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ELEVATED TEMPERATURE DEFORMATION BEHAVIOR OF HIGH STRENGTH Al-Cu-Mg-Ag BASED ALLOY REINFORCED BY TiB_2 PARTICLES

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Abstract

TiB_2 has been known as an outstanding reinforcement particle in aluminum matrix composite due to its excellent properties compared to other particulate reinforcements. There has been intensive effort undertaken recently in development of Al-TiB_2 composites, however no literature appears to be available on the application of TiB_2 reinforcement in very high strength wrought Al-Cu-Mg-Ag alloys. In this study, the elevated temperature deformation behavior of TiB_2 reinforced Al-Cu-Mg-Ag alloy is investigated. Microstructural observation prior to deformation process shown fine equiaxed grain with TiB_2 particle distributed homogeneously along the grain boundaries and occasionally in the interior of the grains. Uniaxial compression testing was conducted to understand the thermo-mechanical behavior of this material at elevated temperature. The flow behavior during hot deformation processing of Al-Cu-Mg-Ag-TiB_2 composites was analyzed. Emphasis is given on the description of dynamic recrystallization by means of electron back scattered diffraction.

Introduction

Aluminum matrix composites (AMCs) possess superior physical and mechanical properties compared to unreinforced materials, including improved room and elevated temperature strength and stiffness, and increased wear resistance. AMCs emerged as a promising material for several engineering and structural applications both in aerospace and in automotive industry. Although particulate reinforced AMCs do not exhibit as high improvement in strength and stiffness as whisker or fiber reinforced AMCs, particulate reinforced AMCs are more easily fabricated by conventional process such as casting, extrusion or rolling, show lower production costs and their nearly isotropic properties make them commercially attractive for high volume production [1-3].

Among particulate reinforcement materials TiB_2 has been known as an excellent one, mainly due to its high melting point (2790^0C), high hardness (86 HRA or 960 HV) and elastic modulus (530 x 103 GPa), and good resistance to plastic deformation at elevated temperatures. It does not react with molten aluminum, hence it is possible to avoid formation of brittle reaction products at reinforcement – matrix interfaces [4]. Because of those reasons, considerable effort has been devoted to the development of TiB_2 as a particulate reinforcement in AMCs.

The use of Al-Cu-Mg-Ag based high strength alloy as a matrix and TiB_2 as a particulate reinforcement offers unique combination of strength, stiffness and elevated temperature stability. Firstly, addition of Mg and Ag into Al-Cu base alloy improve the age hardening response of the system due to the formation of fine and uniform hexagonal plate like precipitate Ω (Al_2Cu) on the $\{111\}_\alpha$ plane in the Al matrix. Both room and elevated temperature mechanical properties, thermal stability and creep resistance of Al-Cu-Mg-Ag alloy compared to a standard Al-Cu alloy is improved [5, 6]. Further, addition of TiB_2 into Al-Cu alloys refines the grain size of as-cast material leading both to increased plasticity and stress [7]. Moreover, Mandal *et al* have observed significant enhancement in the aging kinetics, yield strength and ultimate tensile strength without any significant loss in ductility in Al-Cu alloy due to the presence of TiB_2 particles [8]. However, increase of microstructural complexity in the composite material tends to reduce the size of safe processing window of this alloying system, especially during industrial scale manufacturing such as hot extrusion or rolling of this high strength AMC. Therefore, a systematic understanding of workability of this particular alloying system is necessary to enable safe and possibly also low cost processing route and to establish control over microstructure and final mechanical properties of this material.

In this paper the hot deformation behavior of TiB_2 reinforced Al-Cu-Mg-Ag alloy was investigated using hot compression test performed at various temperatures and strain rates. The effect of deformation conditions on the flow stress behavior and microstructural evolution, and the relationship between values of Zenner-Holomon parameter (Z) and dynamic softening mechanism of homogenized material was studied using electron backscattered diffraction (EBSD).

Experimental Procedure

The TiB_2 reinforced Al-Cu-Mg-Ag based alloy used in the present investigation was provided by Aeromet Int. PLC in the form of cast bars. Cylindrical compression samples with diameter of 8 mm and a height of 10 mm were machined from the as-cast bars. The specimens were homogenized and directly water quenched after homogenization.

Compression tests were carried out at temperatures of 300^0C, 350^0C and 450^0C and strain rates of $0.01 s^{-1}$, $0.1 s^{-1}$, and $1 s^{-1}$. The specimens were heated to the test temperature, soaked for 10 min for equilibrium and deformed at predetermined strain rate. A thermocouple was inserted to the anvil in order to measure and control actual temperature. Boron nitride lubricant was used to minimize the friction between the specimen and anvil during hot deformation. The specimens were compressed to 0.7 strain and quenched in water immediately to preserve the as-deformed microstructure.

The microstructures prior and after the deformation were investigated. X-ray diffraction and microstructure examination under scanning electron microscopy was conducted to check the phase composition and microstructure of initial as-cast and fully homogenized material. The deformed specimens were sectioned parallel to the applied load. All metallographic samples were prepared using conventional grinding and polishing method. All published micrographs were recorded at the central zone of the polished samples avoiding dead metal zone of the compressed specimens.

Result and Discussion

Initial Microstructure

Fig. 1 shows initial microstructure of TiB_2 reinforced Al-Cu-Mg-Ag alloy prior to deformation process. The microstructures consisted of fine equiaxed grain with average grain size of 23µm. Electron dispersive spectroscopy (EDS) maps showed homogeneously distribution of TiB_2 particle along the grain boundary and triple junction and also occasionally in the interior of the grain. It also shows the distribution of secondary phases (Al_2Cu) along the grain boundaries.

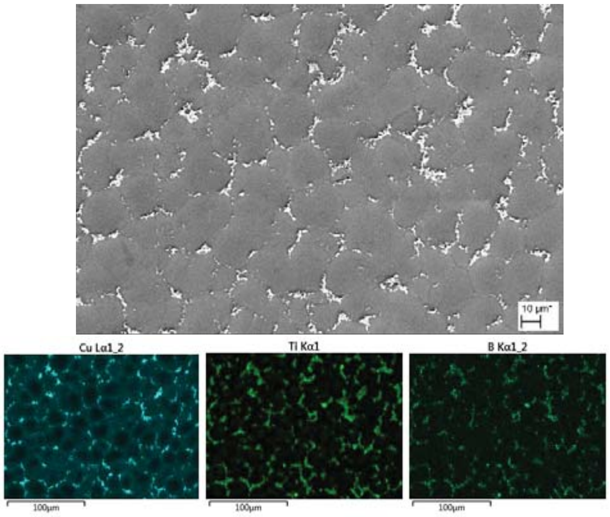

Fig.1. SEM image and EDS maps of Cu, Ti and B of as cast TiB_2 reinforced Al-Cu-Mg-Ag alloy

In addition to reducing the residual stresses and stress relief after the casting, homogenization was conducted to dissolve all Cu-containing secondary phases and to distribute alloying elements uniformly in the Al solid solution. Homogenization process dissolved coarse grain boundary eutectic phases Al_2Cu into matrix, however TiB_2 particles remain unaffected.

Flow Stress Behavior

The flow stress curves obtained during hot compression of homogenized TiB_2 reinforced Al-Cu-Mg-Ag alloy under various temperature and strain rates are shown in Fig. 2. The result shows that at the beginning of deformation process the flow stress increased rapidly and then after reaching a peak it is either nearly steady or decrease, indicating a dynamic flow softening. The highest peak stress of 157 MPa was obtained at the lowest deformation temperature of 300^0C and fastest strain rate of 1 s^{-1} while the lowest peak stress of 27 MPa was acquired at deformation temperature 450^0C and strain rate 0.01 s^{-1}.

The deformation at elevated temperature is a competitive process between dynamic softening and continuous work hardening. In the early stages of deformation work hardening is dominant and could be measured in its magnitude by the value of peak stress; in later stages the dynamic softening (dynamic recovery and dynamic recrystallization) is offsetting the effect of work hardening, with the flow stress either remaining steady or decreasing, based on the kinetics of the softening process [9-11].

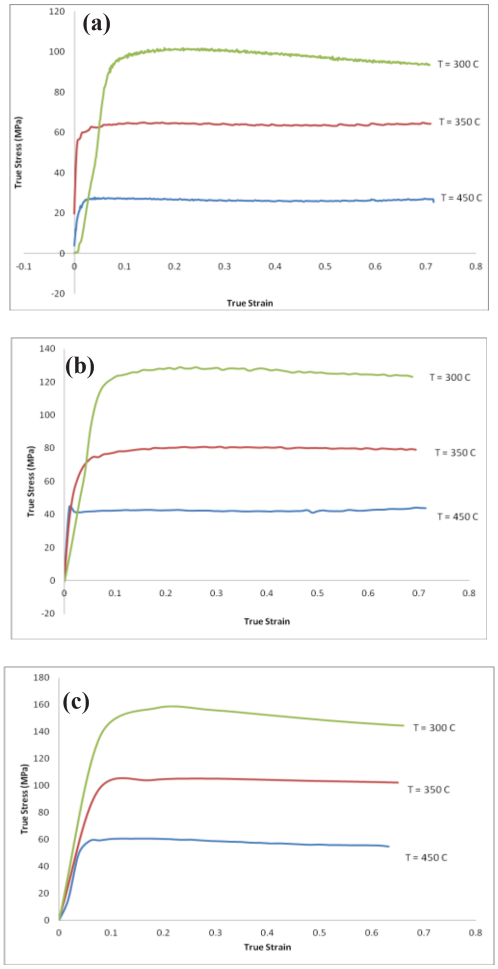

Fig. 2. True stress vs true strain curves for TiB_2 reinforced Al-Cu-Mg-Ag alloy during hot compression deformation a) $\dot{\varepsilon}$ = 0.01 s^{-1}, b) $\dot{\varepsilon}$ = 0.1 s^{-1}, c) $\dot{\varepsilon}$ = 1 s^{-1}

Constitutive Relationship

Constitutive equation is used in high temperature deformation to describe hot working behavior of alloys and to demonstrate the effect of the deformation conditions on the state of the flow stress. The hyperbolic – sine equation developed by Sellars and McTegart [12] is widely used to illustrate the relationship between deformation temperature, strain rate and flow stress:

$$\dot{\varepsilon} = A[\sin h\,(\alpha\sigma)]^n \exp(-\frac{Q}{RT}) \qquad (1)$$

Where n and A are materials constant, α is the stress multiplier, Q is the activation energy for deformation (kJ/mol), R is the universal gas constant (8.341 J/mol K), T is temperature (K), and σ is the flow stress (MPa). The σ value can be either peak stress or flow stress. The stress multiplier α is defined as $\alpha = \beta/n_1$ where β and n_1 are calculated from the slope of the plot of ln ($\dot{\varepsilon}$) – σ and ln ($\dot{\varepsilon}$) – ln (σ) respectively.

By differentiating Eq. (1) an equation for activation energy Q is obtained as follows:

$$Q = R\left[\frac{\vartheta \ln \dot{\varepsilon}}{\vartheta \ln[\sin\mathrm{h}\ (\alpha\sigma)]}\right]_{\dot{\varepsilon}}\left[\frac{\vartheta \ln[\sin h(\vartheta\sigma)]}{\vartheta(\frac{1}{T})}\right] = RnS \qquad (2)$$

Where n is the mean slope of the plots of ln $\dot{\varepsilon}$ - ln [sin h(ασ)] at different temperature and S is the mean slope of plots of ln [sin h(ασ)] – 1/T at various strain rates. The relationship between ln $\dot{\varepsilon}$ - ln [sin h(ασ)] and ln [sin h(ασ)] – 1/T which was derived from the true strain – true stress curve is shown in Fig.3. Then the activation energy can be calculated using Eq.2. The obtained material constant values n, α and activation energy Q are shown in Table 1.

Table 1. Values of material constant and activation energy of TiB_2 reinforced Al-Cu-Mg-Ag alloy

n	α (MPa^{-1})	Q ($kJ\ mol^{-1}$)
5.62	0.0145	203

The activation energy Q is an important physical parameter and indicator of operating deformation mechanism during the hot deformation process. In the present study, the value Q for the TiB_2 reinforced Al-Cu-Mg-Ag alloy is 203 kJ/mol which is slightly higher than the activation energy of similar unreinforced Al-5.3Cu-0.8Mg-0.5Ag-0.3Mn-0.15Zr alloy (196 kJ/mol)reported by Liu *et al* [13] and also higher than Q of pure aluminum (142 kJ/mol) [14]. This value is also higher than wrought Al 2024 reported by Charpentier et al [15]. They reported that wrought Al-2024 exhibit flow softening due to dynamic recovery at temperatures above 250^{0}C with activation energy in the range of 90 – 200 kJ/mol

Generally, high values of activation energy observed in heat treatable alloys or AMCs were related to the presence of alloying elements dissolved in the solid solution, presence of precipitates inside the matrix, or reinforcing ceramic particles [15, 16]. In general, dislocation moving through a solid solution will encounter solute drag by solute atoms or pinning effect by particles inside the matrix, thereby raising the energy required for its movement. In this case it can be deduced that higher Q of the alloy is assumed to be associated with an increase in the Mg and Cu held in the solution by the homogenization process and also by the presence of fine distribution of TiB_2 particles.

In the elevated temperature deformation the deformation temperature (T), strain rate ($\dot{\varepsilon}$) and strain (ε) are all influencing the evolution of microstructure during hot deformation. The strain rate and deformation temperature are often incorporated into a single parameter called Zener Hollomon parameter (Z), which is defined as:

$$Z = \dot{\varepsilon}\exp\left(\frac{Q}{RT}\right) \qquad (3)$$

The Zener Holomon parameter (Z) obtained from the presented data is shown in Table 2. The highest Z value was obtained when the alloy was hot compressed at 300^{0}C, with the fastest strain rate of 1 s^{-1}; the lowest Z value was obtained when the alloy was deformed at temperature 450^{0}C with strain rate 0.01 s^{-1}. High Z values suggest climbing and sliding of dislocation accompanied by rearrangement of dislocations into walls of low angle subgrain boundaries. At lower Z values, where diffusion processes and the dislocations movement are more significant, merging of some grains and the low angle grain boundaries transformation into high angle grain boundaries through absorbing dislocations is possible (for details see section on microstructure below).

Table 2. Z value under typical deformation conditions

Deformation Condition	Z (s^{-1})		
	0.01 s^{-1}	0.1 s^{-1}	1 s^{-1}
300^{0}C	3.42 x 10^{16}	3.42 x 10^{17}	3.42 x 10^{18}
350^{0}C	1.11 x 10^{15}	1.11 x 10^{16}	1.11 x 10^{17}
450^{0}C	4.89 x 10^{12}	4.89 x 10^{13}	4.89 x 10^{14}

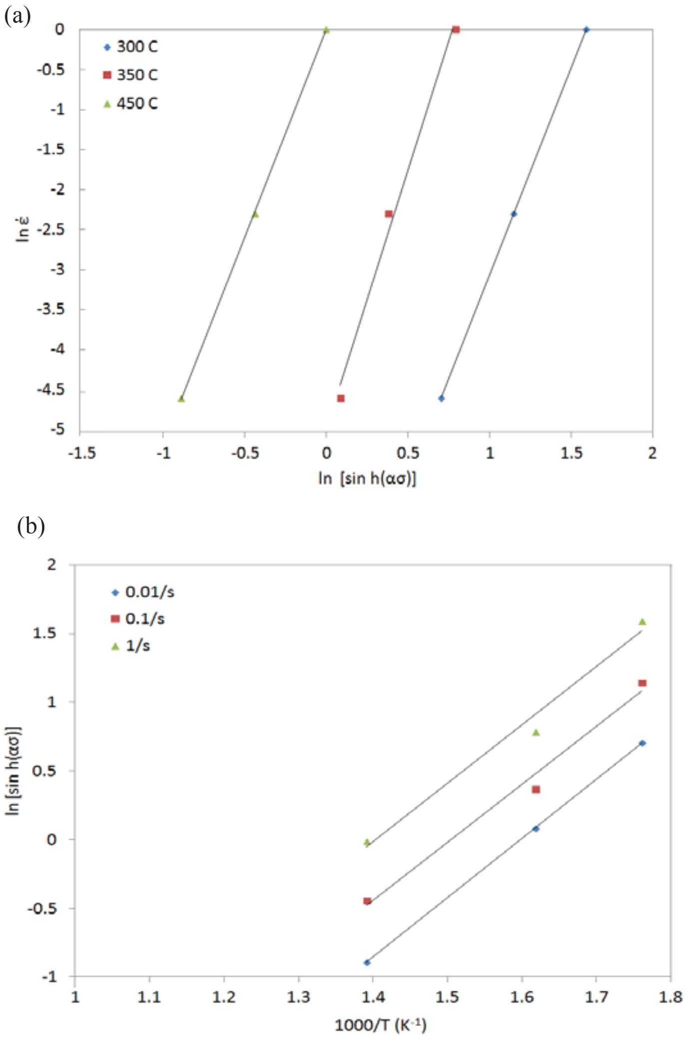

Fig. 3. Relationship between (a) ln $\dot{\varepsilon}$ - ln [sin h(ασ)] and (b) ln [sin h(ασ)] – 1/T

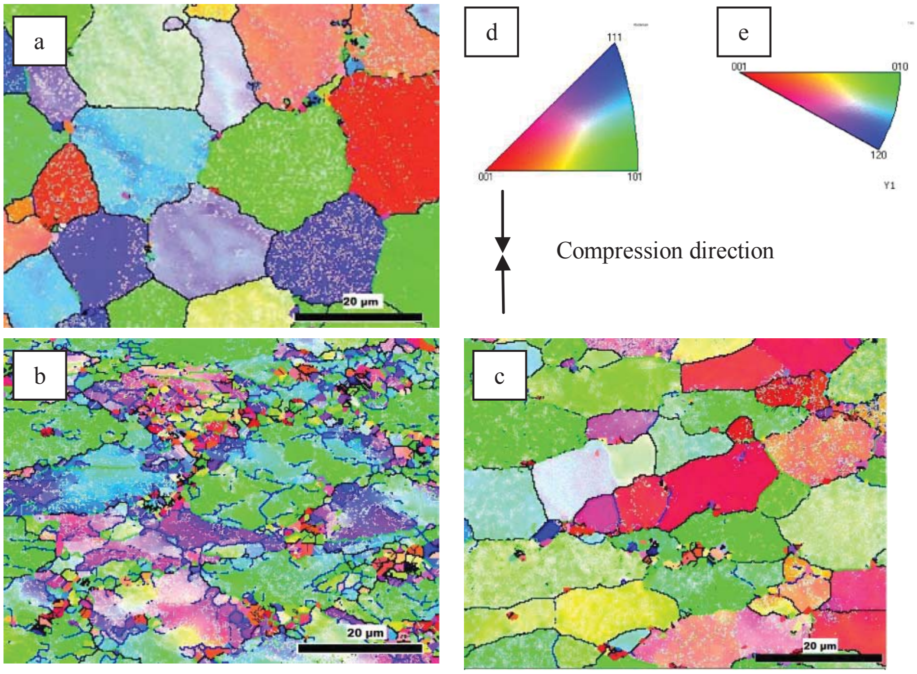

Fig. 4. IPF maps of TiB_2 reinforced Al-Cu-Mg-Ag alloy of (a) as homogenized and under different deformation condition (b) 300^0C at strain rates 1 s^{-1}, (c) 450^0C at strain rates 0.01 s^{-1}, representative of the color code used to identify crystallographic orientation on standard stereographic projection for (d) Al and (e) TiB_2

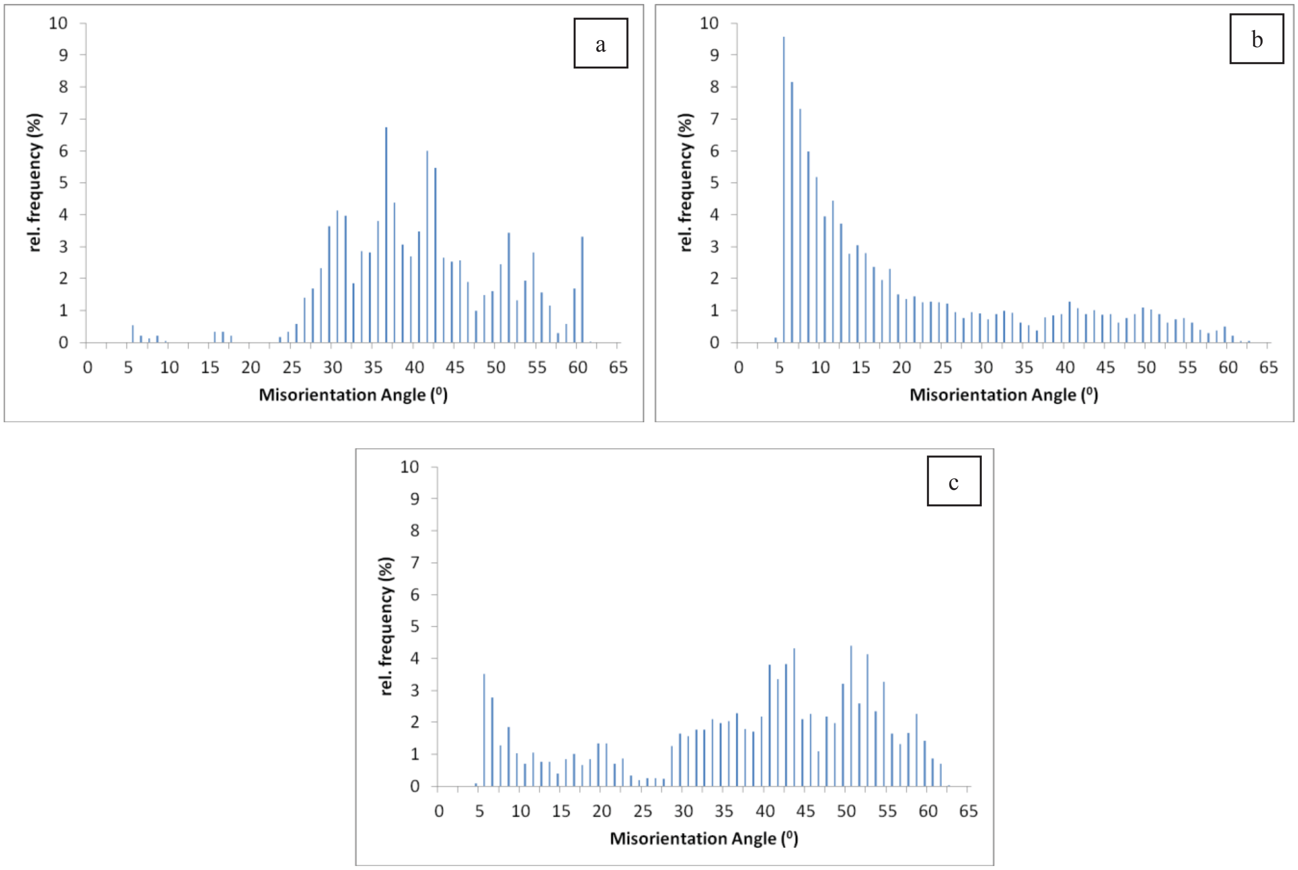

Fig.5. Grain boundary misorientation distribution (a) as homogenized, (b) 300^0C, $1s^{-1}$; (c) 450^0C, 0.01 s^{-1}

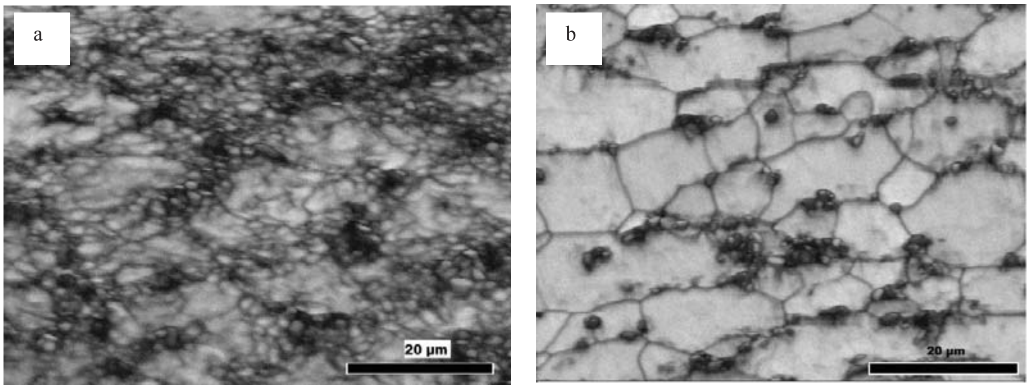

Fig.6. Band contrast image of TiB_2 reinforced Al-Cu-Mg-Ag alloy under different deformation conditions (a) 300^0C, $1s^{-1}$; (b) 450^0C, 0.01 s^{-1}

Microstructure Evolution

Electron backscattered diffraction (EBSD) method was conducted to study the microstructure evolution during hot deformation process. The mechanism of dynamic softening active during hot deformation was described on basis of analysis of inverse pole figure (IPF) maps provided by EBSD as shown in Fig. 4.

The EBSD analysis result shows that the homogenized TiB_2 reinforced Al-Cu-Mg-Ag alloy consisted of high angle grain boundaries, with misorientation more than 30^0 as shown in Fig.5. When the alloy was deformed at 300^0C with 1 s^{-1} strain rate, low angle grain boundaries were observed with dominant misorientation below 10^0, indicating high density of cell and subgrain structure formation. As temperature of deformation increased to 450^0C with lower strain rate 0.01 s^{-1} applied, the density of low angle grain boundary decreased due to possible further polygonization and growth of recrystalized grains active at higher temperatures. Misorientations lower than 5^0 were excluded from the results to avoid effect of misindexed areas.

As dynamic recovery occurred, dislocations generated as a result of plastic deformation were rearranged to form low energy dislocation walls that define polygonized subsgrains as shown in the band contrast image in Fig. 6 (a). The microstructure shows deformed grains which are divided into subgrains. As the subgrains structure develops, the serrations begin to appear and usually the apex serrations of one grain occur where the grain boundary migration has taken place along a subgrain boundary into a neighboring grain. This local migration of the grain boundaries are constantly being eliminated and reformed during continuous dynamic recovery [17].

The recrystallized nucleus formed and the grain boundaries become straight and clear as shown in Fig. 6(b). Afterwards, the recrystallized grain grew through migration of dislocations with high angle grain boundaries becoming straight and clear. With increasing deformation temperature and decreasing the strain rate the main softening mechanism is transformed from dynamic recovery to dynamic recrystallization. Correspondingly, the flow stress decreased dramatically with decreasing Z value.

Further work is being carried out on the unreinforced high strength Al-Cu-Mg-Ag alloy to compare the contribution of TiB_2 and its effect on the deformation ability of this particular AMC material. Transmission Electron Microscopy is also under way to provide more detailed analysis of the role of TiB_2 and its interaction with dislocation movement.

Conclusions

1. The highest peak stress of TiB_2 reinforced Al-Cu-Mg-Ag alloy is 157 MPa which was obtained at deformation temperature 300^0C and strain rate 1 s^{-1} while the lowest peak stress is 27 MPa which was acquired at deformation temperature 450^0C and strain rate 0.01 s^{-1}.
2. The main softening mechanism of TiB_2 reinforced Al-Cu-Mg-Ag alloy during deformation at high Z value is dynamic recovery, while at the low Z value continuous dynamic recrystallization was observed.

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