

*Executive Summary of the Thesis Entitled*

# **Processing and Characterization of In-Situ Aluminium Matrix Composite by Stir Casting Method**

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## **Doctor of Philosophy**

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## **Background**

Aluminium is most versatile metal because of unique combinations of properties which can be achieved through various routes and processing methods. This makes aluminium and its alloys most suitable for various applications from soft and thin wrapping foil to giant machinery parts such as aerospace, automobile, defence, domestic purpose, etc. since past many years. Many advance applications due to some of the excellent properties at low cost. Major properties which are concerned in aluminium and its alloys are strength, density, durability, machinability, etc. Aluminium can resist all types of oxidation compare to ferrous alloys. This is due to spontaneous formation of highly coherent and continuous inert  $\text{Al}_2\text{O}_3$  thin film over the surface of the aluminium.

Composite is the tailor made materials so one can adjust the properties as per the requirements. Making a good interface between the matrix and reinforcements in the composite materials is prime requirement of the composite making industries. The stress which is applied on the composite should be shared across the interface. Making of interface becomes even more difficult when the reinforcement materials are immune to the chemical reactions. Ceramic particles are non reactive to even at higher temperature and hence there will be wettability issue.

Use of aluminium to make aluminium matrix composite is trend now-a-days either by powder metallurgy route or casting route. Aluminium as the matrix metal can serve many useful properties such as improvement in ductility, strength, toughness, hardness, etc. Aluminium Matrix Composites (AMCs) can be classified into four types depending on the type of reinforcement added into the matrix like:

- Particle-reinforced AMCs (P-AMCs)
- Whisker-or short fibre-reinforced AMCs (SF-AMCs)
- Continuous fibre-reinforced AMCs (CF-AMCs)
- Mono filament-reinforced AMCs (MF-AMCs)

There are various processing methods to fabricate the particle-reinforced aluminium metal matrix composites (P-AMCs). But based on the addition of reinforcement particles to the metal matrix, there are two different processing techniques like ex-situ and in-situ.

In ex-situ method, reinforcements are synthesised and modified externally before adding them into the liquid metal. The composites manufactured by ex-situ method always having poor

thermodynamic stability between matrix and reinforcement. They are also having poor high temperature properties. Several defects such as agglomeration and uneven microstructure can be seen in ex-situ composites [1].

In in-situ process, the metal matrix composite is developed by adding the reinforcement directly to the molten matrix material. The reinforcement is developed within the matrix by adding various ceramic particles such as oxides, carbides, nitrides or even halide salts to the molten matrix material. The reinforcement is developed by the exothermic reaction between the elements or their compounds with molten metal matrix. In in-situ method, reinforcements are synthesised and modified internally in the molten matrix metal where they transform to another thermodynamically stable phase. Such induced phases are more coherent to the matrix which improves the properties of the composite by principle of combined action. In-situ generated particles also have fine size, uniform distribution and improved interfacial bonding [1, 2].

In-situ technique is suitable to produce new composite materials that are not obtained by conventional methods. The in-situ process is having several advantages like uniform distribution of reinforcement, development of fine reinforcement particles and fine interface between the matrix and reinforcement. Synthesis of MMCs by casting is the economical processing route to develop in-situ metal matrix composites with the uniform distribution of the reinforcement.

Many researchers have put their efforts in past four decades to make in-situ aluminium matrix composites using various reinforcements such as particulate reinforcements like  $\text{MnO}_2$  [2],  $\text{Al}_2\text{O}_3$  [3-10],  $\text{SiC}$  [9, 11-13],  $\text{SiO}_2$  [7, 14],  $\text{ZrO}_2$  [8, 15], graphite [16-21],  $\text{B}_4\text{C}$  [22], Mica [23],  $\text{TiB}_2$  [24],  $\text{MoS}_2$  [25],  $\text{TiO}_2$  [26],  $\text{TiC}$  [27, 28],  $\text{Al}_3\text{Zr}$  [28],  $\text{Fe}_3\text{O}_4$  [30],  $\text{Fe}_2\text{O}_3$  [31],  $\text{ZnO}$  [32], , carbon and boron fiber [33], rice hulk [34], etc. Higher mechanical properties can be achieved in all above cases as compared to the base alloy.

## **Problem Formulation**

Metal matrix composite materials are known for their superior properties compared to the conventional metals and their alloys. Improved microstructures and mechanical properties of the composite materials are attributed to the type of matrix and reinforcement materials. Various chemical reactions between matrix and reinforcement at processing temperature generate new phases.

In present work, commercially pure aluminium (LM 0 grade) was used as the matrix material. Aluminium metal widely used for varieties of applications because of its -

- Low density ( $2.67 \text{ gm/cm}^3$ ),
- Ease of manufacturing due to low melting point and high ductility,
- High thermal and electrical conductivity,
- Excellent corrosion resistance,
- Low cost, etc.

MnO<sub>2</sub> powder was used as reinforcing constituent. MnO<sub>2</sub> is economical, easily available and strengthen the soft matrix likes aluminium. MnO<sub>2</sub> is generally immune to various chemical reactions and hence it requires the presence of wetting agents such as transition metals (Li, Mg, Ca, Ti, Zr and P). The purpose of these wetting agents is to improve the wetting characteristics of the immune (generally ceramic) reinforcement particles by reducing the surface tension [8].

Magnesium metal was used in current study as wetting agent for MnO<sub>2</sub> reinforcement. It does not only improve the wetting property but it also enhances the strength of the system by solid solution strengthening. Magnesium metal reacts with the aluminium matrix and reinforcement particles at the processing temperature and generates various in-situ phases. These in-situ phases are so small that they improve the mechanical properties such as strength and hardness. The strength can be increased due to dispersion strengthening effect. Magnesium metal is light in weight compare to the aluminium. Also, it is highly reactive with oxygen at high temperature.

**Table 1:** Parameters and properties studied

Sr. No.		Description
1	Parameters	Amount of reinforcement, Sequence of reinforcement addition,
2	Properties	Density, Ductility, Hardness, Tensile strength, Chemical analysis (spectroscopy) XRD and Microstructure.

There are many methods to generate in-situ composite materials. Most economical and less time consuming mass production method is the stir casting. Hence in present work, stir casting method was used to produce in-situ aluminium metal matrix composite (AMMC) materials. Four

blade stirrer was used in this work with fixed rotational speed. In stir casting method, due to continuous stirring effect, dispersion of generated in-situ phases is good which ultimately reduces the segregation problem. The microstructure and mechanical properties can be improved.

In this technique, attention is needed to avoid gas entrapment while addition of oxides and wetting agents. To decrease the chances of the porosity in the resultant in-situ composite, proper melt treatment has to be carried out before pouring of molten composite bath. Different parameters and properties studied are listed in following table 1.

## Objectives of the work

Followings are the objectives of the research work:

1. To optimise the amount of wetting agent for commercially pure aluminium,
2. To study the variation of reinforcement and its sequence of addition,
3. To study generated in-situ phases after optimised the wetting agent and reinforcement oxides,
4. To study the microstructure and mechanical properties of generated in-situ composite material and
5. To compare the properties of existing material with new in-situ route.

## Scope of the work

Making in-situ aluminium matrix composite (AMMC) materials requires extreme care. As mentioned above, many researchers have studied in-situ composite materials through various methods. During the review of the literatures, it was noted that most of all researchers have followed melting and casting route in which the reinforcement materials have been added after the melting of matrix system. In this sequence of addition, different defects were observed in resultant composite materials such as segregation and porosity. Some researchers have tried to use different sequence of addition (in Al-SiO<sub>2</sub> system by Rohatgi et. al. [7] and in Al-MnO<sub>2</sub> by Hamid et. al. [2]).

If reinforcement particles are heated at high temperature, it may pick the gases from an environment. It may generate poor interface between matrix and reinforcements while actual

processing. Thus addition of wetting agent is very important to change the surface chemistry of the reinforcement particles.

To understand the same problem and to increase the wettability of the particles TWO addition sequences of oxide were studied. The details of sequences are -

1. **Sequence A:** MnO<sub>2</sub> particles are added after the melting of the matrix metal (existing approach) and
2. **Sequence B:** MnO<sub>2</sub> particles are added before the melting of the matrix metal (new approach).

This comparative study between sequence A and sequence B revealed many interesting facts. Characterisation part of the research work shows the improvements in the properties by adopting new sequence compared to the existing sequence of the addition.

In industries, there is always requirement of the materials which are having improved properties. In this work, matrix metal was commercially pure aluminium (LM 0 grade) and reinforcement particles were MnO<sub>2</sub> powder which are not highly expensive. The manufacturing method adopted was the stir casting method which is again simple and economical method for mass production. This research work is not particular application oriented study but generated in-situ composite materials shows enhanced properties which can help to replace some existing materials.

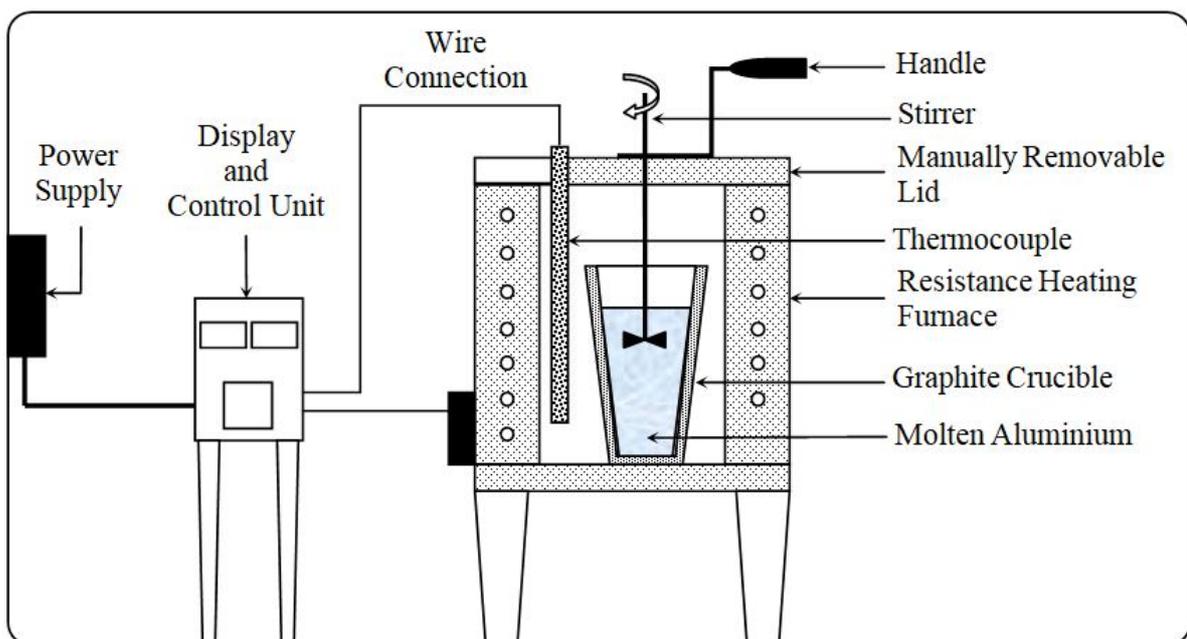
## Thesis Layout

This thesis is containing 07 chapters. The description of each chapter is given below:

1. **Chapter 1:** This chapter is describing about background of the work, problem evaluation and novelty, research gap, scope and objective of the work, importance research findings and research methodology used.
2. **Chapter 2:** This chapter contains the detailed literature review starting from the history of the composite materials, classification and manufacturing of composites, solidification behaviours, aluminium matrix composite materials, ex-situ and in-situ composites and applications of the composites in various fields. This chapter also contains the detailed review of relevant research articles which were published in various books and journals.

3. **Chapter 3:** In this chapter, detailed research methodology (procedure) is explained using simple flow charts. This chapter includes experimental set up details, various difficulties faced during melting and different characterisation methods.
4. **Chapter 4:** Results and related discussion was explained in this chapter. It starts from the raw materials analysis to final in-situ composite analysis (phase wise) with evidence in form of the tables, graphs and photographs.
5. **Chapter 5:** This is concluding chapter of the research work showing research output in brief.
6. **Chapter 6:** In this chapter, few points were discussed which remained untouched during this work. Also this chapter shows important information related to future scope of the work.
7. **Chapter 7:** This chapter shows the publications. Total 03 publications from each phase study were presented in this chapter. All these three publications are of international standard from which 02 are published in peer reviews SCOPUS journal whereas 01 paper was presented in international conference and published in its proceeding.

## Experimental set up



**Figure 1:** Schematic diagram of the experimental set up used in present study

The aluminium melting furnace used in this work is consists of the followings:

**1. Main melting zone:**

It is essentially square cross sectioned zone consisting outer mild steel wall and inner stainless steel wall having hole at the bottom side. In between outer and inner wall, heating coils are arranged as to provide resistance heating for the melting. Heating coils are made up of Kanthal wire.

**2. Thermocouple:**

It is Chromel-Alumel type thermocouple inserted into the melting zone to predict the temperature of the melt by displaying on to the temperature display panel in the degree Celsius unit.

**3. Temperature controller:**

It is connected to the heating coil as well as to the thermocouple. The automatic rely regulates the temperature to the preset limit and helps to maintain optimum temperature.

**4. Stirrer:**

It is typical turbine type four blade stainless steel stirrer which was used during stirring

**5. Speed and temperature controller:**

It is essentially used to control the speed of stirrer and the temperature of the bath via controlling the speed of motor. It is also used to indicate the actual speed of stirrer in working condition. This can be achieved by putting sensor near the bottom of the motor. Speed controller display shows the speed in the form of rpm and temperature of the bath.

**6. Metallic die:**

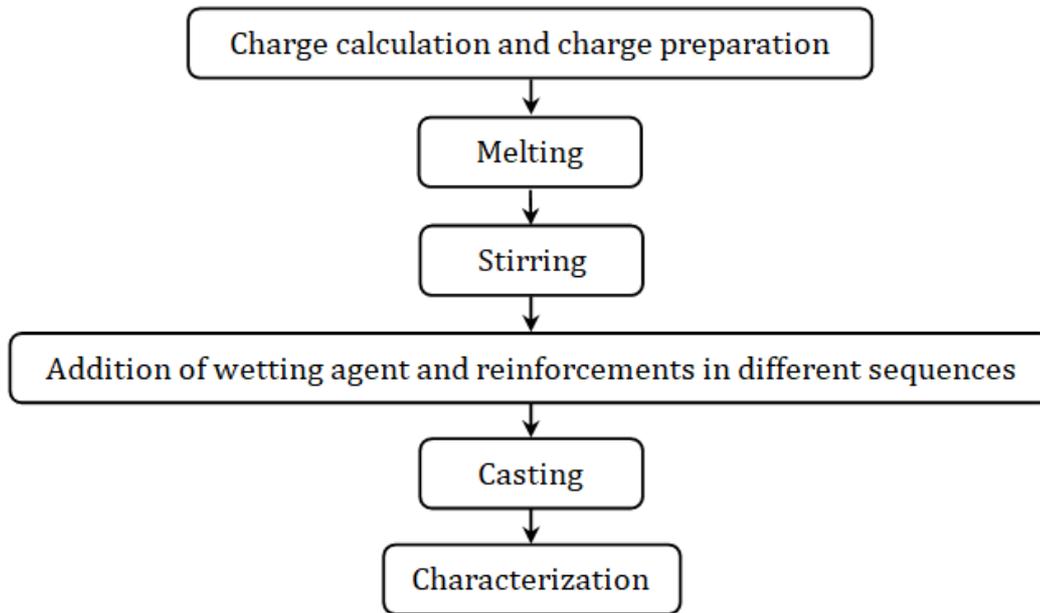
It is a metallic split die having 5 rod shape cavities in which liquid composite bath was poured and solidified

**7. Auxiliary Equipments:**

- |                                      |                                |
|--------------------------------------|--------------------------------|
| (a) <i>Al rod</i>                    | (f) <i>Spoon type rod</i>      |
| (b) <i>Toggle</i>                    | (g) <i>Glows</i>               |
| (c) <i>Strobometer or Techometer</i> | (h) <i>Oxy acetylene flame</i> |
| (d) <i>Muffle furnace or oven</i>    | (i) <i>Hammer</i>              |
| (e) <i>Drossing basket</i>           | (j) <i>Hacksaw blade</i>       |

## ✚ Research Methodology

In present work, melting and casting route was followed for fabrication of aluminium matrix composite with MnO<sub>2</sub> reinforcement. Following is the summarised flow chart of the experimental work presented in figure 2.



**Figure 2:** Generalised flow sheet of present work methodology.

Detailed methodology of each phase experimentation is explained in chapter 3 along with comparison between the existing addition sequence A with newly adopted addition sequence B.

## ✚ Research Findings

The main aim of present research work is to improve the properties of commercially pure aluminium (CPA) by varying different parameters like amount of particulate reinforcement and its sequence of addition.

Selected reinforcement is MnO<sub>2</sub> because it is stable up to the temperature at around 500°C, above which decomposition take place [35] at 535°C and generates oxide particles. Optimization of magnesium was also checked for proper wetting of generated oxides or any other phases. The process parameters are reported in table 2. To check the effects of additions, detail analysis has been carried out in three different phases of experiments:

1. **Phase I:** Optimization of magnesium content into CPA
2. **Phase II:** Effect of variation of MnO<sub>2</sub> content by changing its addition sequence into CPA and

3. **Phase III:** Effect of variation of MnO<sub>2</sub> by changing its addition sequence along with optimised magnesium metal from phase I into CPA.

**Table 2:** Process Parameters

Sr. No.	Process Parameter	Process Variables		
		Phase I	Phase II	Phase III
1	Amount of Commercially Pure Al	1 kg	1 kg	1 kg
2	Processing temperature	720 °C	720 °C	720 °C
3	Time of processing	15 min	15 min	15 min
4	Stirrer speed	100 RPM	100 RPM	100 RPM
5	Amount of Commercially Pure Mg	0.05, 0.15, 0.5, 1, 1.5, 2, 3, 4, 5, 6 and 7*	--	3*
6	Amount of MnO <sub>2</sub>	--	0.5, 1, 1.5, 2, 2.5, 3, 3.5 and 4*	1, 2.5 and 4*
7	Sequence of MnO <sub>2</sub> addition	--	A and B <sup>#</sup>	A and B <sup>#</sup>

\*All compositions are in weight percent unless stated otherwise

<sup>#</sup>Sequence A: MnO<sub>2</sub> added after melting of commercially pure aluminium (conventional route) and Sequence B: MnO<sub>2</sub> added before melting of commercially pure aluminium (non-conventional route)

Phase I analysis – in Al–Mg system, magnesium metal was optimized with commercially pure aluminium (CPA) system. It was found that 3 wt % Mg was giving superior mechanical properties.

In phase II, analysis of Al-MnO<sub>2</sub> system, optimisation of MnO<sub>2</sub> powder was carried out with CPA. In this phase, both sequences A and B were followed to check its effect on final composite properties. It was noted that sequence B (MnO<sub>2</sub> powder added before the melting of matrix metal) and 2.5 wt % MnO<sub>2</sub> was giving best results compared to the sequence A (MnO<sub>2</sub> powder added after the melting of matrix metal).

Finally in phase III, with optimised magnesium metal plus the variations of MnO<sub>2</sub> by changing its addition sequences were analysed. It was found that the Al – 3 wt % Mg – 2.5 wt % MnO<sub>2</sub> system showed best results. In this study again sequence B was found the best compared to

sequence A. It was found that the in-situ phases which are generated in the matrix due to various chemical reactions are more favourable when we follow the sequence B.

After the processing of the aluminium matrix in-situ composites, various characterizations have been performed to investigate mechanical properties. Various mechanical properties such as ductility, density, hardness and strength were analysed as shown in table 1. Short summary of each phase such as variations in reinforcement additions, chemical analysis, mechanical property characterization, XRD analysis and microstructural evaluation are also discussed as following.

### **Phase I: Effect of weight percentage of magnesium in CPA**

In phase I experiments, the amount of Mg was optimised through various trials. The amount of magnesium was varied from 0.05 to 7 wt % by keeping all other parameters constant. Phase I study involved the addition of commercially pure magnesium into commercially pure aluminium. As shown in table 2 of process parameters, **11 runs** of experiments were performed to check the effect of magnesium in CPA. One run of only CPA without magnesium addition with similar processing conditions was also taken for comparison purpose. These experiments were performed to determine the optimum level of magnesium for present CPA which used as matrix material. 700 gm of CPA, 720°C processing temperature and 15 minutes processing time were kept same for all the experiments. Magnesium amount was varied as 0.05, 0.15, 0.5, 1, 1.5, 2, 3, 4, 5, 6 and 7 wt %.

As the weight percentage of magnesium was increased, the recovery was also increased. At the same time, amount of oxygen was reduced. The amount of trace elements like Si, Mn and Fe were found minimum. Due to the presence of different elements, the presence of various phases was confirmed. Magnesium is strengthening element which forms solid solution strengthening into the CPA matrix. Hence it has improved various mechanical properties. X-ray diffraction (XRD) analysis of Al-3 wt % Mg system was carried out as it was showing maximum values of properties especially the strength. Presence of  $MgSiO_3$ ,  $Al_2Mg_3$ , Mg-Si-O,  $Al_2O_3$  and free Mg phases were confirmed. These in-situ phases were generated into the CPA matrix at processing temperature. Due to the presence of these multi in-situ phases, different mechanical properties were increased.

In present study of phase I, the density of the system was found decreasing throughout the variations of magnesium addition in CPA due to increasing the concentration of Mg which is having lower density ( $1.74 \text{ gm/ cm}^3$ ) compared to that of the aluminium ( $2.7 \text{ gm/ cm}^3$ ). Ductility

of overall system was also decreased as concentration of magnesium increased which was due to the formation of brittle phases such as oxides and carbides into the Al-Mg system as stated in microstructural analysis. Such phases are although light in weight but brittle in nature. Upon plastic deformation of the sample, such phases break into fine angular particles and act as stress raiser in the matrix. Hence as compared to the base metal, the ductility was not improved. Hardness (BHN) was found increasing starting from the initial experiments upto 3 wt % Mg. After 3 wt % Mg, hardness has continued to increase but the strength has dropped. The ultimate tensile strength (UTS) was found increasing initially and attained maximum value at 3 wt % of magnesium concentration. At 3 wt % Mg, highest UTS was achieved almost 217 MPa as compared to 115 MPa in base metal CPA. The UTS value was decreased to 167 MPa in 7 wt % of magnesium addition. Hence specific strength, i.e., strength to density ratio also increased from 42 kN-m/kg of base metal to 81 kN-m/kg at 3 wt % Mg and then decreased to 64 kN-m/kg at 7 wt % of magnesium concentration. After 4 wt % of magnesium, the slope of the graph became almost steady in case of tensile strength. Due to the formation of various intermetallics phases into the CPA matrix, strength was found increasing as compared to the base metal. As solubility of magnesium into aluminium decreases below 100°C, the solid solution strengthening effect can be observed maximum at 3 wt % of magnesium as compared to different experiments in this Al-Mg system.

Microstructural observation (both optical and scanning electron microscopy (SEM)) revealed that by increasing of magnesium amount, unreacted magnesium has observed at the grain boundaries along with other intermetallic phases. This leads to change in the morphology of the grain structure. Due to the formation of various solidification fronts, engulfment and pushing effects are responsible for the gathering of these micro particles at the grain boundaries. Such engulfment and pushing effect was found nominal up to 3 wt % of Mg and increased significantly after 3 wt % of Mg. Heavy cluster of intermetallic phases were observed in 4 wt %, 5 wt %, 6 wt % and 7 wt % of Mg. Unreacted magnesium also increased due to the limited solubility of magnesium metal in aluminium. Below 100°C, the solubility of magnesium reduces and becomes less than 3 wt %. But above 300°C, even 7 wt % Mg can be dissolved in aluminium melt easily as per the equilibrium phase diagram of Al-Mg. In this work, 3 wt % Mg can help to improve the wettability of the resulting in-situ intermetallic phases. Microstructures of the Al-Mg system show flawless grain structures. The presence of intermetallics was confirmed by XRD analysis. Microstructure of 3 wt % Mg is more uniform and equiaxed as compare to other microstructures. As the amount of magnesium increased, free magnesium was found in the structure as dark black phase.

**Phase II: Effect of variation of MnO<sub>2</sub> by changing its addition sequence in CPA**

In phase II, amount of reinforcing phase (MnO<sub>2</sub>) was optimised without addition of magnesium as wetting agent. Phase II was conducted in two different approaches. In first approach named sequence A, MnO<sub>2</sub> reinforcing phase was added **after** melting of aluminium as conventionally practiced. In second approach which is named sequence B, the reinforcing phase MnO<sub>2</sub> was added **before** the melting of the aluminium, i.e. MnO<sub>2</sub> powder was mixed with the charging materials with solid CPA. During sequence B, due to gradual heating of MnO<sub>2</sub> particles and more time involvement in various reactions, it increased the recovery Mn from MnO<sub>2</sub>. In this study, the addition of MnO<sub>2</sub> into commercially pure aluminium (CPA) was carried out without addition of commercially pure magnesium. As shown in table 2 of process parameters, there were **16 runs** of experiments carried out to check the effect of MnO<sub>2</sub> by changing the sequence of addition. In both the sequences, fixed amount of MnO<sub>2</sub> was varied like 0.5, 1, 1.5, 2, 2.5, 3, 3.5 and 4 wt %.

Recovery of manganese was found highest in 2.5 wt % MnO<sub>2</sub> of sequence B. It has been observed that Fe/Si ratio was found lowest in 2.5 wt % MnO<sub>2</sub> of sequence B. The X-Ray diffraction (XRD) analysis of 2.5 wt % MnO<sub>2</sub> was carried out for both sequences A and B experiments. Typical X-Ray pattern indicates the presence of oxides and complex carbides such as FeSiO<sub>3</sub>, MnAl<sub>2</sub>O<sub>3</sub>, Mn<sub>3</sub>AlC and Al<sub>2</sub>O<sub>3</sub> into the CPA matrix. Raw MnO<sub>2</sub> contained the carbon and hence in resultant in-situ composite presence of complex carbides were seen. MnO<sub>2</sub> addition was very difficult beyond 2.5 wt %. The density of MnO<sub>2</sub> powder is very less as compared to CPA, we found floating of these powder on CPA liquid bath. The rejection of MnO<sub>2</sub> powder by the liquid bath was increased beyond 2.5 wt % level. This was the reason for choosing only 2.5 wt % MnO<sub>2</sub> sample for XRD analysis for sequence B.

The density results were dropping from the initial value throughout the addition of MnO<sub>2</sub> in sequence A and B. Generation of light weight phases such as MnO and (Mn, Mg)<sub>2</sub>SiO<sub>4</sub> are responsible for decreasing the density of the system. The ductility values were remained almost same as no marginal changes were observed in the sequence A whereas it was found increasing in the sequence B. Hardness (BHN) distribution was remained almost unchanged in sequence A. But in sequence B, hardness value was increased as amount of MnO<sub>2</sub> added. The slight reduction in ultimate tensile strength (UTS) values were observed in sequence A while in sequence B, it was increased as the amount of MnO<sub>2</sub> was increased. Although the highest hardness values were observed in 2.5 wt % MnO<sub>2</sub> combination in the both sequences A and B whereas the highest

UTS was found in sequence A at 2.5 wt % MnO<sub>2</sub> compositions and at 2 wt % MnO<sub>2</sub> in sequence B.

Compare to sequence A, sequence B microstructures show more uniformity as far as morphologies of grains, grain boundaries and other resultant phases were concerned. By comparing both sequence results, the addition of MnO<sub>2</sub> as in sequence A showed more bulky grain boundaries and segregated phases whereas in sequence B, thin grain boundaries were observed. Also, the resultant phases were well distributed in sequence B.

**Phase III: Effect of variation in MnO<sub>2</sub> by changing its addition sequence along with the optimised magnesium from phase I study**

In phase III, combined effects of reinforcing phase MnO<sub>2</sub> along with wetting agent magnesium were studied. In this phase amount of magnesium was kept fixed at 3 wt. % which was optimised from phase I results while amount of MnO<sub>2</sub> was varied like 1, 2.5 and 4 wt. % for both sequences of addition. Addition of MnO<sub>2</sub> reinforcing phase was kept limited upto 4 wt. % because beyond this limit, it was observed that the liquid CPA rejected the excess powder of MnO<sub>2</sub>. These experiments were carried out in two different approaches as indicated in phase II. In first approach (sequence A), MnO<sub>2</sub> added after melting of commercially pure aluminium whereas in second approach (sequence B), reinforcing phase was added before the melting of CPA. The results of both approaches were analysed in the next chapter. Formations of in-situ phases were confirmed which are improving various properties. Phase III study involved the addition of magnesium and MnO<sub>2</sub> both into commercially pure aluminium in two different sequences (A and B as mentioned in table 2). There were **total 06** runs of experiments, three in each sequence, have performed to check the effect of both magnesium and MnO<sub>2</sub> by changing its sequence of addition on various properties on CPA matrix. From phase I results, optimum value of magnesium in commercially pure aluminium was 3 wt %. Hence, keeping this result in mind, the magnesium content at 3 wt % was fixed, whereas MnO<sub>2</sub> concentration was varied as 0.5, 2.5 and 4 wt % in two different sequences (A and B).

Quantitative and qualitative analysis of various elements like Mg, Si, Mn and Fe recovered were performed. It is observed that Si and Fe are presence in raw materials. It was found that magnesium and manganese recoveries were higher in sequence B as compared to sequence A. Spectroscopy testing was performed for recovery of results. The presence of the in-situ complex carbide compounds such as Mg-Fe-SiO<sub>3</sub>, MnAl<sub>2</sub>O<sub>3</sub>, Mg-Si-O, Al<sub>2</sub>O<sub>3</sub>, Mn<sub>3</sub>AlC and Mg were

confirmed in the XRD patterns. It was noted that the peak height of  $Mn_3AlC$  in 2.5 wt %  $MnO_2$  of sequence B experiments are higher as compared to all the combinations of sequence A experiments. It could be the main reason to have improved mechanical properties in this system following sequence B.

Presence of heavier and lighter phases in the matrix affects the level of density values of resultant composite material. In sequence B, lowest density was observed due to formation of light  $Al_2Mg_3$  (molecular weight is 126.88 gm/mole) in-situ phase. In sequence A, formation of  $Al_3Mg_2$  were observed which is comparatively heavier (molecular weight is 129.56 gm/ mole) as indicated in XRD analysis. Beyond 2.5 wt %  $MnO_2$  addition, density values were increased in both the sequences A and B. Ductility values are found decreasing as the amount of  $MnO_2$  addition increased. This was due to formation of hard phases in the matrix. Lowest reading of the ductility was noted in sequence A experiments. Overall both ductility and density were marginally lowered in both sequences A and B as compared to the base matrix material. In both A and B sequences, similar pattern of incremental hardness (BHN) were reported upto 2.5 wt %  $MnO_2$  addition. Afterwards, hardness values were reduced. Maximum value of hardness property was reported in sequence B at 2.5 wt %  $MnO_2$ . It can be seen that in sequence A, the ultimate tensile strength (UTS) results were decreasing and found minimum at 2.5 wt %  $MnO_2$  while in sequence B, the results were marginally higher and found maximum at 2.5 wt %  $MnO_2$ . Both hardness and tensile strength results in sequence B were found higher mainly due to in-situ formation of  $MnAl_6$  along with cemented carbide  $Mn_3AlC$ . Formation of such phases is due to higher interaction time between  $MnO_2$  and aluminium as it was added before melting of CPA. It means in sequence B, during entire melting procedure from room temperature to processing temperature,  $MnO_2$  powder had more time to interact with CPA, from mushy stage to liquid stage. The presence of fine dispersoids in the soft aluminium matrix, acts as the barriers in the motion of dislocations. Thus it results into the dispersion strengthening which can be easily justified using classical Orowan theory. This phenomenon increases the flow stress of the entire composite system. The strengthening is generally found maximum where fine dispersoids are well distributed in the matrix.

Microstructure observations indicate the fineness of the intermetallics which were formed during processing. Precipitation and segregation of various Al-Mn intermetallics and carbide compounds were observed in the microstructures. Comparatively, in sequence B the intermetallics observed were well distributed throughout the matrix. In 1 wt % and 4 wt %  $MnO_2$ , more dense segregation was observed than 2.5 wt %  $MnO_2$  systems. In the

microstructure, highest refinement was observed in Al-3 wt % Mg-2.5 wt % MnO<sub>2</sub> system in sequence B. More phase formation was appeared in Al-3 wt % Mg-1 wt % MnO<sub>2</sub>. Also the distribution of Al-Mn complex carbide was found uniform.

## Conclusion

The following final conclusions can be made from all above phases are:

1. From phase I study, Al-3 wt % Mg system was giving highest values of mechanical properties for present commercially pure Al metal.
2. From phase II study, the sequence B for MnO<sub>2</sub> addition is found more promising, i.e. when MnO<sub>2</sub> added in commercially pure aluminium before its melting by considering the changes in mechanical properties. The result of mechanical properties graph were steeper in sequence B compared to sequence A. In sequence B, the microstructures were much more refined and average value of Mn recovery is improved.
3. From phase III study, by keeping magnesium amount fixed at 3 wt % as optimized in phase I and after changing addition sequence the best sequence is B. In sequence B, the micromechanical results were more favourable then sequence A. Average recovered magnesium and manganese both were higher in sequence B as compared to A. It helps to promote the formation various in-situ phases as indicated in XRD analysis which ultimately strengthen the matrix.

Using above approach, the manufacturing of light weight - AMMCs can be promoted to achieve good micromechanical properties at lower cost as compared to the conventional materials and method of manufacturing.

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