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Microstructure and creep behavior of the extruded Mg–4Y–4Sm–0.5Zr alloy

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ABSTRACT

The microstructure and creep behavior of the extruded Mg–4Y–4Sm–0.5Zr alloy were investigated. It was shown that dynamic recrystallization (DRX) took place during extrusion at 673 K and equilibrium β precipitates were observed in the extruded alloys. Numerous fine globular β′ precipitates homogeneously formed within grains when the as-extruded alloy was aged at 473 K for 16 h, leading to effectively strengthening. The as-extruded alloy exhibits good creep resistance. The creep stress exponent n is 4.42 and the creep activation energy Q is 140.6 kJ mol^{-1} suggesting that dislocation climb plays a dominant role when the alloy is crept at temperatures ranging from 453 K to 493 K and at a stress of 180 MPa.

1. Introduction

There has been a rapidly growing interest in the development of high-strength, creep-resistant magnesium alloys for the automotive and aerospace industries [1]. But the use of magnesium alloys has been limited in the past owing to their poor mechanical properties and low formability [2].

It has been demonstrated that rare earth metals (RE) are the most effective elements to improve the strength properties of magnesium especially at elevated temperatures [3,4]. A recently developed Mg–4Y–4Sm–0.5Zr alloy was shown to exhibit promising mechanical properties [5]. The microstructure evolution and mechanical properties of the as-cast Mg–4Y–4Sm–0.5Zr alloy during heat treatments were further investigated [6,7].

Generally, wrought magnesium alloys have superior mechanical properties compared to cast magnesium alloys, due to such mechanisms as the refinement of grains, the elimination of casting defects and to the homogenization of the microstructure during the plastic processing processes [8]. However, the microstructure and mechanical properties, especially the high temperature creep resistance of the wrought Mg–4Y–4Sm–0.5Zr alloy were few investigated.

The aim of the present work is to investigate the microstructure and mechanical properties of the extruded Mg–4Y–4Sm–0.5Zr alloy. Furthermore, the creep behavior of this alloy will be presented.

2. Experimental method

The material used in this investigation is an extruded Mg–4Y–4Sm–0.5Zr alloy. The alloy was homogenized at 798 K for 8 h before extrusion. Details of the cast and homogenization procedure were described in an earlier publication [6]. The homogenized Mg–4Y–4Sm–0.5Zr alloy was extruded at 673 K with an extrusion ratio of 45:1. The extruded plates of 42 mm width and 6 mm thickness were air-cooled to ambient temperature. After extrusion, some of the specimens were isothermally aged according to the optimized parameters at 473 K for 16 h in oil-bath [6].

Tensile creep tests under constant stresses were conducted on the as-extruded and extruded-plus-aged materials with the stress axis parallel to the extrusion direction. Samples were tested in air at temperatures of 453 K, 473 K, 493 K and 523 K and at stresses ranging from 60 MPa to 220 MPa.

The room temperature tensile properties were tested by a Zwick/Roell testing machine with the tensile axis parallel to the extrusion direction.

The microstructures of the alloy were examined using a LEICA MEF4M optical microscope and a JEOL-3010 transmission electron microscope (TEM) operating at 300 kV. The average grain size was measured by the linear intercept method.

3. Results and discussion

3.1. Microstructure evolution

The microstructure of the homogenized Mg–4Y–4Sm–0.5Zr alloy is shown in Fig. 1. Almost all the eutectic phases were

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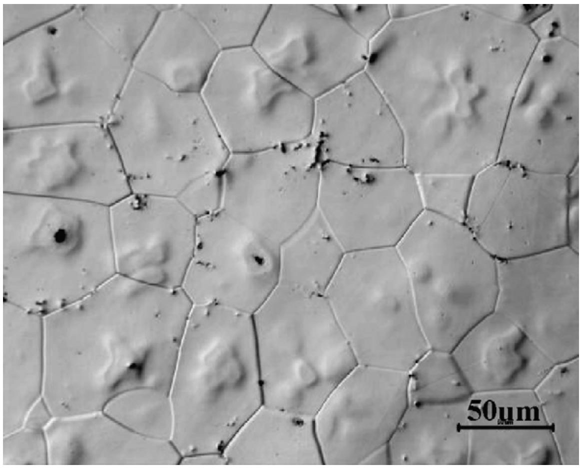

Fig. 1. Optical micrograph of the homogenized Mg–4Y–4Sm–0.5Zr alloy.

solutionized into the Mg matrix, and the average grain size is about 50 μm. However, a small amount of quadrate phases with a FCC crystal structure (a = 0.5581 nm) still remained at and near the grain-boundaries [5].

The microstructure of the as-extruded Mg–4Y–4Sm–0.5Zr alloy is shown in Fig. 2 (a) and consists of fine and equiaxed grains with a mean grain size of approximately 17 μm, suggesting that dynamic recrystallization (DRX) took place during the extrusion process at 673 K. However, the grain size is not homogeneous and bands of small and larger grains are observed parallel to the extrusion direction with precipitates preferentially located in the bands where small grains are present. Fig. 2(b) shows a TEM image of the precipitates in the as-extruded alloy. These precipitates are about 0.5–1 μm in diameter. The inserted diffraction patterns indicate that these precipitates are the equilibrium β phase with an fcc crystal structure (a = 2.246 nm). It is suspected that these precipitates were generated during extrusion process.

A selected area electron diffraction (SAED) pattern and a typical high-resolution transmission electron microscopy (HRTEM) image for the as-extruded alloy aged for 16 h at 473 K are shown in Fig. 3(a) and (b) with the electron beam parallel to the $[0\,0\,0\,1]_a$. The SAED pattern shown in Fig. 3(a), is identical to that of the β′ phase observed in the as-cast Mg–4Y–4Sm–0.5Zr alloy [5]. This intermediate phase β′ has a base-centered orthorhombic structure (a = 0.640 nm, b = 2.223 nm, c = 0.521 nm) [9]. The HREM image in Fig. 3(b) reveals that the β′ precipitates have a globular section in the basal Mg-plane. These so-called globular precipitates are approximately 10 nm. They are homogeneously distributed within grains.

3.2. Tensile properties

The tensile mechanical properties at room temperature are shown in Fig. 4 for the homogenized, as-extruded and extruded-plus-aged Mg–4Y–4Sm–0.5Zr alloy. The as-extruded alloy shows higher yield strength (YS) and ultimate tensile strength (UTS) than the homogenized alloy. This is most probably due to the grain refinement produced by extrusion. However, it is somewhat surprising that the elongation (EL) of the as-extruded alloy decreases despite of the grain refinement. This is probably due to the dynamic precipitation during the extrusion process and the inhomogeneous microstructure of the as-extruded alloy. After an ageing heat treatment, the YS and UTS sharply increase further due to the formation of the β′ precipitates (as shown in Fig. 3(b)) which are very efficient to strengthen the alloy. The extruded Mg–4Y–4Sm–0.5Zr alloy

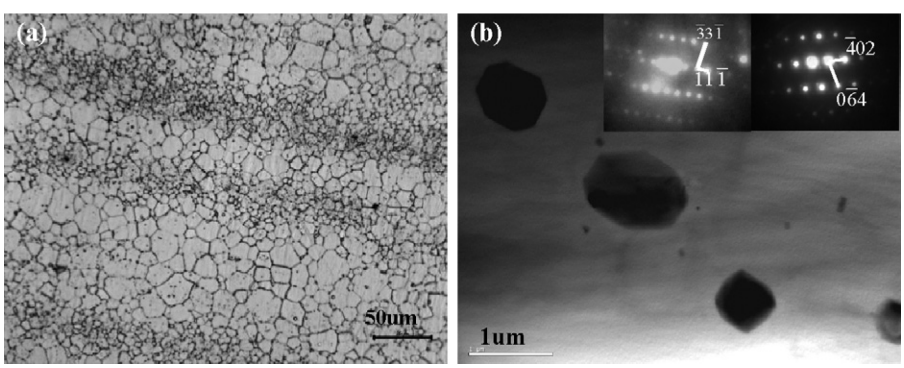

Fig. 2. Microstructure of the as-extruded Mg–4Y–4Sm–0.5Zr alloy. (a) Optical micrograph. (b) TEM image of the precipitates in the as-extruded alloy. The inserts show the diffraction patterns corresponding to the precipitates.

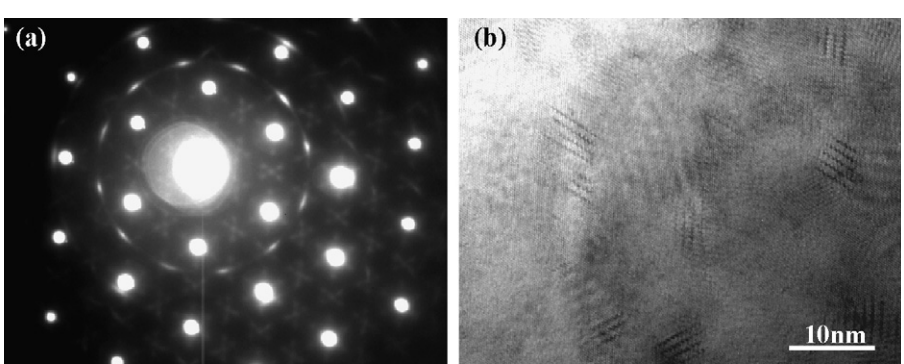

Fig. 3. SAED pattern and HRTEM image of precipitates within the grains in the extruded Mg–4Y–4Sm–0.5Zr alloy after ageing at 473 K for 16 h, beam direction: $[0\,0\,0\,1]_a$: (a) SAED pattern; (b) HRTEM image.

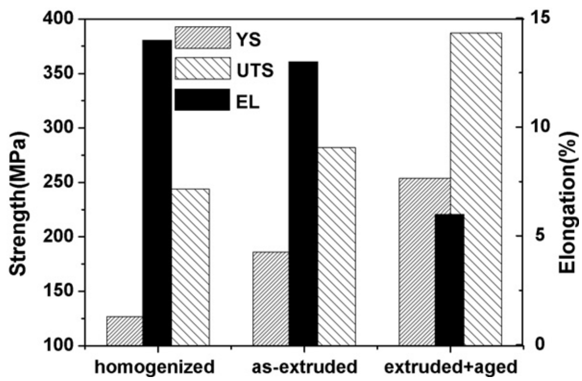

Fig. 4. Tensile properties of the Mg–4Y–4Sm–0.5Zr alloy in different states.

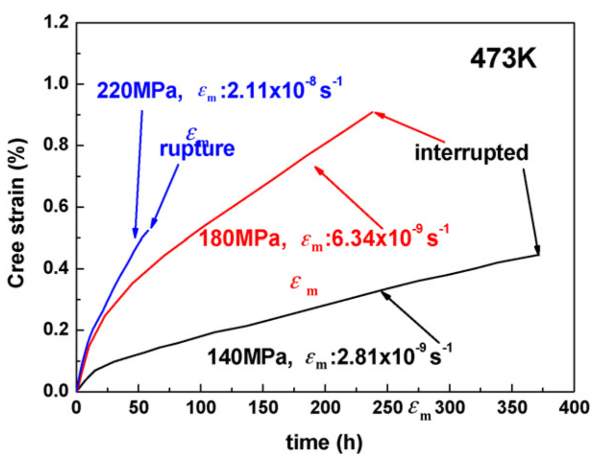

Fig. 5. Creep curves of the as-extruded Mg–4Y–4Sm–0.5Zr alloy at 473 K and at different stresses from 140 MPa to 220 MPa.

after the ageing heat treatment shows thus the highest UTS which is equal to 378 MPa.

3.3. Creep behavior

The creep curves of the as-extruded alloy crept at 473 K and at stresses of 140 MPa, 180 MPa and 220 MPa are shown in Fig. 5. The steady state creep rates ($\dot{\varepsilon}_m$) of the alloy at the various stresses are also presented in Fig. 5. $\dot{\varepsilon}_m$ normally increases with increasing creep stresses. The alloy exhibits good creep resistance up to 180 MPa. At 220 MPa, however, the sample fractured after 56 h creep but at a small strain of 0.52%. To characterize the creep behavior, the relationship between the steady state creep rates and the applied stresses is plotted in a double logarithmic plot as shown in Fig. 6. The slope of the curve which corresponds to the creep stress exponent n is constant and equal to 4.42 at 473 K.

The creep curves and the steady state creep rates of the alloy crept at stress of 180 MPa and at creep temperatures of 453 K, 473 K and 493 K are shown in Fig. 7. The steady state creep rates increase with increasing creep temperatures. The alloy exhibits good creep resistance up to 473 K, but the creep resistance deteriorates when the alloy is crept at 493 K.

The creep activation energy can be obtained from the temperature dependence of the minimum creep rate:

$$\dot{\varepsilon} = A\sigma^n exp\left(\frac{-Q}{RT}\right) \tag{1}$$

where $\dot{\varepsilon}$ is the steady state creep rate, A a constant, σ the creep stress, n the stress exponent, Q the activation energy, R the gas constant and

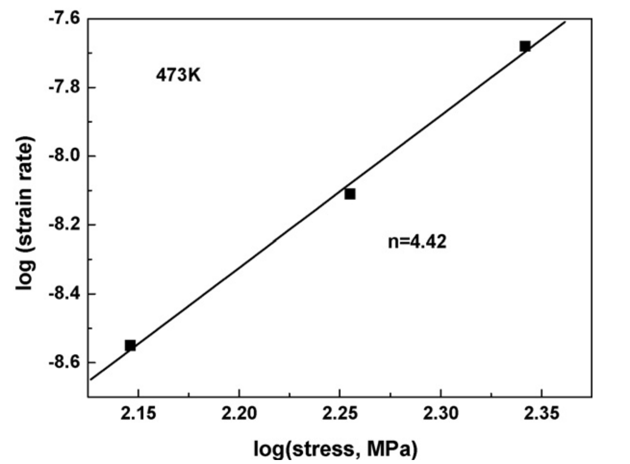

Fig. 6. log(strain rate)–log(stress) curve of the as-extruded Mg–4Y–4Sm–0.5Zr alloy crept at 473 K.

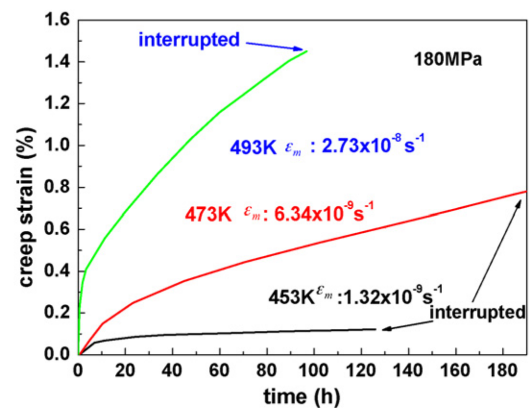

Fig. 7. Creep curves of the as-extruded Mg–4Y–4Sm–0.5Zr alloy at 180 MPa and at different temperatures from 453 K to 493 K.

T is the temperature. Based on Eq. (1), the creep activation energy Q can be calculated at constant stress by the following equation:

$$Q = \left.\frac{\partial \ln \dot{\varepsilon}}{\partial(-1/RT)}\right|_{\sigma = \text{const.}} \tag{2}$$

The corresponding plot representing the variation of $\ln(\dot{\varepsilon})$ as a function of $1/T$ is shown in Fig. 8. The creep activation energy for the as-extruded Mg–4Y–4Sm–0.5Zr alloy at the stress of 180 MPa is

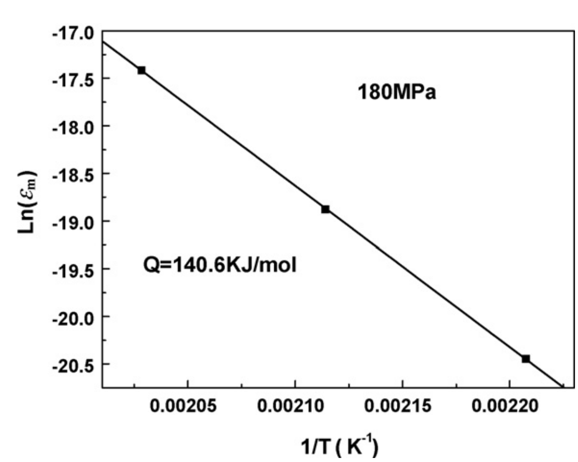

Fig. 8. $\ln(\dot{\varepsilon})$–$(1/T)$ curve of the as-extruded Mg–4Y–4Sm–0.5Zr alloy crept at 180 MPa.

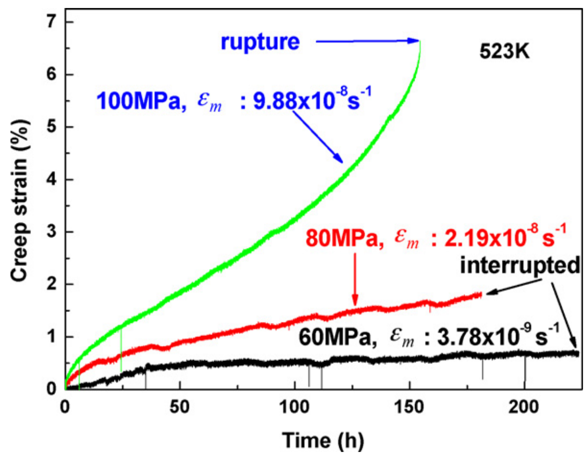

Fig. 9. Creep curves of the as-extruded Mg–4Y–4Sm–0.5Zr alloy at 523 K and at different stresses from 60 MPa to 100 MPa.

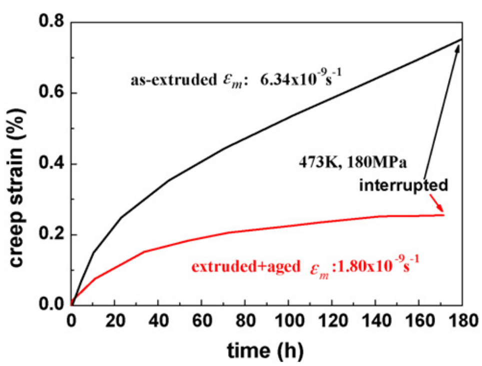

Fig. 10. Creep curves of the as-extruded and extruded-plus-aged Mg–4Y–4Sm–0.5Zr alloy at 473 K and 180 MPa.

equal to 140.6 kJ mol^{-1}, which is very close to the activation energy for the lattice self diffusion of magnesium (135 kJ mol^{-1}) [10].

Both the values of the creep stress exponent n and the creep activation energy Q suggest that dislocation climb plays a dominant role when the alloy is crept at temperatures ranging from 453 K to 493 K and at the stress of 180 MPa.

Other creep experiments were carried out on the as-extruded alloy at 523 K for stresses equal to 60 MPa, 80 MPa and 100 MPa. The corresponding creep curves are shown in Fig. 9 together with the values of the steady state creep rates. The creep resistance sharply deteriorates when the alloy is crept at 523 K particularly at a stress of 100 MPa. This is probably related to the activation of non-basal slip systems above 473 K and the fact that the precipitates formed during creep above 473 K are much less efficient to strengthen the alloy.

Creep experiments were also carried out on the extruded-plus-aged alloy at 473 K and at 180 MPa for comparison with the as-extruded alloy. The creep curves and the values of the steady state creep rates are given in Fig. 10. It can be seen that the creep resistance of the extruded-plus-aged alloy is obviously better than that of the as-extruded alloy. The β′ precipitates formed during ageing enhance the creep resistance of the as-extruded alloy.

4. Conclusions

(1) DRX took place and equilibrium β precipitates dynamically formed during extrusion at 673 K. After ageing at 473 K for 16 h, large numbers of fine globular β′ precipitates homogeneously precipitated within the grains. These are efficient to strengthen the alloy.
(2) After extrusion and ageing, the room temperature mechanical properties of the Mg–4Y–4Sm–0.5Zr alloy evidently increase. Values of UTS of 387 MPa, of yield strength of 254 MPa and of elongation of 6% are attained in the extruded-plus-aged state.
(3) The as-extruded Mg–4Y–4Sm–0.5Zr alloy exhibits good creep resistance at 473 K and under a stress of 180 MPa. The creep stress exponent n is 4.42 and the creep activation energy Q is 140.6 kJ mol^{-1} suggesting that dislocation climb plays a dominant role when the alloy is crept at temperatures ranging from 453 K to 493 K and at a stress of 180 MPa.

Acknowledgements

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References

[1] Z.C. Li, H. Zhang, L. Liu, Y.B. Xu, Mater. Lett. 58 (2004) 3021–3024.
[2] S. Barbagallo, P. Cavaliere, E. Cerri, Mater. Sci. Eng. A 367 (2004) 9–16.
[3] L.L. Rokhlin, T.V. Dobatkina, N.I. Nikitina, Mater. Sci. Forum 419 (2003) 291–296.
[4] T. Mohri, M. Mabuchi, N. Satio, M. Nakamura, Mater. Sci. Eng. A 257 (1998) 287–294.
[5] D. Li, Q. Wang, W. Ding, Mater. Sci. Eng. A 428 (2006) 295–300.
[6] D. Li, Q. Wang, W. Ding, Mater. Sci. Eng. A 448 (2007) 165–170.
[7] D. Li, Q. Wang, W. Ding, J. Alloys Compd. 465 (2008) 119–126.
[8] H. Ding, L. Liu, S. Kamado, W. Ding, Y. Kojima, J. Alloys Compd. 456 (2008) 400–406.
[9] C. Antion, P. Donnadieu, F. Perrard, A. Deschamps, C. Tassin, A. Pisch, Acta Mater. 51 (2003) 5335–5348.
[10] J.G. Wang, L.M. Hsiung, T.G. Nieh, M. Mabuchi, Mater. Sci. Eng. A 315 (2001) 81–88.