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ARTICLE

Superplasticity Study of TA15 Alloy Based on Variable *m* Value Method

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Abstract: Superplasticity of TA15 alloy was investigated based on the variable strain rate sensitivity exponent *m* value method. The superplastic tensile tests were conducted at 1053~1223 K. Results show that the elongation of TA15 alloy is 580%~1922%. The microstructure analysis shows that the grains are coarsened with increasing the temperatures but remain the equiaxed structure during the deformation. In addition, the phase transformation $\alpha \rightarrow \beta$ occurs at 1223 K, resulting in the serious reduction in superplastic property. Compared with that treated by the constant strain rate method, the superplasticity of TA15 alloy treated by the variable *m* value method is greatly enhanced. During the superplastic deformation, the mechanical properties and microstructure evolution are in good agreement with the Ashby-Verrall model. Therefore, it can be inferred that the dominant superplastic mechanism for TA15 alloy based on variable *m* value method is the grain boundary glide accommodated by diffusion creep.

Key words: TA15 alloy; superplasticity; microstructure evolution; *m* value

The superplastic polycrystalline materials can undergo extensive neck-free tensile deformation prior to fracture under the appropriate conditions of temperature and strain rate[1,2]. The superplastic deformation (SPD) is characterized by low flow stress and high uniformity, which brings considerable commercial interests in complex components prepared by superplastic forming (SPF). A great number of efforts have been made to investigate the superplastic behavior of various metallic alloys, such as titanium-based, aluminum-based, and nickel-based alloys. As the metallic structural materials, titanium alloys are widely used in aerospace industry because of their excellent mechanical properties[3,4]. TA15 alloy is an α-type titanium alloy with high aluminum equivalence, and has increasingly extensive application in the aerospace field because its mechanical properties are close to those of α/β titanium alloys[5], which is used for large-scale complex integral components, such as bulkhead, girder, and junction, therefore reducing the aircraft mass and improving the structure performance[6]. However, these components with complex shape are difficult to form through the conventional forging process because the deformation resistance of TA15 alloy is high and its deformation temperature range is narrow. Therefore, SPF appears to be a cost-effective process for manufacturing the large complex components. During the past several decades, the superplasticity of titanium alloy has been widely investigated, but most studies focus on the duplex phase titanium alloys, such as Ti-6Al-4V and TC11[7-10]. The superplasticity of the near α titanium alloy is rarely reported.

Superplastic tensile test is usually conducted at constant velocity or constant strain rate (CSR) [11,12]. The strain rate sensitivity exponent (m) is an important parameter to represent the superplastic performance of materials. The greater the *m* value, the better the superplasticity of the material[13,14]. However, the maximum *m* value cannot remain during the deformation under the condition of constant velocity or CSR. Therefore, it is crucial to maintain the maximum *m* value during the deformation. A new SPD method based on variable *m* value is proposed: the measurement of *m* value and concurrent control of strain rate are simultaneously conducted, so the variation trend of both parameters can be identified. By adjusting the strain rate, the *m* value can be changed cyclically to obtain the maximum *m*

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value during SPD process. Based on the identified variation trend of m value and its maximum value in the whole cyclic process, the optimum superplastic properties of the material can be obtained. In this research, the superplastic behavior and its influence factors of TA15 alloy were investigated by the variable m method, and the SPD mechanism of the alloy was also discussed.

1 Experiment

Generally, during SPD, the relationship between the flow stress and the strain rate can be often described by Backoften equation[15], as follows:

$$\sigma = K\dot{\varepsilon}^m \quad (1)$$

where σ is the flow stress, K is a material constant, $\dot{\varepsilon}$ is the strain rate, and m is the strain rate sensitivity exponent. In the study, the m values were calculated by the velocity jump method. Based on Eq.(1), m value could be derived as follows:

$$m_i=\frac{\lg(F_{i+1}/F_i)}{\lg(v_{i+1}/v_i)} \quad (2)$$

where F_i is the instantaneous load when the velocity is v_i; i is the positive integer. During deformation, a series of F_i and v_i could be dynamically measured. Therefore, based on Eq. (2), the instantaneous m value could be calculated. The deformation velocity could be controlled during SPD to maintain the maximum m value in the entire cyclic process. When the deformation velocity is in the increasing stage, the velocity can be increased continuously if $m_i>m_{i-1}$, but the velocity stops increasing immediately and begins to decline if $m_i<m_{i-1}$. When the deformation velocity is in the decreasing stage, the velocity can be decreased continuously if $m_i>m_{i-1}$, but the velocity stops decreasing promptly and begins to increase if $m_i<m_{i-1}$. A specific computer control system was developed according to the flowchart in Fig.1.

The chemical composition of TA15 alloy is listed in Table 1. The β-transus temperature of the alloy is about 1255 K. The as-received material was processed by thermo-mechanical processing to improve the mechanical properties. The bar was forged at β phase field firstly, and then subjected to the final forging at α/β phase field. Specimens for tensile tests were wire-cut from the treated alloy with a diameter of 5 mm and a gauge length of 15 mm, as shown in Fig.2.

The superplastic tensile tests were conducted on a SANS CMT4104 electronic tensile machine and the machine velocity could be adjusted continuously in the range of 0.001~500 mm/min. The tensile testing temperatures were 1053~1223 K[16]. The tensile specimens were heated in a three-zone high temperature furnace with the temperature fluctuation of <277 K, and the specimens were homogenized for 15 min before the tests. In order to prevent the early damage or fracture due to high temperature oxidation during deformation, the specimen was coated with a thin special glass lubricant layer firstly. After deformation, the specimens were cooled rapidly to preserve the deformed microstructure by hydrocooling.

The microstructure of TA15 alloy was observed by optical microscope (OM), scanning electron microscope (SEM), and transmission electron microscope (TEM). The XJP-6 OM was used and the linear intercept procedure was employed for grain size measurement. SEM analysis was conducted on a FEI Quanta 200 SEM. The Kroll's agent (2vol% HF+4vol% HNO_3+94vol% H_2O) was used to etch the specimens for 3~5 s. TEM analysis was performed on a FEI Tecnai G2 F30 TEM. An accelerating voltage of 300 kV was used. Thin foils

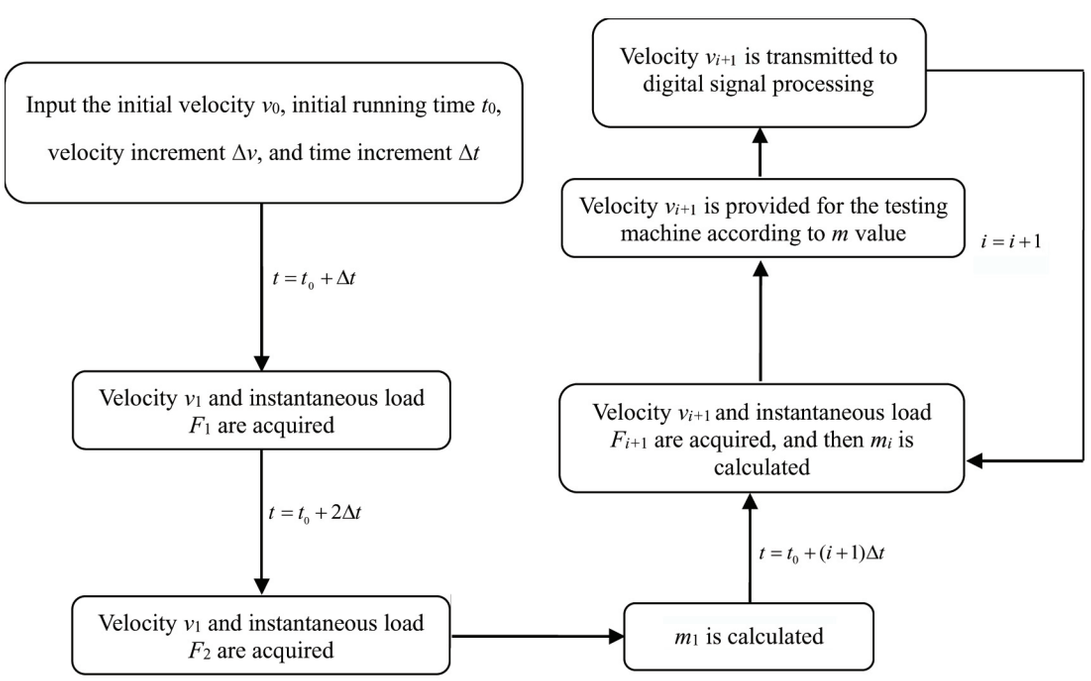

Fig.1 Calculation flowchart of variable m value during SPD

Table 1 Chemical composition of TA15 alloy (wt%)

Al	Mo	V	Zr	Si	Ti
5.5~7.0	0.5~2.0	0.8~2.5	1.5~2.5	0.15	Bal.

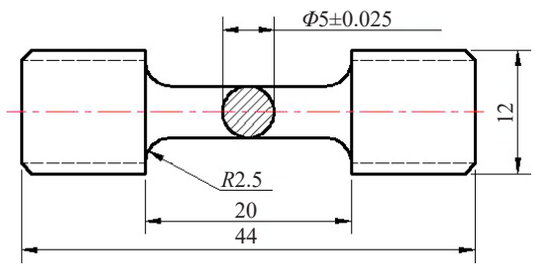

Fig.2 Schematic diagram of superplastic TA15 tensile specimen

for TEM observation were prepared by twin jet electron-polishing with an electrolytic solution of 6vol% perchloric acid, 30vol% butyl alcohol, and 64vol% methanol at ~50 V and 243 K.

2 Results

2.1 Mechanical properties

Fig. 3 shows the microstructure of the as-received and processed specimens. According to Fig. 3a, the initial microstructure is extremely inhomogeneous, consisting of the coarse strip α phase and a small amount of β phase. As shown in Fig. 3b, after thermo-mechanical processing, the initial coarse microstructure is refined effectively and the primary α phase is well-distributed in β_T matrix. The volume fraction of primary α phase increases to ~95% and the average grain size is ~2 μm. Additionally, Fig. 3c indicates that the refined α-grains with high dislocation density still have the equiaxed shape.

The tensile flow stress-true strain curves of TA15 alloys are shown in Fig. 4a. It is seen that the strain hardening occurs at the beginning of deformation due to rapid dislocation multiplication. The lower the temperature, the more serious the strain hardening, which results in the quick increase in flow stress. The strain hardening is weakened gradually with increasing the deformation temperature. Therefore, the maximum value of flow stress is 105 MPa at 1053 K, whereas the peak value of flow stress is only 15 MPa at 1223 K. Besides, all the curves exhibit a dynamic strain softening phenomenon after the peak stress with further increasing the strain, which becomes more obvious at 1053 and 1073 K. Therefore, the flow stress decreases quickly until fracture. At 1053~1223 K, the strain softening effect is obviously stronger than the strain hardening effect, which hinders SPD of the alloys. Additionally, the curves have a relatively steady trend under low stress at 1123~1223 K, which is a typical characteristic of SPD.

Fig. 4b shows the flow stress-true strain curves under CSR deformation[17], which is quite different from those based on variable m value method. Firstly, the flow stress-true strain curves obtained by variable m value method are not smooth and the fluctuation is obvious, especially those at the low temperatures, as shown in Fig. 4a. During the whole deformation process, the velocity of the tensile testing machine was continuously adjusted to obtain the maximum m value, resulting in fluctuation of the stress-strain curves. Secondly, the strain hardening occurs again at the late deformation stage. Based on the variable m value method, the deformation time is longer due to the lower strain rate ($\leqslant 10^{-4}$ s^{-1}), which is beneficial to full dynamic recrystallization of TA15 alloys. Thus, the grains grow extensively. Simultaneously, the dislocations are piled up against the grain boundaries due to grain growth, resulting in the stress concentration. However, the flow stress is not sufficient to initiate the intragranular dislocation glide, thereby hindering the grain boundary glide. The flow stress of TA15 alloys at different temperatures in Fig. 4a increases quickly to the peak value and then decreases rapidly.

Temperature is an important influence factor for SPD. Fig. 5 shows the effect of temperature on the elongation of TA15 alloys. The superplastic behavior of TA15 alloys can be obtained based on variable m value method, whereas the elongation obtained by CSR method[17] is barely satisfactory. Even at the relatively low temperature of 1053 K, the specimen demonstrates a superplastic elongation of 860%. It can also be seen that the elongation is increased noticeably with increasing the temperature. The maximum elongation of 1922% is obtained at 1173 K, and then the elongation is

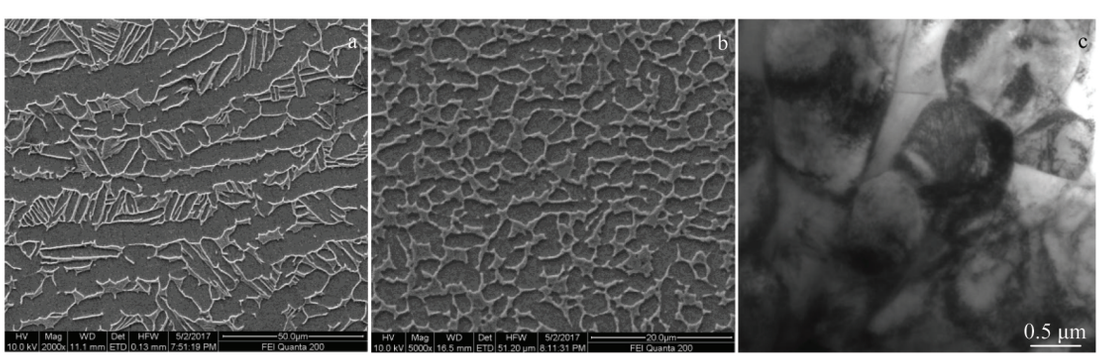

Fig.3 SEM (a, b) and TEM (c) microstructures of as-received (a) and processed specimens (b, c)

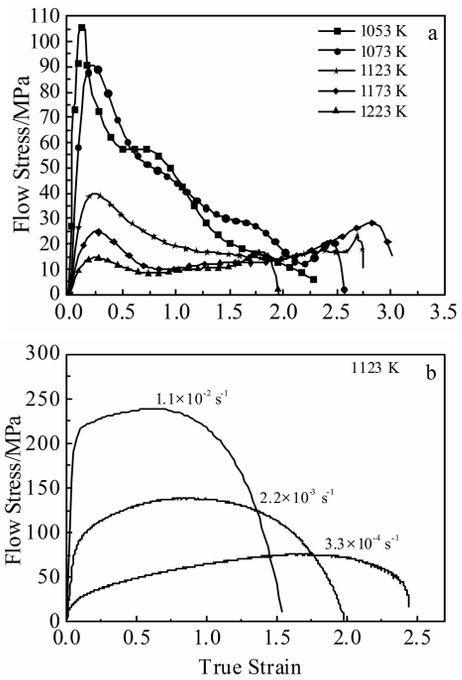

Fig.4 Flow stress-true strain curves based on variable *m* value method (a) and CSR method[17] (b)

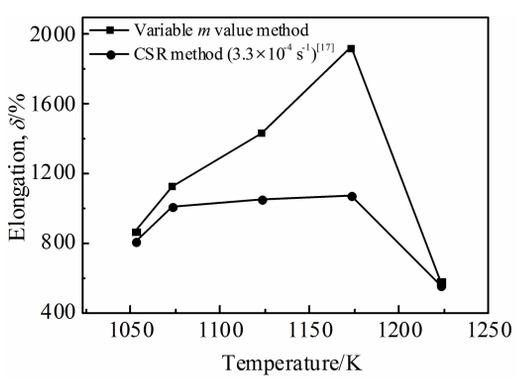

Fig.5 Effect of temperature on elongation of TA15 alloys

decreased with further increasing the temperature. It is known that SPD is realized by grain boundary glide, intragranular dislocation motion, and dislocation diffusion, among which the grain boundary glide is the main deformation mechanism[18,19]. With increasing the temperature, the atom free energy of TA15 alloys is increased greatly and the critical resolved shear stress is decreased. Additionally, the grain boundary becomes increasingly unstable and its viscosity is decreased gradually. Therefore, the grain boundary glide and diffusion are enhanced and the superplasticity is improved. With continuously increasing the temperature, the grain boundary binding force declines greatly. When the deformation temperature exceeds the β-transus temperature, the phase transformation occurs, leading to unbalanced proportion between α and β phases. Thus, the superplasticity declines sharply and the elongation is only 580% at 1223 K. Fig. 6 shows specimen appearances after SPD at different temperatures, indicating that the TA15 alloys show superplastic elongation until failure. In addition, the deformation of specimen with higher elongation is more uniform, which is in agreement with the steady flow state.

2.2 Strain rate sensitivity exponent *m*

Due to the similarity between creep and SPD, the equation describing the creep relationship between the strain rate and the flow stress is usually used to describe the steady state flow deformation during SPD[20]:

$$\dot{\varepsilon} = A\sigma^n \exp\left(-\frac{Q}{RT}\right) \tag{3}$$

where $\dot{\varepsilon}$ is the strain rate (s^{-1}); σ is the flow stress (MPa); n is the stress exponent, which is the inverse of the strain rate sensitivity exponent m; A is the constant independent of temperature; Q is the activation energy of deformation ($kJ \cdot mol^{-1}$); R is the gas constant ($kJ \cdot mol^{-1} \cdot K^{-1}$); T is the absolute temperature (K).

The strain rate sensitivity exponent m can be obtained, as follows:

$$m = \frac{\partial \ln \sigma}{\partial \ln \dot{\varepsilon}} \tag{4}$$

According to Eq. (4), the m values can be obtained by the software during SPD. By fitting the data, the relationship curves between the strain rate sensitivity exponent m and the strain rate $\dot{\varepsilon}$ are shown in Fig. 7, presenting the similar bell-cover shapes, which is the typical superplastic characteristic of materials[21]. After the m value reaches a peak, it declines with further increasing the strain rate. Under the same strain rate, the m value is increased with the increasing temperature from 1053 K to 1173 K. When the temperature is 1223 K, the m value declines. In general, the effect trends of temperature on the m value and the elongation are similar. Besides, the obtained m values are basically greater than 0.3. The maximum m value is 0.61 and 0.37 at 1173 and 1053 K, respectively. Compared with the m values obtained by CSR method[17], the m values in this research are improved.

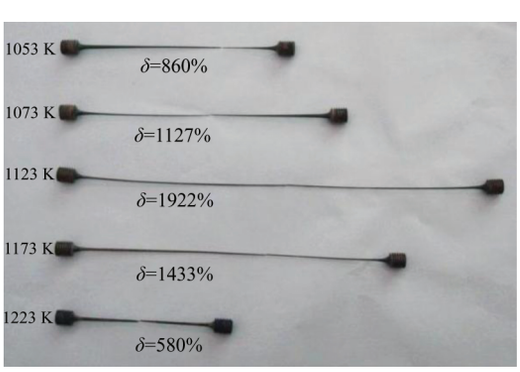

Fig.6 Appearances of specimens after SPD at different temperatures

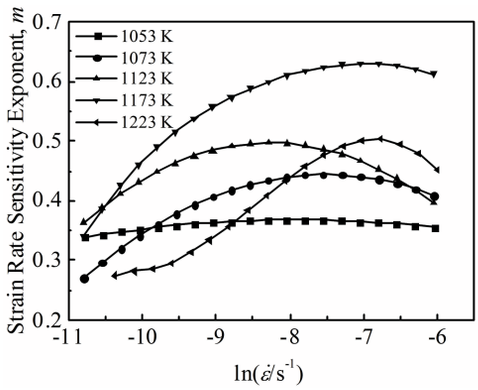

Fig.7 Relationship of m-ln$\dot{\varepsilon}$ at different temperatures

2.3 Microstructure evolution

Fig. 8 shows the microstructure of specimens after SPD at different temperatures. The microstructures of the grip part are coarsened after SPD, whereas the overall grip part is not deformed. The microstructure is coarsened in various degrees with increasing the temperature. After SPD at 1053 and 1073 K, the partial α phases are mutually merged and grow due to static recrystallization, leading to slight growth of α grain. After SPD at temperatures above 1073 K, lots of equiaxed α phases begin to grow together into the larger lamellar α phases and the boundaries of α phases are curved, as shown in Fig. 8c and 8d. However, when the TA15 alloy is deformed at 1223 K, the $\alpha \rightarrow \beta$ phase transformation occurs. Thus, the volume fraction of primary equiaxed α phases is decreased sharply and β phases are also merged mutually and grow, as shown in Fig.8e.

The grain growth in grip part is caused by thermal effects, whereas that in the near fracture part is mainly caused by SPD, namely dynamic grain growth (DGG)[22]. As shown in Fig. 8f~8h, the partial α grains are mutually merged and grow when the TA15 alloy is deformed at temperatures below 1173 K. Meanwhile, many small equiaxed α grains appear in β_T matrix. In the entire deformation process, the occurrence of dynamic recrystallization in the deformed α grains is promoted by the accumulated distortion energy, which results in the precipitation of fine equiaxed α grains in β_T matrix. The microstructure of the near fracture part is coarsened seriously with increasing the temperature. As shown in Fig. 8i, lots of α phases grow together into the coarser lamellar α phases at 1173 K. Besides, when the temperature is elevated to 1223 K, the volume fraction of primary β phases in the near fracture part is increased significantly because the $\alpha \rightarrow \beta$ phase transformation occurs, as shown in Fig. 8j. The maximum m value reaches 0.49 at 1223 K, but the superplasticity of the TA15 alloy is decreased sharply due to inappropriate proportion between α and β phases. As shown in Fig. 8e and 8j, the β grain boundary is very clear and β grains grow together into larger grains. Simultaneously, the acicular α phases appear in the β phase because the deformed specimens are cooled quickly by water.

Fig.9 shows TEM images of the specimens before and after SPD at 1173 K. Few dislocations in α grains or at grain boundaries can be observed after SPD. During SPD, the grain boundary glide is accommodated by annihilation of the dislocations. With increasing the temperature and strain, the dislocations gradually disappear. As shown in Fig.9b and 9c, α grains are coarsened obviously, whereas the grains still keep the equiaxed shape and α/α grain boundaries become curved, which are the typical SPD characteristics[23]. The dynamically recrystallized grain can also be observed at the triple junction in Fig.9c.

3 Discussion

The results demonstrate that TA15 alloys exhibit high elongation by the variable m value method for the following reasons. Firstly, the fine grains are obtained by the thermo-mechanical processing. The fine grains and more grain boundaries are favorable for grain boundary glide, thereby improving the superplasticity of alloys. Secondly, in order to achieve the maximum m value, the alloy is deformed under the proper strain rate by adjusting the velocity of the tensile machine. In addition, it is well known that grain boundary glide is usually accompanied by other processes to maintain the integrity of the grains and to suppress the fracture of specimen.

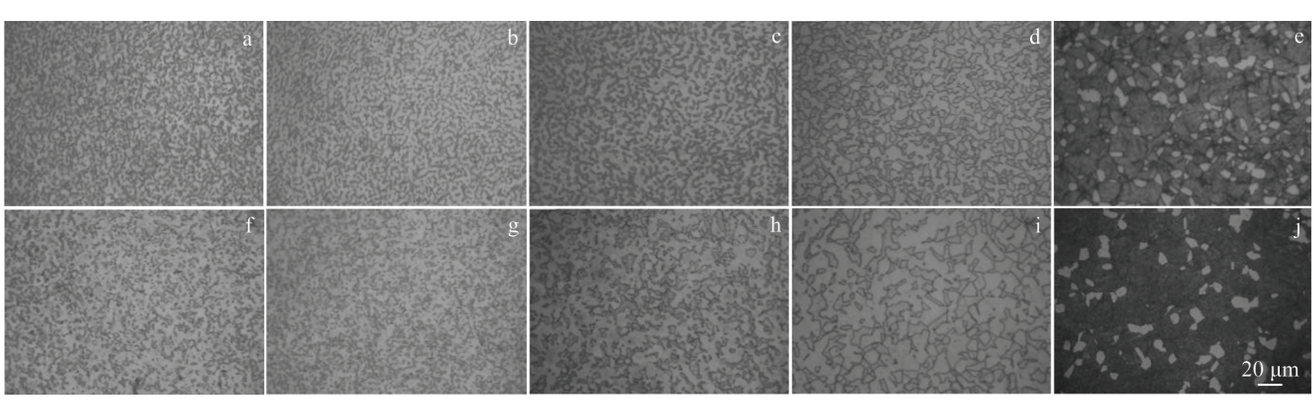

Fig.8 Microstructures of TA15 alloys after SPD at 1053 K (a, f), 1073 K (b, g), 1123 K (c, h), 1173 K (d, i) and 1223 K (e, j): (a~e) grip part and (f~j) near fracture part

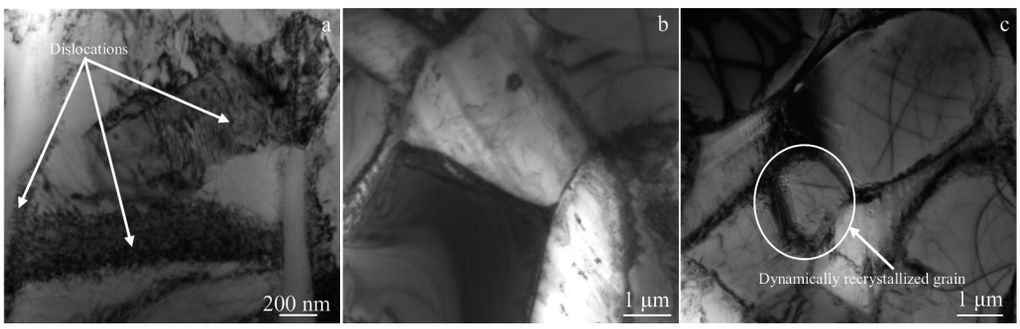

Fig.9 TEM images of TA15 alloys before (a) and after (b, c) SPD at 1173 K

As shown in Fig. 10 and Fig. 11, it takes a long time to complete SPD and the microstructure is coarsened seriously. Meanwhile, the coarsened microstructure shows that the grain boundary diffusion and migration are very active during SPD. Therefore, some characteristics of microstructure evolution are in accordance with the classical diffusion creep model proposed by Coble and Nabarro-Herring[24]. However, the fact that the grains still keep the equiaxed shape cannot be explained. Moreover, according to the classical diffusion creep model, the theoretical m value should be 1, which is not consistent with the experiment results. Thus, the mechanism of SPD based on variable m value cannot be thoroughly explained by the classical diffusion creep theory.

Some superplastic characteristics of TA15 alloys can also be explained by the grain boundary glide with diffusion creep[25] through the Ashby-Verrall model for SPD (Fig. 12). After thermo-mechanical processing, the grain size of TA15 alloy is refined greatly, so the total grain boundary area becomes larger. According to the mechanism of the diffusion creep, the grain boundary becomes the vacancy source. During SPD, lots of vacancies are produced at grain boundaries when the grain boundary glide and grain rotation occur, leading to a sharp increase in vacancy. Also, the supersaturation degree of vacancy concentration is higher, which greatly accelerates the diffusion process in TA15 alloys. Therefore, the high temperature and abundant grain boundaries provide the beneficial conditions for vacancy diffusion of TA15 alloys. Under the conditions of low strain rate and long deformation duration, which are also the typical characteristics of SPD based on the variable m value method, the diffusion creep is the main influence factor of grain boundary glide.

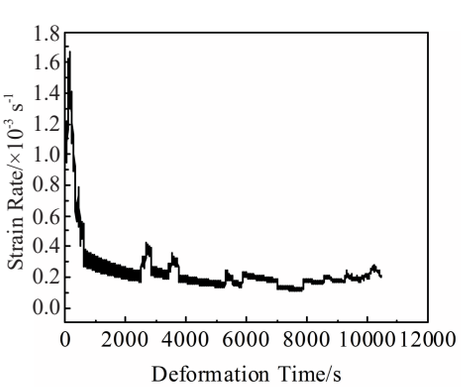

Fig.10 Relationship of strain rate-deformation time of TA15 alloys by variable m value method at 1173 K

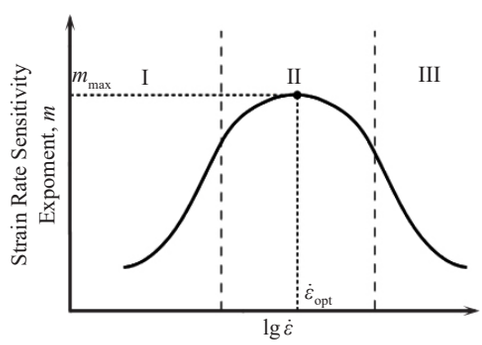

Fig.11 Typical relationship of m-lg$\dot{\varepsilon}$

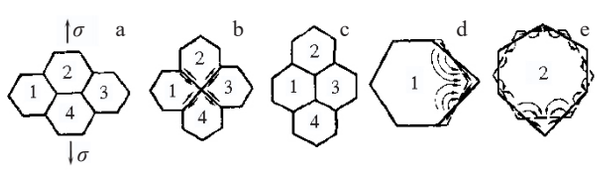

Fig.12 Ashby-Verrall model for SPD at different stages[25]: (a) initial state; (b) intermediate state; (c) final state; (d) intracrystalline diffusion; (e) grain boundary diffusion

Additionally, the intergranular dislocations play an important role in SPD process. The non-intrinsic grain boundary dislocations are formed by the interaction between the intragranular dislocations and grain boundaries, which promotes the grain boundary glide. However, the flow stress is very small due to the low strain rate. Therefore, the Frank-Read dislocation in the interior grains is difficult to initiate, which results in small dislocation density of the alloys. Due to insufficient dislocation motions in the interior grains, the non-intrinsic grain boundary dislocations caused by interaction between intergranular dislocations and grain boundaries are deficient, thereby hindering the grain boundary glide. Thus, the diffusion creep becomes the main coordination mechanism of grain boundary glide. Therefore, it can be inferred that the

dominant mechanism of SPD is the grain boundary glide accompanied by diffusion creep in TA15 alloys.

4 Conclusions

1) The strain rate sensitivity exponent *m* of TA15 alloys based on the variable *m* value method is basically greater than 0.3 and the elongation of TA15 alloys is 580%~1922% after superplastic deformation at 1053~1223 K.

2) With increasing the deformation temperature, the microstructure is coarsened gradually, but the grains still keep the equiaxed shape during deformation. The $\alpha \rightarrow \beta$ phase transformation occurs at 1223 K, leading to sharp decrease in superplastic properties.

3) It can be inferred that the grain boundary glide accompanied by diffusion creep in TA15 alloys is the main mechanism of superplastic deformation.

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TA15合金基于变*m*值法超塑性研究

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摘　要：基于变应变速率敏感性指数*m*值的方法对TA15合金超塑性进行了研究，在1053~1223 K温度范围内进行了超塑性拉伸实验。结果表明：TA15合金的延伸率为580%~1922%。微观组织分析表明合金变形过程中晶粒随温度升高而逐渐长大，但仍保持等轴状，在1223 K时发生$\alpha \rightarrow \beta$相转变，超塑性能严重下降。与恒应变速率法相比较，该方法大幅度提高了TA15合金的超塑性能。此外，超塑性变形过程中，力学性能和微观组织演变特征与Ashby-Verrall模型较吻合，因此推断出TA15合金基于变*m*值法超塑性变形的主要机制是扩散蠕变协调的晶界滑移。

关键词：TA15合金；超塑性；微观组织演变；*m*值

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