Study on hot deformation behaviour and microstructure of Zr-4 alloy

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Abstract. The isothermal compression experiment of Zr-4 alloy was accomplished by using Gleeble-3500 thermomechanical simulator. The hot deformation behaviour at the strain rate of 0.001 ~10s⁻¹ and the deformation temperature of 750 ~1000 °C was researched. The characteristics of flow stress were studied. The result is that the flow stress has a great influence on the deformation temperature and the strain rate. Based on the experimental data, the strain rate sensitivity map (m) was established. Through microstructure observation, the zones at lower m value are mainly flow localization (750 °C/10s⁻¹) and kink (950 °C/10s⁻¹). The zones at higher m value are mainly spheroidization. The result is that at strain rate of 0.01~0.1s⁻¹/deformation temperature of 900~1000 °C is the suitable deformation parameter of Zr-4 alloy.

1. Introduction

Zr-4 alloy is widely used in fuel element cladding tubes, fuel element grids, vessel tubes, pressure tube structures and cladding materials in nuclear reactors because of its good hot conductivity and low absorption of hot neutrons [1-3]. Generally, the hot deformation process is indispensable while using Zr-4 alloys to manufacture cladding materials, which can affect the final quality of products such as microstructure, mechanical properties and dimensional accuracy. During the manufacturing process, hot deformation has an important influence on the deformation mechanism of Zr-4 alloy. Strain rate and deformation temperature have the greatest effect on structure and mechanical properties of materials [4,5]. Therefore, it is necessary to systematically study the hot deformation behaviour of Zr-4 alloy.

In this article, the isothermal compression experiment of Zr-4 alloy was accomplished by Gleeble-3500 thermomechanical simulator. The flow stress curve was researched. The strain rate sensitivity map was constructed. The effect of different strain rate and deformation temperature on the structure evolution of hot deformation was analysed. The process parameters of hot deformation were optimized.

2. Materials and experimental

The original structure of Zr-4 shows in Fig.1. The original structure consists mainly of α-Zr. The β grain boundaries are clear. There are a lot of α phases with different orientations inside the coarse β grains, and the α phases are relatively straight and parallel. The isothermal compression experiment of Zr-4 alloy was accomplished by Gleeble-3500 thermomechanical simulator at the temperatures of 750, 800, 850, 900, 950 and 1000 °C / the strain rates of 0.001, 0.01, 0.1, 1 and 10s⁻¹. The specimen size is
Φ8 mm×12 mm. All specimens were subjected to 70% height reduction (true strain of 1.2). The metallographic specimens were ground, polished, dried and then corroded with 15ml HF, 40ml HNO₃ and 45ml H₂O mixture. The microstructures of the specimens were characterized on a XJP-6A metallographic microscope.

Figure 1. Original structure of Zr-4 alloy

3. Analysis of flow curve

Fig. 2 is the flow curve of Zr-4 alloy at deformation temperature of 750~1000℃ / strain rate of 0.001~10s⁻¹. The flow stresses decrease with the decline of strain rate and the rise of deformation temperature. Under different factors, the decline degree of flow stress are different, and finally reach the stable values. The main reasons are that during hot compression, the work hardening made the flow stress of Zr-4 alloy increases rapidly. And when it reaches the peak stress, the work hardening and flow softening cancel out each other and reach dynamic equilibrium finally. The majority of flow curves are stable, in other words, the flow stress does not change obviously with the rise of strain when the true strain reaches a definite value.[6, 7].

At the strain rate of 10s⁻¹, the flow curves at different temperatures are wave-like, and the flow stress begins to show a continuous small range of stress oscillation, showing a discontinuous yielding phenomenon, which may be caused by kink, adiabatic shear and local plastic flow.

Figure 2. The flow curves of Zr-4 alloy: (a) T=900℃; (b) ɛ̇ =10s⁻¹

4. Analysis of strain rate sensitivity (m)

m value reflects the ability of material to resist local uneven deformation. The higher value of m, the material structure is more uniformity during the process of deformation. m value can be obtained by formula

\[ m = \frac{\partial (\ln \sigma)}{\partial (\ln \dot{\varepsilon})} \]

, and the strain rate sensitivity (m) map at strain of 1.2 was shown in Fig. 3.

According to the map, at the temperature of 750~825℃ / the strain rate of 0.01~0.1s⁻¹, the m value is higher than 0.23. At the temperature of 900~1000℃ / the strain rate of 0.01~0.1s⁻¹, the m value is higher than 0.25. The m value peak zone often corresponds to the favourable deformation mechanism, for example spheroidization, recrystallization and superplasticity. In other temperature and strain rate
zones, $m$ value is relatively low. During the process of deformation, the zones with higher $m$ value can be selected first.

![Image of strain rate sensitivity map](image)

**Figure 3.** The strain rate sensitivity map of Zr-4 alloy at strain of 1.2.

4.1 Microstructure analysis of characterization zone

At the deformation temperature of 750°C / the strain rate of 10s$^{-1}$ in Fig.4 (a), the structure was elongated to be fibrous and uneven. A flow localization phenomenon occurs in structure. Because of the high strain rate, short deformation time and low deformation temperature, the specimen will occur obvious hot effect in the process of deformation, resulting in flow localization phenomena. The appearance of flow localization means that the structure has serious non-uniform deformation. $M$ value in this zone is also relatively low. At the deformation temperature of 950°C/ the strain rate of 10s$^{-1}$ in Fig.4 (a), the non-uniform size of the laths and different degrees of kink occur in the structure, which is mainly on account of the phase transformation of the alloy from high temperature and rapid cooling. In this zone, the structure is prone to instability during processing. The value of $m$ in this zone is also relatively low. These zones should not be considered during the processing.

At the deformation temperature of 800°C / the strain rate of 0.01s$^{-1}$. During the cooling process from heating to deformation temperature, the grain size in the structure hardly changes. It can be seen that the phenomenon of spheroidization has begun to appear in the structure. At the strain rate of 0.1s$^{-1}$/the deformation temperature of 950°C in the Fig.5 (b). It can be found that the original structure has changed. A large number of spheroidization phenomena occur at the grain boundary. This is mainly due to the high temperature and high driving force, the original lamellar structure is broken, resulting in spheroidization. In the corresponding region, the value of $m$ is the largest and the structure is relatively uniformity.

To sum up, the result is that at strain rate of 0.01~0.1s$^{-1}$/deformation temperature of 900~1000°C is the suitable deformation parameter of Zr-4 alloy.

![Image of microstructures](image)

**Figure 4.** Microstructures of Zr-4 alloy under different hot deformation parameters 750°C/10s$^{-1}$ (a) and 950°C/10s$^{-1}$ (b)
5. Conclusions

(1) The flow stress of the alloy has a great influence on the deformation temperature and the strain rate. Under different conditions, the decline degree of flow stress curve is different, and the flow stress finally reaches a stable value. When the strain rate is 10s⁻¹, the true stress-true strain curves at different temperatures are wave-like.

(2) The strain rate sensitivity map of Zr-4 at a strain of 1.2 was obtained. The result is that at strain rate of 0.01~0.1s⁻¹/deformation temperature of 900~1000°C is the suitable deformation parameter of Zr-4 alloy. The deformation mechanism is spheroidization.

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References

[1] Derek O. Northwood, The development and applications of zirconium alloys, Mater. Des. 6(2) (1985)58-70.
[2] S.J. Zinkle, G.S. Was, Materials challenges in nuclear energy, Acta Mater. 61 (3) (2013) 735-758.
[3] S. K. Jha, N. Keskar, K. I. V. Narayan, K. V. M. Krishna, D. Srivastava, G. K. Dey, N. Saibaba, Microstructural and textural evolution during hot deformation of dilute Zr-Sn alloy, J. Nucl. Mater. 482(2016)12-18.
[4] R. Kapoor, A. Sarkar, J. Singh, I. Samajdar, D. Raabe, Effect of strain rate on twinning in a Zr alloy, Scripta Mater. 74 (2014) 72-75.
[5] G. C. Kaschner, C. N. Tomé, I. J. Beyerlein, S. C. Vogel, D. W. Brown, R. J. McCabe, Role of twinning in the hardening response of zirconium during temperature reloads, Acta Mater. 54(11) (2006)2887-2896.
[6] Y. Yang, X. D. Peng, F. J. Ren, H. M. Wen, J. F. Su, W. D. Xie, Constitutive modeling and hot deformation behavior of duplex structured Mg-Li-Al-Sr Alloy, J. Mater. Sci. Technol. 32 (12)(2016) 1289-1296.
[7] Q. H. Zang, H. S. Yu, Y. S. Lee, M. S. Kim, H. W. Kim, Effects of initial microstructure on hot deformation behaviour of Al-7.9Zn-2.7Mg-2.0Cu (wt%) alloy, Mater. Charact. 151 (2019) 404-413.

Figure 5. Microstructures of Zr-4 alloy under different deformation parameters 800°C/0.01s⁻¹ (a) 950°C / 0.1s⁻¹ (b)