

PRODUCTION OF ALUMINIUM MATRIX IN - SITU PARTICULATE COMPOSITES

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Abstract

Aluminium-alumina (Al_2O_3) and Al-spinel (MgAl_2O_4), MgO in-situ particulate composites were prepared by reacting several oxides such as CuO, MnO_2 , Fe_2O_3 and TiO_2 particles with molten pure Al and Al-2 Mg alloy respectively. 2-5 wt% of the oxides were incorporated into pure Al and Al-2 Mg alloy melt by vortex method. After addition of the particles into the melt, the liquid composites were solidified in crucibles. To identify the phases formed in the composites, parts of the composites were remelted and treated with 0.3 wt% hexachloroethane. The particles were analysed by X-ray diffractometry. Al_2O_3 particles were detected in pure Al base composites and MgAl_2O_4 and MgO particles were detected in Al-2 Mg alloy base composites. Most of the particles formed in-situ were in the size range 1-3 μm . The present work shows the possibility of producing Al matrix in-situ particle composites reinforced with Al_2O_3 , MgAl_2O_4 and MgO.

Introduction

Aluminium matrix ceramic particle composites are materials of recent interest due to their superior properties such as specific modulus, tensile strength at room temperature and elevated temperatures and wear resistance compared to the conventional alloys [1-8]. Generally the reinforcing ceramic particles are directly incorporated into liquid Al to prepare the composites by casting route. However, the problems encountered in this approach are the nonwettability of the particles by molten Al, segregation of the particles in the matrix and excessive interfacial reactions that causes degradation of the mechanical properties [9]. It has been observed that the interface of the reinforcement and the matrix is susceptible to crack initiation and propagation during mechanical deformation. This could be due to nonwetting or extensive reaction at the particle-matrix interface [4,10,11]. One approach to control the interfacial reaction is the variable codeposition of multiphase materials (VCM) process [9,12,13], where reinforcing particles are incorporated into a stream of atomized liquid metal. Liquid metal infiltration into fibre or particle preforms can also control the interfacial reaction [14], if the time for infiltration is minimized. Another approach to minimize the interfacial reaction is the preparation of in-situ composites. In this approach, the

reinforcing phases are formed within the matrix by some chemical reaction between the matrix and some externally added compounds. The advantages expected in such composites are better compatibility of the phases formed in-situ with the matrix, cleanliness of the interface and the possibility of producing reinforcing phases with varied morphology [15]. Thus TiB_2 [16] and TiC [17] particles have been formed in Al matrix by reactions between Al and some other externally added solid or gaseous elements / compounds.

The objective of the present work is to prepare Al- Al_2O_3 and Al- MgAl_2O_4 in-situ particle composites by addition of CuO , MnO_2 , Fe_2O_3 , Cr_2O_3 , TiO_2 , V_2O_5 and ZnO particles into pure Al and Al-2 Mg alloy melts respectively. Al - Al_2O_3 particle composites have been produced and characterised in many studies [4,5] by external addition of Al_2O_3 particles in Al matrix. MgAl_2O_4 particle reinforcement was aimed to produce in Al matrix, since these particles are hard [18] and oxidation of Al-Mg alloy can produce MgAl_2O_4 [19]. Hence 2-5 wt% of these oxide particles were incorporated into Al/Al-2Mg alloy melts by vortex method and were allowed to react with the latter. To detect the phases formed by such reactions, x-ray diffraction study (XRD) and electron probe microanalysis (EPMA) were carried out. Differential thermal analysis (DTA) was carried out to find the dissolution of reduced elements in the Al matrix.

Experimental Procedure

Commercially pure Al and Mg were used to prepare the composites. The size of the CuO , MnO_2 , Fe_2O_3 , Cr_2O_3 , TiO_2 , V_2O_5 and ZnO particles (Analar grade) was in the range of 1-90 μm .

To prepare the composites, 400-500g of Al was melted in an electrical resistance furnace. In case of Al-2 Mg alloy based composites, required amount of Mg was plunged into liquid Al. The liquid metal was stirred with a graphite impeller revolving at 1200 rpm to form a vortex in it. Required amount of the oxide particles were slowly added into the vortex formed in Al/Al-2Mg alloy. After complete addition of the particles, the melts were stirred for another 2 min approximately and subsequently the melts were solidified in crucibles in still air. Specimens for DTA and EPMA were taken from the ingots solidified in crucibles. The DTA experiments were carried out in a Mettler 2000C analyser with $\alpha\text{-Al}_2\text{O}_3$ as a reference. The specimens for EPMA were polished mechanically by conventional method and etched with Keller's reagent. These were observed in a Jeol JCSA 733 analyser having an optical microscope attachment.

For x-ray diffraction study, parts of the composite ingots were remelted at 700°C and treated with 0.3 wt% hexachloroethane (C_2Cl_6) to extract the particles from the composites. While single treatment was sufficient to extract the particles from all the

composites based on pure Al, two or three treatments were required to extract the particles from Al-2 Mg alloy based composites. The x-ray diffraction experiments were carried out in a Phillips PW 1840 diffractometer using CuK_α radiation in the 2θ range 15° to 80° for the particles extracted from pure Al based composites and in a Siemens D500 diffractometer using Mo K_α radiation in the 2θ range 10° to 40° for the particles extracted from Al-2 Mg alloy based composites.

Results and Discussion

DTA STUDY

All the DTA curves of the composites showed endothermic peaks near 654°C for pure Al and other composites based on pure Al except Al-5CuO and Al-5ZnO composites. In these cases the endothermic peaks started at a lower temperature which is indicative of dissolution of Cu and Zn in Al matrix. However, the dissolution of Cu in Al matrix was considerable whereas that of Zn in Al matrix was much less. In Al-2 Mg alloy based composites, Cu was dissolved considerably in Al alloy matrix whereas it was negligible in the other composites.

X-RAY DIFFRACTION STUDY

The phases identified in the extracted particles from pure Al and Al-2Mg alloy based composites are presented in Table - I. In the composites based on pure Al, $\alpha\text{-Al}_2\text{O}_3$ particles formed with the addition of all the oxide particles except ZnO. Unreacted oxide particles and pure Cu, Fe and Cr were also detected in the extracted particles. The following reaction produced $\alpha\text{-Al}_2\text{O}_3$ particles in the Al matrix :



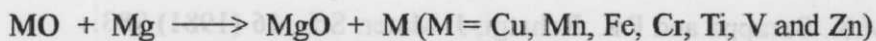
Cu formed by the reaction was partially dissolved in the matrix as confirmed by DTA study. Rest of the Cu was present in the matrix as pure Cu particles. EPMA with optical microscope attachment confirmed this observation. Similarly pure Fe and Cr particles were also confirmed by EPMA. In case of composites with MnO_2 addition, pure Mn or its intermetallic compounds could not be detected by XRD and EPMA. However Ti was present in the particles in the form of Al_3Ti and probably Al-Ti intermetallic phases. The size of the Al_2O_3 particles were 1 - 3 μm approximately and were rounded in shape. The reaction was found to be nearly complete with TiO_2 addition.

TABLE - I
Phases identified in extracted particles from pure Al and Al-2 Mg alloy based composites

Oxide particles added	Phases identified	
	Pure Al based composites	Al-2Mg alloy based composites
CuO	α -Al ₂ O ₃ , Cu, CuO (CuAl ₂)	MgAl ₂ O ₄ , Cu, (CuO, MgO)
MnO ₂ (MnAl ₂ O ₄)	α -Al ₂ O ₃ , Mn ₂ O ₃	MgAl ₂ O ₄ , (MgO, Mn,
Fe ₂ O ₃	α -Al ₂ O ₃ , Fe ₂ O ₃ , Fe	MgAl ₂ O ₄ , MgO, Fe, (Fe ₂ O ₃)
Cr ₂ O ₃	α -Al ₂ O ₃ , Cr ₂ O ₃ , Cr	MgAl ₂ O ₄ , MgO, Cr, (Cr ₂ O ₃)
TiO ₂	α -Al ₂ O ₃ , Al ₃ Ti, (TiO ₂)	MgAl ₂ O ₄ , MgO, Ti, (TiO ₂)
V ₂ O ₅	α -Al ₂ O ₃ , V ₂ O ₅ , (AlV ₂ O ₄)	MgAl ₂ O ₄ , MgO, AlV ₂ O ₄
ZnO	ZnO, (γ -Al ₂ O ₃)	MgAl ₂ O ₄ , MgO, (Zn, ZnO)

Note : The phases in parentheses were not confirmed as the corresponding peaks were either too small or the peaks deviated slightly from those of the standard values.

In the composites based on Al - 2 Mg alloy, MgAl₂O₄ and MgO were formed by the reaction between the matrix alloy and the oxide particles as shown in the following reactions.



Particles of reduced elements were also detected in the extracted particles from these composites. Cu was dissolved partially in the matrix as confirmed by DTA study. Pure Cu particles were observed in the matrix by EPMA with optical microscope attachment. The reaction between the Al-2 Mg alloy and the oxide particles were nearly complete in all the composites. MgAl₂O₄ particles as confirmed by EPMA were in the size range of 1-3 μ m approximately and rounded in shape.

Fine particles have been proved to be effective reinforcements in Al matrix, as similar volume fraction of finer particles increases the yield strength and tensile strength to a greater extent compared to coarser particles [5]. Generally, incorporation of finer particles into molten Al alloy is difficult due to clustering. The cluster of particles in a composite act as source of crack generation during deformation. It is interesting to note that the fine particles formed in Al matrix of the present work were not clustered. Another advantage of finer reinforcement is that coarser particles are generally more susceptible to cracking [20]. Hence the finer particles formed in the Al matrix of the present work are expected to be more effective reinforcements compared

to the conventionally produced composites where the particles are generally much coarser.

Conclusion

Al_2O_3 particles were formed in the composites based on pure Al containing CuO, MnO_2 and other oxide particles whereas MgAl_2O_4 and MgO particles had formed in the composites based on Al-2Mg alloy. Reactions were more complete in the composites based on Al-2 Mg alloy. The reinforcing particles are very fine (1-3 μm) and rounded in shape. The present work shows the possibility of producing Al_2O_3 , MgAl_2O_4 and MgO reinforced Al matrix in - situ particle composites.

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Introduction

The sloping nature of the solvus curve in the phase diagram of Al-Cr binary alloy system has led to considerable studies on the probable age-hardening behaviour in this alloy system [1-3]. It is reported earlier [2] that in Al-0.5 Cr alloy the precipitation of chromium aluminides takes place at the lattice defects and that there are two different types of precipitate morphologies seen in the alloy when aged at different temperatures. Chromium is known to have a tendency to form large sized intermetallics with aluminium, making the system quench sensitive [4,5]. It is also reported that trace additions and/or cold working before aging can have significant influence on the aging behaviour of a number of aluminium alloys [6]. Moreover the above alloy is known to possess a very high corrosion and oxidation resistance [2,3].

On the basis of available report on the effect of silicon in reducing the quench sensitivity of Al-Cr alloy it appears possible to develop Al-Cr-Si alloys with good age-hardening. In the present investigation, attempts are made to optimise the composition of the ternary Al-Cr-Si alloy in terms of its age-hardening response.