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# In situ fabrication and microstructure of $Al_2O_3$ particles reinforced aluminum matrix composites

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ABSTRACT

$Al_2O_{3p}$/Al composites were prepared by direct melt reaction process. The thermodynamics of in situ chemical reactions between molten aluminum and $CeO_2$ powder was studied. The XRD results show that the components of the as-prepared composites consist of $Al_2O_3$ and Al phases. For the as-cast composite specimens, SEM, EDX, TEM and SAD were used to analyze the reinforcement phases and interface characters of composites. The results show that the in situ generated $Al_2O_3$ particles, whose sizes are 100–200 nm, have various irregular shapes and disperse uniformly in matrix. TEM observation shows that the interface between particle and matrix is clean. Furthermore, there is no fixed orientation relationship between $Al_2O_3$ particles and aluminum matrix. Only $[1\bar{2}10]//[111]$ orientation parallel relationship with low exponent is found. Therefore, the composites have isotropic properties. Besides characters mentioned above, there are large amount of high density dislocations and the generated extensive fine subgrains around $Al_2O_3$ particles. These features are favorable for improving composite performances. As a result, the composites are comprehensively strengthened not only by $Al_2O_3$ particles, but also by the high density dislocations and fine subgrains.



## 1. Introduction

In recent years with the increasing demands for high-performance structural materials, particle reinforced aluminum matrix composites (PRAMCs) have attracted a lot of attention due to their excellent performances, such as light density, high specific stiffness, high specific strength and good thermal stability compared with pure aluminum and their alloys [1–4]. The PRAMCs can be fabricated by ex situ synthesis and in situ synthesis. In ex situ methods, the particles, which are prepared separately prior to the fabrication of the composites, are added into the metal. There are some defects and difficulties to fabricate composites by ex situ methods. For example, the particle size is difficult to be controlled at nanoscale. Furthermore, the distribution of fine particles in matrix is comparatively nonuniform. Another defect is that the interfacial reactions are likely to occur between the reinforcement and the matrix during fabricating process. These defects take a deadly effect on material properties [4,5]. In in situ process, the reinforcement phases are formed in metallic matrix through in situ chemical reaction between elements or between elements and compounds during composite fabrication process. There are many advantages for PRAMCs fabricated by in situ process. For example, the in situ formed reinforcement phases are thermodynamically stable, free of surface contamination and disperse more uniformly in matrix, leading to stronger particle–matrix bonding. At the same time, the in situ formed reinforcement phases have finer sizes. These outstanding features make in situ PRAMCs possess excellent mechanical properties and economical viability than their ex situ counterparts [6,7]. Now, a variety of processing techniques have been developed to fabricate PRAMCs by in situ process, such as direct melt reaction (DMR) process [1,3,7], reactive hot pressing (RHP) [8,9], and self-propagating high temperature synthesis (SHS) [10,11]. The DMR process is considered one of the most promising in situ synthesis techniques for commercial applications due to its simplicity, low cost and near net-shape forming capability [6].

Our previous studies on PRAMCs have shown that titanium boride ($TiB_2$) and alumina ($Al_2O_3$) can be formed in situ by means of DMR process. The $TiB_2$ or $Al_2O_3$ particles reinforced aluminum matrix exhibits high hardness, superior wear resistance, high melting point, good thermal stability, high stiffness and high strength at elevated temperature [1,7]. In this paper, Al–$CeO_2$($Ce_2(CO_3)_3$) components were selected to fabricate in situ $Al_2O_3$ particles reinforced aluminum matrix composites through DMR process. The microstructures of raw materials and composites, thermodynamics process of in situ chemical reaction and distinctive characteristics of interfaces and crystal structures are further studied. The unknown effects of in situ $Al_2O_3$ reinforcing particles in improving the creep

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and wear resistance of the Al-based alloys will be further reported later.

## 2. Experimental

The raw materials were pure aluminum ingots (99.97%Al) and cerium carbonate ($Ce_2(CO_3)_3{\cdot}nH_2O$). The cerium carbonate powder was preheated to dehydrate in electric drying oven at 250 °C for 3 h. During the heat process the cerium oxide generated due to the decomposition of cerium carbonate. Then the dried $CeO_2$ powder was cooled, grounded and screened. At the same time, aluminum ingot was melted in an electric furnace under argon atmosphere and held at 850 °C. Certain amount of dehydrated reactant powder was added and incorporated with mechanical stirring. The in situ reaction would take place instantly. Then the resultants of the chemical reaction, i.e. $Al_2O_3$, acted as the reinforcing particles in the matrix. The designed volume and weight fraction of reinforcing particles ($Al_2O_3$) were 4.5 vol.% and 3 wt.% respectively. After holding the reaction temperature at 850 °C for 30 min, the molten metal of PRAMCs was degassed and deslagged. Then the molten metal was cast into a copper mould.

Scanning Electron Microscope (SEM, JEOL-JXA-840A) and incidental Electronic Dispersive X-ray spectroscopy (EDX) were used to observe the morphology of raw materials, reinforcement phases and elementary composition and amounts in particles. The volume fraction was determined through Image II professional package. The high resolution Transmission Electronic Microscope (TEM, JEOL-JEM-2100) and Selected Area Diffraction (SAD) were combined to observe the high magnified images and analyze the kinds and orientation relationship between phases. The X-Ray Diffraction (XRD, Rigaku D/max2500) was employed to analyze the kinds of reinforcement phases.

## 3. Results and discussion

### 3.1. Thermodynamics of in situ reaction

Fig. 1a demonstrates the morphology of reactants preparing for reacting with molten aluminum. The morphology of reactants demonstrates as flat bars. The thickness is less than 5 μm. The utmost diameters are variable, the sizes of which are in the range of 50–100 μm. It can be clearly seen that there are some cracks or splits on its surface, which is favorable to react with aluminum melt. From the EDX result, as shown as Fig. 1b, the atom ratio of cerium and oxygen elements are almost 1:2. So the reactant is deduced as $CeO_2$. It is asserted that the $Ce_2(CO_3)_3$ decomposes as $CeO_2$ and $CO_2$ when it is preheated at 200–300 °C in the drying oven without any protective atmosphere, which is illustrated as formula (1):

$$Ce_2(CO_3)_3 \rightarrow CeO_2 + CO_2\uparrow \quad (200\text{–}300\,^{\circ}C) \tag{1}$$

The generated $CeO_2$ has high reactive activity. It will react with molten aluminum when they contact. XRD is utilized to determine the kinds of resultant of reaction. The result is shown as Fig. 2. It reveals that $Al_2O_3$ is the only resultant in the composite except aluminum matrix.

According to the XRD result the in situ chemical reaction between aluminum matrix and $CeO_2$ can be expressed as formula (2) [10]:

$$4Al + 3CeO_2 \rightarrow 2Al_2O_3 + 3Ce \quad (800\text{–}900\,^{\circ}C) \tag{2}$$

$$\Delta G^{\Theta}_{CeO_2} = -1029\,260 + 214.22T \quad (J/mol\,O_2) \tag{3}$$

$$\Delta G^{\Theta}_{Al_2O_3} = -1120\,480 + 214.22T \quad (J/mol\,O_2) \tag{4}$$

From formulae (3) and (4) it can be seen that the entropy values of $CeO_2$ and $Al_2O_3$ are nearly equal. So the reaction between aluminum and $CeO_2$ components has little relevance to the reac-

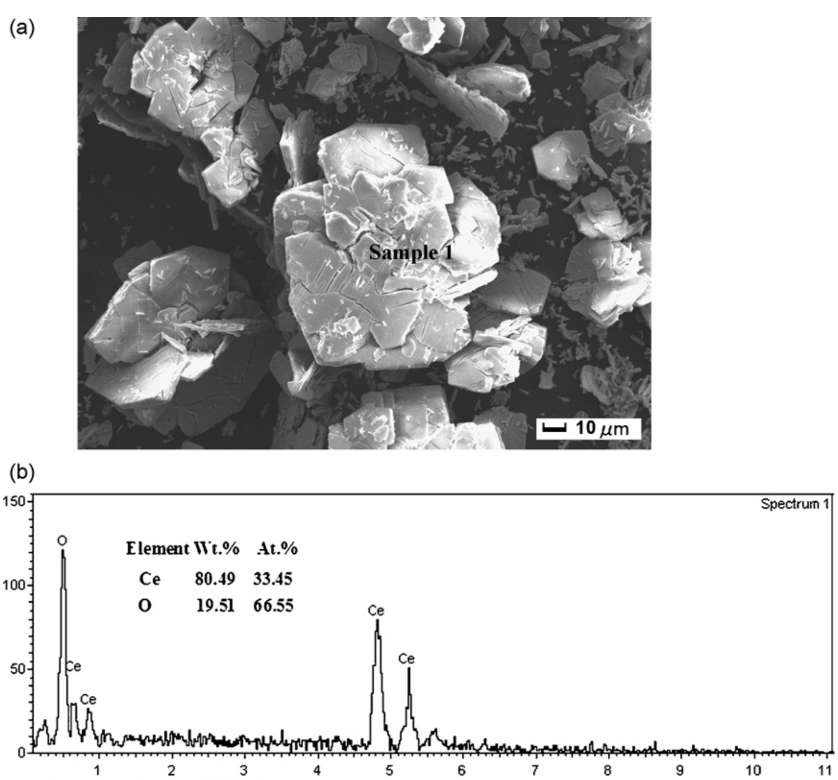


**Fig. 1.** Morphology and components analysis of raw materials. (a) Morphology of raw materials (500×) and (b) EDX result of spectrum 1.