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Materials

In-situ fabrication of particulate reinforced aluminum matrix composites under high-frequency pulsed electromagnetic field

Guirong Li, Yutao Zhao, Qixun Dai, Hongjie Zhang, and Hongming Wang

School of Materials Science and Engineering, Jiangsu University, Zhenjiang 212013, China

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Abstract: Pulsed magnetic field is generated when imposing pulse signal on high-frequency magnetic field. Distribution of the inner magnetic intensity in induction coils tends to be uniform. Furthermore oscillation and disturbance phenomena appear in the melt. *In-situ* Al_2O_3 and Al_3Zr particulate reinforced aluminum matrix composites have been synthesized by direct melt reaction using Al-$Zr(CO_3)_2$ components under a foreign field. The size of reinforced particulates is 2-3 μm. They are well distributed in the matrix. Thermodynamic and kinetic analysis show that high-frequency pulsed magnetic field accelerates heat and mass transfer processes and improves the kinetic condition of *in-situ* fabrication.

Key words: *in-situ* synthesis; aluminum matrix composites; pulsed magnetic field; thermodynamics; kinetics

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1. Introduction

Recently much attention has been paid to the development of effective fabrication processes for particulate reinforced metal matrix composites (PRMMCs) [1-2]. Among these, the *in-situ* formed particulate reinforced aluminum matrix composites have distinct advantages over the conventional ones, such as high specific tenacity and rigidity, clean interfaces, fine particulates, and excellent abradability [3-4]. Especially the direct melt reaction method (DMRM) is simple, with low cost, and a possibility of near net shape forming [5-6]. The particulate granularity and distribution status chiefly influence the mechanical properties and abradability of composites. Therefore, improving the fabrication process is a major method to optimize the microstructure of composites and enhance their comprehensive properties. In this study, under a high-frequency pulsed electromagnetic field, the *in-situ* Al_2O_3 and Al_3Zr particulate reinforced aluminum matrix composites have been synthesized using the direct melt reaction method in zirconium carbonate, with molten aluminum. The action mechanism of the foreign field was investigated. Moreover the dispersion behavior, patterns, and size of the *in-situ* formed reinforcements were also studied.

2. Experimental procedures

Raw materials selected were aluminum ingot (99.95%) and zirconium carbonate ($Zr(CO_3)_2 \cdot nH_2O$) powder (99.20%). Zirconium carbonate powder was preheated to dehydrate the bounded water in an electric oven at 523 K for 3 h. The dried $Zr(CO_3)_2$ powder was cooled, ground, and screened. At the same time, the aluminum ingot was molten in an electric furnace under argon atmosphere. Then the melt was poured into two similar graphite crucibles. One concluded the *in-situ* synthesis in the electric oven. The other was placed in a high-frequency pulsed electromagnetic field generator. The input power was 18 kW and frequency was 20 kHz in the induction coils. When the temperature of the melt reached 1143 K the dehydrated $Zr(CO_3)_2$ powder was added and pushed into the melt by a graphite bell followed by mechanical stirring to incorporate with the molten aluminum. During this process the *in-situ* Al_2O_3 and Al_3Zr particulates formed in the liquid aluminum. After 30 min, inspired gases were degassed and extra slags were removed by the refining slag. Subsequently the melt

Corresponding author: Guirong Li, **E-mail:** whmlgr@ujs.edu.cn

was cast into a permanent mould at 993 K. The composite was sampled and polished for microstructure observation with the help of JEOL-JXA-840 scanning electronic microscope (SEM) and Rigku D/max 2500 X-ray diffraction (XRD).

The inductive generative voltage E in the crucible was measured by alternating current mill voltmeter, using the small coil method when the input frequency was fixed [7]. The effective value of magnetic induction intensity B_e was calculated according to Eq. (1),

$$B_e = \frac{E}{4.44 fNS} \quad (1)$$

where f is the input frequency, Hz; N is the turn number of test coils; S is the cross-sectional area of test coils. When f=20 kHz, N=10 and S=4.75×10^{-5} m^2, the calculated B_e is 0.02-0.03 mT.

3. Results and discussion

3.1. Distribution of magnetic field

Fig. 1 is the comparative distribution diagrams of inductive generative voltages in high-frequency magnetic fields with and without pulse signal. Shown as Fig. 1(a), when there is no pulse signal the inductive generative voltages distribute symmetrically about the middle altitude location, by and large along the axial line. The approximate sequence is $E_{top}<E_{bottom}<E_{0.5\,h}<E_{0.25\,h}<E_{0.75\,h}$. When imposing pulse signal on a high-frequency magnetic field, shown as Fig. 1(b), the magnetic induction intensity is weakened as a whole compared to Fig. 1(a). Furthermore, the magnetic gradient is reduced at the lower part of induction coils. It is just the *in-situ* reaction area, where the magnetic distribution is comparatively stable.

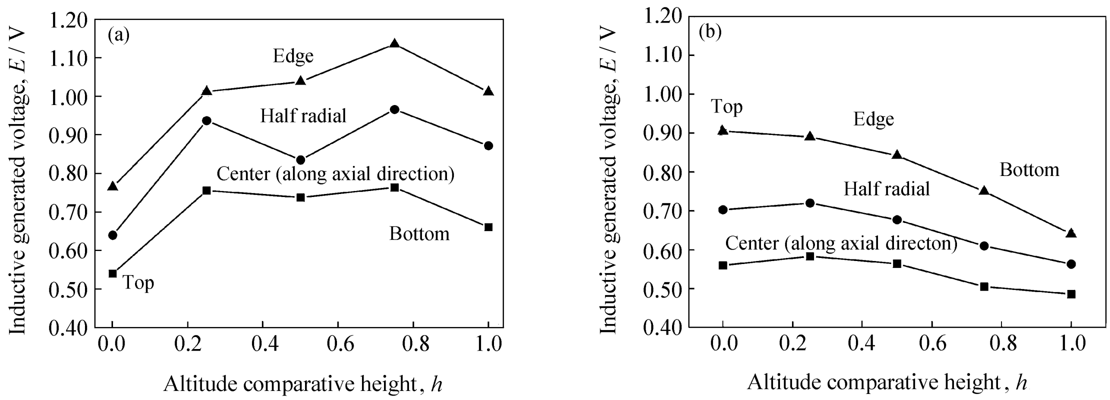

Fig. 1. Distribution diagrams of inductive generated voltages in high-frequency magnetic fields with and without pulse signal: (a) in high-frequency electromagnetic field; (b) in high-frequency pulsed magnetic field.

As is known, inductive current and voltage generated from the high-frequency alternative electromagnetic field distribute uniformly in the melt. Current density is the greatest on the surface of the charge material. It decreases gradually from the outer to the inner locations along the radical direction. When reaching a certain depth the current density is almost zero. The current density centralizes at the lateral superficial coats because of the Kelvin effect of inductive current [8]. But when impressed by the pulse signal, such as imposing low-frequency signal on high-frequency wave, it can increase the penetration depth of the generative current and reduce the distributional gradient of the magnetic intensity. The amplitude and frequency of the pulse signal loaded on the high-frequency magnetic field are 500 mV and 10 kHz respectively.

3.2. XRD and microstructure of composites

Fig. 2 is the XRD result of composites synthesized by Al-Zr$(CO_3)_2$ components. It shows that the endogenous particulates are Al_2O_3 and Al_3Zr. Their elastic moduli at 1363 K are 379 GPa and 308 GPa respectively. They are all elastic particulate reinforced phases. Fig. 3 shows the microstructure of $(Al_2O_3+Al_3Zr)_p$/Al composites synthesized under high-frequency magnetic field with and without imposing pulse signal. The generative amount and volume fractions of particulate are enhanced significantly. They are well distributed in the aluminum matrix. The size of the particulates is 2-3 μm. The round or elliptical phases are Al_2O_3 and the irregular polygon ones are Al_3Zr.

3.3. Thermodynamics and kinetics of in-situ reaction

The *in-situ* reaction processes of synthesizing $(Al_2O_3+Al_3Zr)_p$/Al composites by Al-Zr$(CO_3)_2$ components were investigated based on the classical thermodynamic and kinetic theories. First, ZrO_2 and CO_2

CO_2 were generated as a result of the decomposition of zirconium carbonate. Next the thermit reaction, that is, hyperthermia aluminum reduced ZrO_2:

$$3ZrO_{2(s)} + 13Al_{(l)} = 3Al_3Zr_{(s)} + 2Al_2O_{3(s)} \quad (2)$$

$\Delta G^{\ominus} = -1000065.4 + 756T$ $(J\cdot mol^{-1}\cdot K^{-1})$ [9].

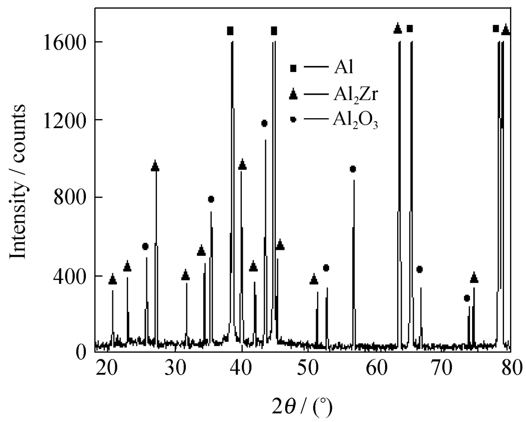

Fig. 2. XRD analysis of the particulate phases in Al-Zr(CO_3)$_2$ components.

The equilibrium temperature of the above reaction was 1322.8 K. When the temperature of the reaction system was lower than 1322.8 K that the thermit reaction would take place spontaneously. In fact the melt temperature was measured continuously with a handy thermo detector from reactants adding till the end of reaction. It was found that the temperature of the reaction system was lower than 1273 K during the whole *in-situ* reaction. Therefore, the *in-situ* reaction could proceed thoroughly. The entire reaction equation can be summarized as:

$$3Zr(CO_3)_2+13Al_{(l)}=6CO_2\uparrow +2Al_2O_3+3Al_3Zr \quad (3)$$

The thermit reducing reaction is the key step of the *in-situ* fabrication process, which is a multiphase solid-liquid reaction system. Only contact between reactants would result in the reaction. When there is no pulse signal, mass transfer and diffusion proceed depending on the concentration gradient in the melt. The transmission speed is slow. However, when imposing pulse signal with certain intensity on the high-frequency magnetic field, the pulsed magnetic field is generated. The magnetic induction intensity in the melt tends to be uniform, and the corresponding magnetic forces drive the melt flow. Actually agitation, stirring, and turbulence phenomena occur in the reaction system. Furthermore, carbon dioxide stirs the melt, which is the outcome of the decomposition of zirconium carbonate. The up and down vibrations of the liquid level could be observed during the synthesizing process. Diffusion and mass transfer of reactants and resultants are accelerated, which improve the kinetic conditions of fabrication.

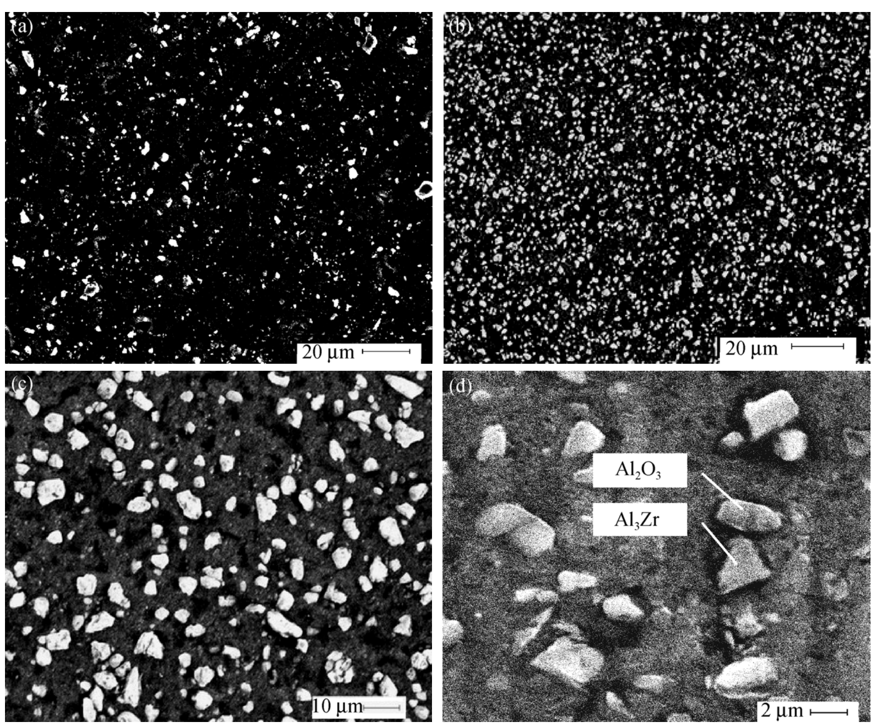

Fig. 3. Microstructures of *in-situ* synthesized (Al_2O_3+Al_3Zr)$_p$/Al composites synthesized under high-frequency magnetic field with and without pulse signal: (a) microstructure of composites synthesized without pulse signal; (b), (c), (d) microstructure, particulate sizes, and features of composites synthesized with pulse signal.

In nucleation and growth stage of particulates, the enhanced heat and mass transfer speed raises the contact opportunity of the matrix and solid zirconium dioxide. Generating the probability of Al_2O_3 and Al_3Zr reinforced phases adds up. At the same time the electromagnetic field imposes on the melt in the form of an electromagnetic wave, which augments the structural and energy fluctuations in the inner melt and decreases the critical nucleating power to a certain extent [10]. It attributes to the nucleation of Al_2O_3 and Al_3Zr particulates. In the final diffusion stage the stirring action can accelerate the mix of aluminum matrix and reinforced particulates, which promote the dispersed distributions of the reinforced particulates in the aluminum matrix.

4. Conclusions

(1) A high-frequency pulsed electromagnetic field is generated by imposing pulse signals on the high-frequency electromagnetic field. It weakens the Kelvin effect of current intensity. Distribution of magnetic density tends to be uniform in the inner coils.

(2) $(Al_2O_3 + Al_3Zr)_p$/Al composites have been fabricated under the foreign field. The size of reinforced particulates is 2-3 μm. They are well distributed in the matrix. Agitation, stirring, and turbulence phenomena appear in the melt during the synthesizing process. Incremental mass and heat transfer speed accelerate the *in-situ* fabrication process.

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