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Microstructural stability of *in-situ* TiB_2/Al composite during solution treatment

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The microstructural stability including grain growth and texture of *in-situ* TiB_2/Al composite during solution treatment (ST) at T4 state (natural aging) was investigated for the first time. The results show that the nano TiB_2 particles have a strong influence on the stability of grain size (GS) and textures during ST. The composite has exhibited improved stability of GS attributing to the Zener pinning of TiB_2 particles, resulting in no obvious increase in GS after the ST for 24 h. A modified Zener equation has been proposed for the composite with non-uniform particle distribution, which can make a good prediction on the limiting GS. For textures of the 2024 alloy and TiB_2/2024 composite, there is not much difference in the intensity of ⟨111⟩ fiber before the ST, while the ⟨l00⟩ fiber is stronger in the composite. The ⟨111⟩ and ⟨l00⟩ double fibers of the composite are stabilized by TiB_2 particles during ST at T4 state. However, the ⟨111⟩ and ⟨100⟩ fibers of the alloy are gradually replaced by ⟨113⟩, ⟨102⟩ and ⟨302⟩ ones due to the recrystallization and grain growth. Accordingly, the composite exhibits much stronger fiber textures than the alloy after the heat treatment.

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1. Introduction

In-situ TiB_2/Al composites have received the increasing attention in recent years owing to their outstanding mechanical properties, such as high modulus [1], strength [2,3] and fatigue resistance [4,5]. The *in-situ* TiB_2 particles are usually sub-micron or nano size and thermally stable, which should play an important role in the microstructural stability of deformed TiB_2/Al composites. However, the TiB_2 particles are usually non-uniformly distributed in such composites because most of the TiB_2 particles are pushed to the grain boundaries (GBs) during solidification [6]. Since the particles can exert "Zener pinning" effect on GBs and the pinning force is inversely proportional to the particle radius [7–9], it is expected the non-uniformly distributed nano TiB_2 particles may have a strong effect on the grain growth and texture.

Grain growth is a common phenomenon occurring when a metallic material is annealed after deformation. The driving force of grain growth has originated from the reduction in total GB energy. Experiencing the grain growth process, the grain size (GS, d) has become larger, and the strength is reduced in the metallic material [10–13]. In the previous studies, most of the theoretical models for the particle pinning grain growth are based on the alloys with uniformly distributed precipitates, which show the limiting grain size is greatly related to the radius and volume fraction of particles [7,9]. Flores et al. [14] found the clustered (cluster order, <5 particles) second phase particles (4–7 μm) cannot significantly control the GS in nodular cast irons, but they treated the second phase particles as random spread. Payton et al. [15] also found that the wide GS distribution can lead to grain coarsening. Moreover, most of the dispersoids or second phase particles are liable to coarsen, dissolve and move during annealing, resulting in the reduction of pinning force [16,17].

For the particle reinforced Al composites, although the particles also have "Zener pinning" effect on grain growth, the conventional large particles (>1 μm) relative to nano particles should have poor pinning force according to the Zener relationship [8,9]. Lai et al. [6] reported that the grain size of 15vol.%B_4C/Al composite increased from 43 ± 17 μm to 51 ± 20 μm when the aging time from 10 h to 2000 h at 350 °C. It is apparent that the GS is relatively stable. However, the heat treatment temperature is low and the size of B_4C particles is unknown. The authors did not discuss the reason for this phenomenon. Jiang et al. [18] investigated the grain growth mechanism in the nano B_4C particle-reinforced ultrafine grained (UFG) 5083Al matrix nanocomposites. They found that grain growth occurred by grain rotation and GB migration during thermomechanical consolidation, though the composite contained 5 vol.% B_4C nano particles. It is noted that the material used in their

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study is bulk nano-crystalline material prepared by powder metallurgy. The mean GS is ~154 nm and the B_4C particles are <100 nm. Therefore, they found that most of the GBs only had one B_4C particle, resulting in the poor pinning force. It was suggested that the particle size and particle number at GBs should dominate the grain growth. However, the research on the grain growth in the nano particles reinforced Al alloy with micron GS is very few.

Additionally, the effect of particles on the texture in Al composites has been widely investigated [19–24]. From the available data, it is concluded that the intensity of texture is to be weakened for the Al composites reinforced with large particles (>1 μm). Robson et al. [23] reported that the intensities of major deformation texture components were reduced considerably with increasing content of SiC particles in the SiC_p/Al composites due to the wide scattering of the subgrain orientations within deformation zones. Although many evidences have shown that the addition of large ceramic particles can obviously alter the texture evolution of the alloy matrix, the effect of nano particles on the texture in Al composites during grain growth has not been thoroughly understood yet.

The stability of fine grains is important for mechanical properties if the material is to undergo annealing. Although the *in-situ* TiB_2/Al composites have great promising in structural application fields and expected to exhibit good microstructural stability, little information is available on the effects of nano TiB_2 particles on their microstructural stability including grain growth and texture evolution during ST. In the case of non-uniformly distributed or clustered nano ceramic particles, the classical Zener relationship for predicting the limiting GS based on uniform distribution of particles [8,25] may be no longer valid for evaluating the limiting GS.

In comparison with the 2024 alloy, the present work is to investigate the microstructural stability of the hot extruded *in-situ* TiB_2/2024 composite during ST at T4 state. The effect of nano TiB_2 particles on the grain growth and texture evolution was discussed.

2. Experimental Procedures

The 2024 matrix alloy has a nominal composition (in wt.%) of Al-4.3Cu-1.7 Mg-0.5Mn, and the *in-situ* 5 wt.% TiB_2/2024 composite was fabricated by the salt-metal reaction method [26]. The as-cast ingots were homogenized at 490 °C for 24 h, and then were extruded at 450 °C with the extrusion ratio of 35: 1. The T4 heat treatment has been performed on both materials, including high temperature ST and natural aging at room temperature. The ST was performed at 505 °C for different time, subsequently followed by water quenching. Afterwards, the natural aging was carried out for 4 days at least. Comparably, the higher ST temperature of 505 °C, a natural aging at ambient temperature for 2024 Al alloy/matrix composite should exert little significantly influence on the grain structures of the materials [27]. As a result, the microstructural-stability was investigated by analyzing the microstructures after the T4 heat treatment.

Microstructures were examined by scanning electron microscopy (SEM) equipped with an electron backscattered diffraction (EBSD) system and transmission electron microscopy (TEM). The EBSD maps were acquired at 20 kV with step sizes of 0.05–1 μm. The hardness was measured using a Vicker's hardness testing machine (load 20 kg, 15 s). For texture analysis, four incomplete pole figures (PFs) {111}, {200}, {220} and {311} were measured up to a maximum tilt angle of 80° by Schulz back-reflection method using X-Ray diffraction (XRD). The measured surfaces were parallel to the extrusion axis. Orientation distribution functions (ODFs) and inverse pole figures (IPFs) were calculated from the PFs after the background and defocusing corrections by means of Matlab software toolbox MTEX [28].

For TEM observations, the specimens were prepared by ion milling techniques. The EBSD date was analyzed using the commercial software HKL CHANNEL5. The misorientation selected for high angle grain boundaries (HAGBs) in the EBSD maps is >15° (marked by black lines), while that 2°–15° is defined as low angle grain boundaries (LAGBs) (marked by gray lines).

3. Results and Discussions

3.1. Microstructures

Fig.1a shows back scattering electron (BSE) micrographs of nano TiB_2 particles in the *in-situ* TiB_2/2024 composite after T4 heat treatment. In the composite, the TiB_2 particles are non-uniformly distributed in the Al matrix, including the TiB_2 particles clustered at the GBs (Fig. 1a and b) and the uniformly distributed TiB_2 particles inside the grain interiors (Fig. 1a). Fig.1c shows the TEM micrograph of clustered TiB_2 particles. Overall, most of TiB_2 particles have the size ranging from 20 to 60 nm, which has been confirmed by synchrotron XRD in the former report [29]. Additionally, a few dispersed T phases ($Al_{20}Cu_2Mn_3$) that precipitate during the homogenization [30,31] are observed (marked by arrows in Fig. 1b), and they are ~0.4–1 μm in length and ~0.08–0.2 μm in width. Compared with the TiB_2 particles, the T phases have a much greater average size and lower number density (~0.05–0.1 μm^{-2}).

3.2. Grain Growth During ST

Fig. 2 shows the variation of hardness of the 2024 alloy and TiB_2/2024 composite upon the ST time at T4 state. Before 1 h, the hardness of both alloy and composite has a rapid increasing tendency attributing to the GPB zones or Cu-Mg clusters [30,32]. The maximum values of hardness for both materials are reached almost at ~1 h. The hardness of the alloy shows a regular tendency, *i.e.* the reduced hardness with prolonging the ST time due to the coarsening microstructure and recrystallization. Distinctively, the hardness of the composite has reached a plateau through the longer ST duration at T4 state, indicating its microstructural stability.

The EBSD maps and GS distributions from the cross-section of two materials for different ST time at T4 state are shown in Fig. 3.

For the 2024 alloy, the GS has a broad distribution before the ST (Fig. 3a and g), where the average GS of the alloy ($d_{average}$) is 14.8 μm and maximum GS ($d_{maximum}$) is ~90 μm. During the elevated temperature, the grain growth should occur due to the driving force of the reduction in total GB energy [19]. Hence, the non-uniform grain growth is greatly seen at the ST for 1 h (Fig. 3b), where $d_{average}$ (alloy) is 25.8 μm, and $d_{maximum}$ (alloy) is ~120 μm. At the ST for 24 h (Fig. 3c and i), the GS is remarkably coarsened ($d_{average}$ (alloy) = ~80.6 μm).

For the TiB_2/2024 composite, the GS has uniform distribution at the three states (Fig. 3d–f). Before the ST, $d_{average}$ (composite) is 4.2 μm and $d_{maximum}$ (composite) is ~25 μm. At the ST for 1 h, $d_{average}$ (composite) is slightly increased to 5.4 μm, as shown in Fig. 3e. At the ST for 24 h, it is still a little increase in the average GS, *i.e.* $d_{average}$ = 6.3 μm (Fig. 3f and i). Moreover, the increase in the maximum GS of the composite at the three states is quite slight. The increase rate of $d_{maximum}$ is lower than that of the $d_{average}$. Clearly, the grains are coarsening uniformly at a trivial rate with the increasing ST duration.

In comparison with the alloy, the GS is finer and the GS distribution is more uniform in the composite. Conclusively, the grain growth is severe and non-uniform in the alloy, but slight and uniform in the composite. Such difference should be caused by nano TiB_2 particles exerting the pinning effect on the (sub-)GBs. Additionally, the amount of LAGBs in the composite is more than those of the alloy after the ST at T4 state. The recrystallization extent of the alloy has increased significantly, in which only small amounts of ⟨111⟩ grains are unrecrystallized. However, the recrystallization of the composite is strongly inhibited.

Fig. 4 shows the grains with ⟨111⟩ orientation in the EBSD maps after the ST for 24 h. In the alloy, the LAGBs have almost disappeared in the recrystallized grains. During the recrystallization of the alloy, the migration or mergence of the LAGBs should occur and form HAGBs due to the

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