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Microstructure evolution of 6016 aluminum alloy during compression at elevated temperatures by hot rolling emulation

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Abstract: Microstructure evolution of 6016 aluminum alloy during compression at elevated temperatures was studied by single-pass high temperature compression experiments on Gleeble–1500 thermal-mechanical simulator. The microstructures under various deformation conditions were also analyzed by optical microscope (OM) and transmission electron microscope (TEM). The results indicate that during hot compression deformation, apparent activation energy of the alloy is 270.257 kJ/mol, stress exponent is 8.5254, natural logarithm of hyperbolic sine flow stress of the alloy has linear relationship with natural logarithm of temperature compensation Zener-Hollomon (Z-H) parameters, and main deformed microstructures of the alloy at low temperature and low strain rate are dynamic recovery microstructures in contrast to a little geometric dynamic recrystallization microstructure within local area at high temperature. Main softening mechanism during deformation of the alloy at high temperature is dynamic recovery, while the dynamic recrystallization occurs partially only at high temperature and high strain rate. And subgrain size decreases with increase of Z-H parameter.

Key words: 6016 aluminum alloy; hot compression; microstructure evolution; hot rolling

1 Introduction

6xxx aluminum alloy is an Al–Mg–Si series alloy which may be strengthened via heat treatment processes [1,2]. It inherently possesses ideal formability, corrosion stability, weldability, high fatigue strength, as well as medium static strength. It is used as important material to make military or civilian products, such as airplane oil tank, oil tube, sheet metal parts of automobile, ships and other transportation facilities, support of instrument, street light, refrigeration devices, television tower, drilling equipment, missile parts, and rivets wire.

The 6016 aluminum alloy is the preferred choice material of automobile body panels for aluminum car, such as the body panels of Audi A8 aluminum car [3]. The called "all aluminum" car is the first mass-market car in Europe and America with an aluminum chassis, an

all aluminum monocoque, co-developed with Alcoa.

Flow stress is an important fundamental magnitude which can represent characterizations of plastic deformation of metals and alloys. The flow stress is also a fundamental parameter that determines the deformation force and toque and power requirement of metal forming equipment. It is important for us to conduct further study on rheological behavior of metals or alloys during thermal deformation in developing their hot working processes. Meanwhile, there is an important impact on microstructures evolution of materials about their performance during metals' hot deformation [4].

In recent years, it is under spotlight to research on thermal deformation of aluminum alloys domestically and internationally. The formation and changes in texture during hot rolling or intermediate annealing were investigated [5–7]. The formability of these aluminum alloys or the influence of process parameters on the

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microstructure, mechanical properties, grain structure or texture during friction stir spot welding were studied [8,9]. The modeling and experimental researches of ECAP or ECAE process were conducted, which could admittedly improve the strength and ductility in aluminum alloy, but a series of problems remain in application of these processes [10–12].

BUTUC et al [13] studied forming limit diagram along strain path, and LEWANDOWSKA [14] paid attention to the dependence of the deformation microstructure on the strain path for these aluminum alloys. Flow stress and constitutive model of aluminum alloy were built in separate researches [15–17], specially for 6016 aluminum alloy [18,19]. Details on bake hardening response or mechanism were discussed [20,21]. However, little information is available regarding the microstructure evolution on 6016 aluminum alloy during hot deformation but for others, such as Al–Mg alloys [22,23], Al–Cu alloys [12,24] and Al–Zn–Mg–Cu alloys [25–27].

In this work, thermal deformation behavior of 6016 aluminum alloy was investigated with Gleeble–1500 simulation system, and on this basis the material parameters were obtained. Furthermore microstructures and appearance characteristics of the aluminum alloy under different deformation conditions were observed by means of optical microscope (OM) and transmission electron microscope (TEM). The results will be useful to serve as a theoretical basis for development and optimization of thermal processing technology.

2 Experimental

2.1 Preparation of alloys

The nominal chemical composition of the 6016 aluminum alloy under these experiments is shown in Table 1. The alloy bars under hot rolling in factory with above-mentioned chemical composition were heat treated for homogenization at 550 °C for 12 h in electrical resistance furnace and then quenched in water. Afterwards they were processed into cylinder specimens, with a size of 10 mm in diameter and 15 mm in height, for these experiments performed later.

Table 1 Chemical composition of AA6016 (mass fraction, %)

Al	Si	Mg	Cu	Mn
Bal	1.0-1.5	0.25-0.6	0.2	0.2
Cr	Zn	Ti		Fe
0.1	0.2	0.15		0.5

2.2 Experimental methods and procedure

Compression experiments were performed on Gleeble-1500 thermal-mechanical physical simulation

system. Before compression, a piece of 0.25 mm-thick graphite foil was used as a lubricant filled between the ISO-T anvils and the grooves of the cylindrical specimens at each of both ends to reduce the compression friction effects on the stress state during compression. The graphite was cut to a disc with the diameter of 2 mm which was larger than the initial specimen diameter and inserted between the anvil and the specimen. The specimens were electric allov heated with their own resistance at a rate of 10 °C/s and then heat preservation was kept for 3 min before compression. The experiments were performed at 300-500 °C, at some strain rate among $0.1-10 \text{ s}^{-1}$, and into a total compression strain of 0.6 (true strain) by hot compression. Then, the deformation specimens were immediately quenched in water at the end of compression to retain the high temperature deformation microstructures.

Hot deformation microstructures of the alloy were observed with OM of Zeiss and microscopic analyzed by means of JEOL JEM3010 TEM. Before being observed via TEM, the deformation specimens were cut with wire-electrode cutting method into samples of thickness of 0.3 mm in the center along the compression direction, and the cutting samples were ground into thin foils of 0.07 mm in thickness. The TEM samples, made with conventional methods, were thinned by electro polishing in electrolyte, which was composed of nitric acid and methanol in a mass fraction of 25% and 75%, respectively, from -30 °C to -25 °C, within a voltage of 10-12 V.

3 Results and analysis

3.1 Flow stress analysis

When the requirement of force and equipment power for a forming process is to be determined during metal processing, two groups of factors are actually considered: a group being implicit of the metal to be deformed, and the other implying forming process or tool type with their interfacial condition, such as rolling, forging and extrusion. From the point of view of flow stress, the factor group, which is related to the deformed material itself, covers many forming process parameters, such as strain, strain rate, and temperature. Various parameters about the material and forming process affect the flow stress.

It's revealed in the experiments [19] that there is a steady state deformation characteristic for 6016 aluminum alloy during hot compression deformation, i.e, the true stress remains constant generally when true strain really exceeds a certain value. Based on Zener-Hollmon parameter method, the material parameters of 6016 aluminum alloy under thermal deformation can be calculate by the relationships of flow stress σ with deformation temperature *T* and strain rate $\dot{\varepsilon}$. Parameter *Z* of Zener-Hollmon method, meaning the strain rate after compensated against temperature, can be expressed as follows [15,28]:

$$Z = A[\sinh(\alpha\sigma)]^n = \dot{\varepsilon} \exp(Q/RT)$$
(1)

where Z is the Zener–Holloman parameter, A and α are the temperature-independent constants of material, n is the stress exponent, R is the gas constant, T is the temperature of the alloys in Kelvin, and Q is the apparent activation energy for deformation in kJ/mol, which reflects the balance between strain hardening and dynamic softening of the alloy in high-temperature plastic deformation process, and can be calculated as follows [29,30]:

$$Q = R \left\{ \frac{\partial \ln[\sinh(\alpha\sigma)]}{\partial(1/T)} \right\}_{\dot{\varepsilon}} \left\{ \frac{\partial \ln \dot{\varepsilon}}{\partial \ln[\sinh(\alpha\sigma)]} \right\}_{T}$$
(2)

The above-mentioned parameters would be obtained by solving the relationships of $\dot{\varepsilon} - \sigma$ according to different stress levels. The result is: Q=270.257 kJ/mol, $\alpha=0.0183$ mm²/N, n=8.5254, $A=2.62\times10^{17}$ s⁻¹. Then the relationship of the parameter Z in natural logarithm format and σ in a format of natural logarithm of hyperbolic sine can be obtained, as shown in Fig. 1.



Fig. 1 Relationship between flow stress σ and parameter Z of 6016 alloy after hot compression

Figure 1 shows that the relationship between $\ln[\sinh(\alpha\sigma)]$ and $\ln Z$ meets linear function. It indicates that the relationships among flow stress of the alloy, deformation temperature and strain rate in hot compression process of 6016 aluminum alloy, can be described by Arrhenius formula. The process of hot compression deformation of the alloy is under control of the thermal activation procedure, meanwhile the mechanisms of deformation and thermal softening under hot compression are connected with deformation activation energy.

3.2 Microstructure evolution by OM analysis

The microstructures variation of 6016 aluminum alloy under various deformation conditions, e.g., at diverse deformation temperature, at distinct strain rate, as shown in Fig. 2, indicates that the microstructures of the alloy after hot compression deformation are mainly the thermal deformation microstructures generated during hot compression (Figs. 2 (a)–(c)) in the case of low temperature and low strain rate, meanwhile the grain is significantly elongated along the deformation direction, and only dynamic recovery has occurred during the hot deformation. Also, the microstructures variation indicates that jagged grain boundaries appear in the condition of high deformation temperature of 500 °C shown in Fig. 2(d), which means that dynamic recrystallization in the alloy begins. The fine equiaxed crystal grains grow around the long and narrow deformation grains of the alloy in the case of high deformation temperature and high strain rate (Figs. 2 (e), (f)), which means that dynamic recrystallization aggravates and size of recrystallized grains decrease but the number of grains gradually increase.

3.3 Microstructures evolution by TEM analysis

The TEM images of the alloy under different deformation conditions are shown in Fig. 3. Scientific studies [31,32] revealed that usually a wealth of subgrain structure would be generated during aluminum alloys thermal deformation, and meta-structures engender in the initial stages or the transitional stage of deformation. It can be perceived further from Fig. 3 that sub-grain size after hot deformation decreases and dislocation density increases, for 6016 aluminum alloy, as value of $\ln Z$ increases (i.e., lower deformation temperature and larger strain rate). In contrast, with $\ln Z$ value decreases, dislocation density in the interior of the deformation alloy significantly reduces but sub-grain size increases, and sub-grain boundaries are straight and clear.

According to the hot deformation activation theory of metals, in the steady-state deformation stage, the softening of the material is mainly caused by cross-slipping of screw dislocations and climbing of edge dislocation. When dislocations rapidly climb or more than two slip systems start to move at high temperatures, dislocations can be arranged, through the pile effect, perpendicular to the dislocation walls of the slip plane and transform into sub-grains. Furthermore, dislocations depart from the subgrain boundaries under the applied stress and the stability of subgrain boundaries is reduced. Then, vacancies accumulation due to strain makes dislocations climbing rate increase. This results in a large number of dislocations entering by cross slipping and



Fig. 2 OM images of compressed specimens: (a) 300 °C, 0.01 s⁻¹; (b) 400 °C, 0.1 s⁻¹; (c) 400 °C, 1 s⁻¹; (d) 500 °C, 0.01 s⁻¹; (e) 450 °C, 0.1 s⁻¹; (f) 400 °C, 10 s⁻¹



Fig. 3 TEM images of microstructure evolution of compressed specimens: (a) 450 °C, 0.01 s⁻¹ (ln*Z*=40.4); (b) 400 °C, 0.1 s⁻¹ (ln*Z*=46.0); (c) 350 °C, 1 s⁻¹ (ln*Z*=52.2)

climbing into the sub-boundaries and re-forming new sub-boundaries. Applied stress-induced sub-grain boundaries crush and dislocations-induced subgrain boundaries restructuring lead to rapid rearrangement of grain boundaries. This process is called as repeat polygonization. With the decrease of $\ln Z$ values (i.e.,

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temperature increases, strain rate decreases), the ability of atomic thermal activation enhances, and in addition, dislocations climb accelerates as a result of a large number of vacancies. Afterwards, dislocations cancel each other out, restructuring is more thorough, repeat polygonization ratio gets higher, subgrain size becomes larger and substructure reaches higher completion level [33].

4 Discussion

Scientists [23,34,35] demonstrated that the dislocations in aluminum and its alloys are relatively easy to cross slip and climb during high temperature deformation. Because there are large stacking fault energy in the material and relatively small self-diffusion capacity for atoms of the material, consequently, the polygonization sub-grain structure is easy to emerge during the deformation process, which means that dynamic recovery occurs. Therefore, the dynamic recovery is the main softening mechanism of aluminum and its alloys during hot deformation. From the experimental results, the main softening mechanism of the 6016 aluminum alloy in experiment is dynamic recovery in high-temperature plastic deformation process. Meanwhile, only dynamic recovery occurrs for 6016 aluminum alloy at low temperature and low strain rate (Figs. 2(a)-(c)); in contrast, dynamic recrystallization occurs at high temperature and high strain rate within the local geometric deformation area, as shown in Figs. 2(d)-(f)), but only a very few. In the strict sense, the essence of geometric dynamic recrystallization also belongs to the scope of dynamic recovery.

Mechanisms of dynamic recovery cover the following patterns [36]: 1) cross slip of screw dislocation; 2) edge dislocation climbing; 3) non-conservative movement of edge jog on sliding screw dislocation; 4) nail-off of nailed dislocation and entangle-off of the three-dimensional dislocations network. Under the above mentioned experimental conditions, the dynamic recovery of high temperature plastic deformation is the main softening mechanism. Therefore, their microstructure evolution in high temperature deformation process is mainly determined by the hardening and dynamic recovery, and its essence is the generation, motion and annihilation of dislocations.

In the early stages of plastic deformation, dislocation density increases to form the cell structure of dislocations with strain increasing. Afterwards, a large number of crystal dislocation tangles emerge on the wall of dislocation cell structure, and then transform into cellular sub-structure (high-density micro-dislocation network in microscopic view). An increase of dislocation density leads to the occurrence of the dynamic recovery process; meanwhile, dislocations of the center of the cell migrate to the direction of the dislocation cell wall, and further cancel each other out with the opposite sign dislocations of cell wall, resulting in the annihilation of dislocation and leading to the increase of disappearance rate of the dislocations also. Then, this will lead, due to the lower dislocation density, to grain boundaries of local cell gradually sharpening and further evolve into sub-boundaries through dislocation reactions.

In the case of dynamic recovery, with strong activity of subgrain boundaries, subgrain boundaries movement will accelerate due to the moving dislocations. This is because, usually during hot deformation, subgrain structure remains equiaxed throughout, so any crystal boundary must move at the average speed of movement defined through sector component. In order to maintain the equiaxed grain boundary structure, sub-boundaries must be flectional. But only when the composition of dislocation structure components of the grain boundary network within sub-boundaries is continuously changed, can the sub-boundaries be bendable.

In the case of dynamic recovery, sub-boundaries are down into mobile dislocation broken with provide multiple-Burgers constant, which can bendability and thermally-activated mobility of sub-boundaries. The reason why sub-grain boundary mobility enhances more under dynamic conditions than under static condition is due to the mutual exchange reaction between movability dislocations and mobile grain boundaries.

As shown in Fig. 3, sub-grain size and dislocation density are related to the deformation temperature and deformation rate. When temperature rises, movable dislocation activity enhances, thereby the activity of subgrain boundaries improves, and then subgrain grows up into adjacent subgrain. As a result, sub-grain size increases. The larger deformation rate is, the smaller size the formative sub-grain has. Thus, the size of sub-grain can be controlled by adjusting the deformation temperature and strain rate.

5 Conclusions

1) The apparent activation energy of 6016 aluminum alloy during hot compression deformation is 270.257 kJ/mol, and the stress exponent is 8.5254. The natural logarithm of the hyperbolic sine flow stress of 6016 aluminum alloy and the natural logarithm of temperature compensation Z-parameters are in compliance with the linear relationship during hot compression deformation, indicating that the process is controlled by heat activation during hot compression deformation.

2) At low temperature and low strain rate, the main

deformed microstructures of 6016 aluminum alloy are dynamic recovery microstructures; in contrast, a little geometric dynamic recrystallization microstructure exists within the local area of the alloy at high temperature.

3) Internal dislocation density and subgrain size of the alloy are related to the value of natural logarithm of Zener-Hollmon parameter. With the decrease of value of natural logarithm of Zener-Hollmon parameter, internal dislocation density of the deformation alloy significantly reduces, subgrain size increases, and sub-boundaries get clear.

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6016 铝合金热变形实验模拟热轧显微组织演变

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摘 要:采用 Gleeble-1500 热模拟机研究 6016 铝合金单道次高温压缩变形时的显微组织演变。采用光学显微镜 和透射电子显微镜分析合金在不同变形条件下的组织形貌特征。结果表明:在高温压缩变形时,该合金的变形激 活能为 270.257 kJ/mol,硬化指数为 8.5254;流变应力双曲正弦的自然对数值与温度补偿 Zener-Hollomon 参数自 然对数值成线性关系;合金低温、低应变速率时的主要变形组织为动态回复组织,而高温变形时产生局部动态再 结晶组织;该铝合金高温变形时的主要软化机制为动态回复,只有在高温、高应变速率下发生部分的动态再结晶; 合金平均亚晶粒尺寸随温度补偿应变速率 Zener-Hollomon 参数的升高而减小。 关键词: 6016 铝合金;热压缩;显微组织演变;热轧

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