. Direct oxidation is an innovative technique developed to produce ceramic-metal composites by exposing molten metal to oxidizing atmosphere at relatively high temperatures, more than 900 $^{\circ}$ C [4].



Fig. 1 Schematic Diagram of Dimox Process.

The final product is a three-dimensional, interconnected, interpenetrating network of oxide and micro-channels of metal alloy. The reaction product grows towards the oxidizing atmosphere as shown schematically in Fig. 1. The reaction is sustained by wicking of the molten alloy to the surface by capillary forces. The metal is the main component of the alloy, which is oxidized to form in-situ ceramic phase like Al_2O_3 etc. Elements such as magnesium and zinc are present in the melt in small quantities, less than 10%, and help to initiate the oxidization reaction by forming a porous spinel compound and thereby preventing formation of a passive oxide film at the surface as normally occurring in pure aluminium. The alloying elements also alter the surface energies of the melt to facilitate the wetting of the oxide [5].

Several researchers [6] have shown that the cost of manufacturing of composites materials using a conventional casting method is about one-third to half as that of competitive methods and, for high volume production, this cost is expected to reach the level of one-tenth.

The literature review and aspects associated with the processing and properties of composites indicate that extensive research has been conducted on the development of ex situ and in-situ PMMCs. These studies show that:

- 1. Al and its alloys are widely used as matrix material.
- 2. Liquid metallurgy route in general and stir casting in particular is the widely preferred, simple and economical processing method for composites. It is also the only commercially viable technique at present.
- 3. Reinforcements improve the mechanical properties of materials.
- 4. The ex-situ formed composites have reinforcements with size of the order of tens of microns. Amongst several dispersoids, alumina particles are favored for Al and Al-alloy matrix because there is chemical compatibility and no undesirable reaction takes place between the matrix and the reinforcing particles which may lead to degradation of properties.
- 5. The in-situ formed reinforcements are fine in size, of the order of few microns. These reinforcements are thermodynamically stable and do not dissolve at elevated temperatures and have clean interface with the matrix. Several oxides such as TiO₂, MnO₂, SiO₂, CuO, B₂O₃, MoO₃ and ZnO etc. are capable of initiating substitution reaction with Al or its alloys to generate in-situ fine Al₂O₃ particles within the Al melt.
- 6. Heating of powder before addition in to the melt improves wettability.
- 7. As noted earlier [7], a comprehensive understanding of solidification mechanism in synthesis of cast composites has to be achieved for evolution of microstructure and origin of defects before commercial exploitation of this technique to fabricate various components. The control of macro/microstructure may determine the mechanical and tribological properties, providing the basis for using these composites in various applications.

In the present work the investigators have attempted to develop composites prepared by direct oxidation of aluminium under ambient conditions in the laboratory in order to exploit the effect of ambient temperature on composites in terms of its microstructure and mechanical properties and to explore whether oxidation which is a highly undesirable naturally occurring phenomenon, can be used in turn to develop composites. The composites have been developed by stir casting and categorized for mechanical properties. Results have been reported and discussed. Simple oxidation process for synthesis of composites is represented in Fig. 2.



Fig. 2 Schematic Diagram of Simple Oxidation Technique Employed in Present Study.

The present study aims at:

- To fabricate PMMCs by liquid metallurgy through stir casting technique developed by Ray, 1993
 [7] using simple oxidation technique as already indicated in DIMOX process but under ambient conditions to minimize cost.
- 2. To investigate the microstructures of developed composites using optical microscope and SEM.
- 3. To investigate the mechanical properties of the composites.
- 4. To establish a correlation (if any) between microstructures and mechanical properties.

EXPERIMENTAL SET UP

Composition of Aluminium was determined by ED-XRF spectrophotometer make Fischer, Germany and is given in Table I. MMCs were prepared by melting aluminium in a 2.2 kW Kanthal wound resistance heating furnace. After superheating Al melt at 850 °C and holding at this temperature in ambient conditions for several hours, melt was stirred at 800±50 rpm by a turbine blade type of stirrer driven with 1440 rpm, 0.5 HP motor. The stirred slurry was poured in alloy steel die at pouring temperature of 750 °C. Hardness measurements were done on the composites using Vickers Hardness Testing Machine (model-VM 50PC, make FIE Kolhapur) at a load of 5 kg applied for 15 s with diamond indenter as per ASTM E92. Tensile testing was carried out on Tensometer, PC 2000 (Hounsfield type, make-Kudale Instruments Pvt. Ltd., Pune, India) at an extension rate 1 mm/min. At least two specimens conforming to ASTM E8M standard with diameter 5 mm, gage length 25 mm, were tested for each condition and the mean of the readings, is reported.

Microstructures were studied on optical microscope, make Metzer, Mathura-India and on FE-SEM with EDAX model QUANTA 200 FEG (make- FEI Netherlands, Resolution 2 nm, HV 200V-30 kV). Etching was done by Keller's Reagent (1.25 ml HNO₃, 0.75 ml HCl, 0.5 ml HF and 47.5 ml H₂O, applied for 40 s).

RESULTS AND DISCUSSIONS

Chemical composition of Al used in present study is given in Table I.

Material	Chemical composition (wt%)									
	Co	Si	Fe	Zn	Sn	Ni	Mn	Cu	Pb	Al
Al-	0.01	0.50	0.07	0.03	0.69	0.02	0.01	0.06	0.06	98.54
Ingot										

Table : 1. Chemical Composition of Al Used in Present Study

Four types of composites were developed by varying the process parameters in limited range, these are designated as A1, A2, A3 and A4 respectively. The details of composites developed and specific process parameters are given in Table II.

Table:2.Composites Prepared and Their Id Nos. for Further Reference in the text

ID No.	Composite		Group Assigned		
		Holding Time (h)	No. of stirrings (3 min each)	Temp.	
A1	Al-Al ₂ O ₃	3	4 times (Over each 45 Min.)	850 °C	А
A2	Al-Al ₂ O ₃	1	1 time	850 °C	А
A3	Al-Al ₂ O ₃	2	1 time	850 °C	А
A4	Al-Al ₂ O ₃	3	1 time	850 °C	А

SEM Microscopy of Al-Al₂O₃ Composites

Formation of oxide layer at high temperature in Al melt is already known and present study is based on preparing Al based metal matrix composites by breaking this oxide layer through stirring and attempting to dissolve this layer of oxide in molten aluminium metal.



Fig. 3 (A) and (B) Show Unetched SEM Micrographs of Al-Al₂O₃ Composite (A2) Prepared by 1 H Holding and Stirred once at 500X and its Composition Analysis by EDX Respectively.

It is expected that fine Al_2O_3 particles may be observed in matrix of aluminium. Fig 3(a) shows SEM pictures of composites A2, fine particles are seen distributed in the matrix. Very fine spots are visible which may be Al_2O_3 particles. Fig. 3 (b) shows composition of the matrix which is predominantly aluminum.





Fig. 4 (A) Shows Fine Particle White in Colour Marked in the SEM Micrograph of Composites Al at High Magnification. this Particle is Identified as Al₂O₃ in Fig. 4 (B) By EDX. Grain Boundaries and Pores are also Visible Clearly.



Fig. 5 (A) Shows Unetched SEM Micrographs of Al-Al₂O₃ Composite (A1) Prepared by 3 H Holding at 850 ^oc and Stirred 4 times at 3000X. (B) Composition Analysis of Bright White Particle by EDX.



Fig. 6 Shows Unetched SEM Micrographs of Al-Al₂O₃ Composite (A1) Prepared by 3 H Holding at 850 ^Oc and Stirred 4 times at; (A) At 50X; (B) at 100X; (C) at 500X; (D). at 5000X

SEM micrograph of composite A1 is also shown in Fig. 5(a). Another Al_2O_3 particle is identified by EDX in Fig. 5(b). Pores filled with polishing debris and grain boundaries are clearly visible.

Unetched SEM micrograph of composite A1 are shown in Fig 6 (a) through (d) at different magnifications. Particle signatures of very fine size are visible in Fig 6 (a) to (c). In Fig 6 (d) a fine Al_2O_3 particle of the order of one micron is visible neatly bounded with the matrix.

Optical Microscopy of Al-Al₂O₃ Composites (A1)

Fig 7 (a) shows unetched optical micrograph of Al-Al₂O₃ MMC at 100X, prepared at holding time of 3 h, 4 times stirred each after 45 min interval at 850^{0} C (Composite No. A1). A dark spot is observed which may be due to accumulation of debris of polishing paste in the voids created due to porosity. Some fine alumina particles are also observed in the matrix which may have formed as a result of oxidation of aluminium. It is therefore apparent that the oxide layer has got broken in to fine particles during stirring because there was no other means by which these fine particles may have come in the matrix. The casting was done in dust free environment so the question of dust particles or silica entrapment in this much quantity may not be possible. Moreover, it was confirmed in EDX examination (Fig. 4 and 5) that the particles of this size range are none other than Al₂O₃. So, whatever is visible in the matrix may have resulted due to pure aluminium, grains, dendrites (primary and secondary), cells formed due to dendrites and Al₂O₃ particles formed due to oxidation.





Fig 7 Optical Micrograph of Al-Al₂O₃ Composite at 100X, Holding 3 H, 4 times Stirred Each after 45 Min Interval at 850 ^oc; (a) Unetched and; (b) Etched.

Fig 7 (b) shows alumina particles and formation of grains due to intersection of crystal planes in polycrystalline material like aluminium. Etching by Keller's reagent has highlighted the grain boundaries. Grains are large in size indicating soft matrix material.



Fig. 8 Unetched Optical Micrograph of Al-Al₂O₃ Composite at 500X, Holding 3h, 4 times Stirred Each after 45 Min Interval at 850 ^oc.

Fig 8 shows unetched micrograph at 500X showing distribution and clustering of particles, somewhere agglomeration is visible due to failure in breaking of oxide layer as larger chunks or due to accumulation of debris generated during polishing in pores. Scratches are largely visible in matrix as a result of poor polishing.



Fig. 9 Etched Optical Micrograph of Al-Al₂O₃ Composite (A1) at 500X, Holding 3 H, 4 times Stirred Each after 45 Min Interval at 850 ^Oc

Etched micrograph of A1 in Fig. 9 shows some porosity, particles of Al_2O_3 in the size, range of 2-5 μ m. Some grain boundaries are also reflected in the microstructure.



Fig. 10 Unetched Optical Micrograph of Al-Al $_2O_3$ Composite (A2) at 100X (Hold time 1 hour) @ 850^{0} C at100x.

Fig 10 shows unetched microstructure of $Al-Al_2O_3$ composite at 100X at holding time of 1 hour. On comparing with Fig 7 (a) it is observed that particles are present in chunks of broken layer rather than uniformly distributed layer.



Fig. 11 Etched Optical Micrograph of $Al-Al_2O_3$ (A2) Composite at 100X (Hold time 1 hour) at $850^{\circ}C$ at 100X.

Fig. 11 shows etched micrograph displaying predominantly dendrites solidification with primary and secondary arms, a signature of typical cast structure.



Fig. 12 Unetched Optical Micrograph of Al-Al₂O₃ Composite (A2) at 100X (Hold time 1 h) at 850°C.

Fig 12 shows higher magnification of composite A2. Some needle like phases are observed which may be due to local stresses, scratches or partially highlighted grain boundaries.



Fig. 13 Etched Optical Micrograph of Al-Al₂O₃ (A2) Composite At 500X (Hold Time 1 Hour) At 850^oC.

Fig 13 shows etched micrograph of A2 at 500X with traces of over etching. Micrograph details are clearly visible.





Fig 14 shows unetched microstructure of composite A3 at 100X at holding time of 2 h, somewhat similar to Fig 9 but particles are located in smaller chunks over the matrix. A void is also observed in the centre which may have resulted due to porosity and the black colour of void may be due to accumulation of debris during polishing.



Fig. 15 Etched Optical Micrograph of Al-Al₂O₃ Composite (A3) at 100X (Hold time 2 h) at 850^oC.

Fig 15 shows highlighted grain boundaries of composite A1 due to etching. The Fig shows recognizable grain sizes.



Fig. 16 Etched Optical Micrograph of Al-Al_2O_3 Composite (A3) at 500X (Hold time 2 h) at 850° C.

Fig 16 shows etched micrograph of composite A3 at 500X. Cell boundaries are visible clearly; particles and porosity are also clearly observed.



Fig. 17 Unetched Optical Micrograph of Al-Al₂O₃ Composite (A4) at 100X (Hold time 3 h) at 850^{0} C.



Fig. 18 Etched Optical Micrograph of Al-Al₂O₃ Composite (A4) at 100X (Hold time 3 h) at 850⁰C.

Fig 17 and 18 show unetched and etched micrograph of composite A4 prepared by holding time 3 h. Good distribution of particles and dominant cast structure is observed.



Fig. 19 Unetched Optical Micrograph of Al-Al₂O₃ Composite (A4) at 500X (Hold time 3 h) at 850° C.



Fig. 20 Unetched Optical Micrograph of Al-Al₂O₃ Composite (A4) at 500X (Hold time 3 h) at 850^{0} C.

Fig 19 and Fig 20 shows unetched and etched micrographs of composite A4 at 500X. Dendrite arms are visible where particles have clustered. Primary and secondary dendrite arms are highlighted in etched micrograph.



Fig. 21 Shows a comparison of etched micrograph of Composite A1 at; (a) 100X; (b) 500X.

Fig 21 shows comparison of etched microstructure of composite A1 at 100X and 500X respectively prepared by 3 h holding and intermittent stirring. It is apparent that particles are more uniformly distributed by intermittent stirring and cast structure is less dominant when stirring is done at regular intervals



Fig. 22 Etched Micrograph of Composite A4 at; (a) 100X; (b) 500X.

Etched micrographs of composite A4 at 100X and 500X are shown in Fig 22 (a) and (b) respectively. At higher magnification, dendrite cell boundaries are visible, particles are segregated at several locations.



Fig. 23 Etched Micrograph of Composite A2 at; (a) 100X; (b) 500X.

Fig 23 shows micrograph of composite A2 prepared by 1 h holding time and stirred once at 100X and 500X. Secondary dendrites are appearing. Several bubbles are also observed which may be due to over etching.



Fig. 24 Etched Micrograph of Composite A3 at; (a) 100X; (b) 500X.

Fig 24 shows etched micrographs of composite prepared by 2 h holding and stirred once at 100X and 500X respectively. Longer holding time has resulted in finer particles and dendrite cell structure in the matrix. Some scratches and impurities are also visible in the matrix. Etchant has highlighted few grain boundaries but the dark lines observed in the micro structure may be primary dendrites, arms and scratches on the matrix. Particles are also observed uniformly distributed throughout the matrix.

C. Mechanical Properties of Al-Al₂o₃ Composites

Hardness of Al-Al₂O₃ Composites

The basic principle, as with all common measures of hardness, is to observe the questioned material's ability to resist localized plastic deformation from a standard source. The Vickers test can be used for all metals and has one of the widest scales among hardness tests. The unit of hardness given by the test is known as the Vickers Pyramid Number (HV) or Diamond Pyramid Hardness (DPH).

In this work, the Vicker's hardness testing equipment is used to find out the hardness of the test specimens. Hardness testing was done at 5 Kg load. The results of hardness, tensile properties and impact strength of Al-Al₂O₃ composites (A1 to A4) are given in Table III.

ID No.	Composite	Hardness HV5	UTS	Proof Stress	Ductility (% elongation)	Impact Strength [J]
B1	Al-5%MnO ₂	39.18	64.1	38	21.28	27
B2	Al-5% Al ₂ O ₃	37.9	63.7	36	38.36	26
B3	Al-6% Al ₂ O ₃ (MnO ₂)	40.02	65.2	39	19.06	28
B4	Al-7% Al ₂ O ₃ (MnO ₂)	45.56	72.5	51	21.35	30

Table: 3. Mechanical Properties of Al-Al₂0₃ Composites.

Hardness of $Al-Al_2O_3$ composites are shown in Fig 25. Hardness of composite A1 prepared by 3 h holding and intermittent stirring is slightly better than hardness of composite prepared by stirring once which may be due to better distribution of particles in the matrix.



Fig 25 Shows Vickers Hardness Values at 5 Kg Load for Al-Al₂O₃ Composite; (A1) Prepared by 3 H Holding and Stirred 4 times; (A2) Prepared by 1 H Holding and Stirred once; (A3) Prepared by 2 H Holding and Stirred once and; (A4) Prepared By 3 H Holding and Stirred once.

Lesser holding time has resulted in decrease in hardness due to less time for formation of oxide layer on the surface of Al and hence lesser number of particles. Hardness increases with both; increase in holding time at superheated temperature due to more oxidation and; also by increase in number of stirrings which may be due to better dissolution of oxide layer in the matrix and uniform distribution of particles in the matrix. Although, there is very little recognizable difference in hardness of composites A1 to A4.

Tensile Properties of Al-Al₂O₃ Composites

A tensometer is a device used to evaluate the Young's modulus (how much it stretches under strain) of a material and other tensile properties of materials, such as tensile strength. It is usually a

universal testing machine loaded with a sample between 2 grips that are either adjusted manually or automatically to apply outwards force to the specimen along the axis. The machine works either by driving a screw or by hydraulic ram.



Fig 26 Shows UTS in [Mpa] for Al-Al₂O₃ Composite; (A1) Prepared by 3 H Holding and Stirred 4 times; (A2) Prepared by 1 H Holding and Stirred once; (A3) Prepared by 2 H Holding and Stirred once and; (A4) Prepared by 3 H Holding and Stirred once.

Here investigators have used tensometer to determine the tensile strength, 0.2 % offset proof stress and ductility of the prepared composites.

UTS of composite A1 is highest followed by A4, A3 and the lowest UTS is reported for composite A2 as observed in Fig 26. The tensile behavior is in accordance with the hardness and this trend may be attributed to; (i) increase in number of particles in composite A1 due to the larger holding time and stirring the slurry four times and; (ii) tensile strength increases by increase in holding time which may again be attributed to more oxidation at larger holding time and formation of more number of particles.



Fig 27 Shows 0.2% Offset Proof Stress in [Mpa] for Al-Al₂O₃ Composite; (A1) Prepared by 3 H Holding and Stirred 4 times; (A2) Prepared by 1 H Holding and Stirred once; (A3) Prepared by 2 H Holding and Stirred once and; (A4) Prepared by 3 H Holding and Stirred once.

The trend of 0.2% offset proof stress is exactly the same as that of UTS as observed in Fig 27.



Fig 28 Shows ductility (% elongation) for Al-Al₂O₃ composite; (A1) prepared by 3 h holding and stirred 4 times; (A2) prepared by 1 h holding and stirred once; (A3) prepared by 2 h holding and stirred once and; (A4) prepared by 3 h holding and stirred once.

The ductility of the composite is measured in terms of percent elongation and it is found that as the tensile strength is increasing, ductility is decreasing. Ductility is highest in A2 and lowest in composite A1. This behavior may be explained by increase in brittleness of composite by increasing addition of Al_2O_3 particles which are produced in large number as the holding time is increased and the number of stirrings is also increased. More particles result in higher reinforcement and therefore greater obstruction to the movement of dislocation plane during elongation and hence reduced ductility.

The impact strength (impact toughness) of $Al-Al_2O_3$ composites is represented in Fig. 29 by performing charpy test as per standard ASTM E2248-09. It is observed that the impact strength of composite A1 is highest followed by A4, A3 and A2 respectively.



Fig 29 Shows Impact Strength in Joules for Al-Al₂O₃ Composite; (A1) Prepared by 3 H Holding and Stirred 4 times; (A2) Prepared by 1 H Holding and Stirred once; (A3) Prepared by 2 H Holding and Stirred once and; (A4) Prepared by 3 H Holding and Stirred once.

This behavior may be attributed to high hardness and tensile strength of composites A1 and A4 as compared to A3 and A2 due to better reinforcement and more number of particles as evident in optical and SEM micrographs.

Al-Al₂O₃ composites have been successfully prepared in present study by direct oxidation in order to explore whether Al-Al₂O₃ composite can be formed under ambient conditions and also to determine the effect of holding time and number of stirrings on the microstructure and mechanical properties of the composites. Al-Al₂O₃ composites were synthesized successfully under ambient conditions and it was found that the Al₂O₃ particles increased as the holding time and number of stirrings was increased. Generation of Al₂O₃ particles in the matrix was confirmed by EDX examination. The mechanical properties improved as the holding time and number of stirrings was increased. Porosity and typical cast structure was predominant in the composites. As the number of particles was increased, ductility was reduced. Hence, oxidation which is known as a major problem in composite preparation by liquid metallurgy route can be used for preparation of composites and for improving the properties.

CONCLUSIONS

Following broad conclusion can be drawn from this study.

- Metal matrix composites can be successfully prepared by direct oxidation under ambient conditions. Hence, oxidation which is known as a major problem in composite preparation by liquid metallurgy route can be used for preparation of composites and for improving the properties.
- 2. As the aluminium melt is superheated, an oxide layer is formed at its surface and if the melt is stirred, this oxide layer breaks into fine particles of Al₂O₃ and these particles are dispersed in the matrix.
- 3. The dispersed particles are of size range 1 to 5 microns.
- 4. Micrographs have shown that due to prolonged holding and stirring, voids due to porosity are formed in the composites. These voids are large, both in number and size.
- 5. As the holding time increases, number of particles distributed in the matrix increase and the mechanical properties of composites improves but, ductility decrease.
- 6. As the number of stirrings is increased, the number of dispersed particles increases, mechanical properties improves but ductility decrease.

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