Tensile Properties from Room Temperature to 673 K of Mg–0.9 mass%Ca Alloy Containing Lamella Mg₂Ca

Yasumasa Chino¹, Masaaki Kobata^{2,*}, Hajime Iwasaki² and Mamoru Mabuchi¹

¹*Inst. for Structural and Engineering Materials, National Institute of Advanced Industrial Science and Technology, Nagoya 463-8560, Japan*

²*Division of Materials Science and Engineering, Graduate School of Himeji Institute of Technology, Himeji 671-2201, Japan*

Tensile properties were investigated from room temperature to 673 K of a binary Mg–0.9 mass%Ca alloy where Mg₂Ca phase was dispersed as lamella. The 0.2% proof stress significantly increased by the Mg₂Ca phase in the temperature range investigated. It was suggested that strengthening by stable lamella Mg₂Ca in the grains and limitation of deformation related to grain boundaries by Mg₂Ca phase at grain boundaries contribute to improvement of high temperature strength for the Mg–Ca alloy.

(Received June 13, 2002; Accepted August 23, 2002)

Keywords: magnesium alloy, magnesium–calcium alloy, lamella structure, tensile tests, mechanical properties

1. Introduction

Magnesium alloys are promising light-weight materials because of their low densities, high specific strength, high specific elastic modulus and good recyclable ability. Therefore, magnesium alloys have potential applications to automobile parts, electric appliance cases and so on.^{1,2)} However, magnesium alloys often show a large decrease in strength at elevated temperatures and poor creep resistance. For wider applications to automobiles, it is required to improve strength and creep resistance at elevated temperatures. It is well known that addition of rare earth metals increases the high temperature strength and improves the creep resistance of magnesium alloys.^{3–6)} However, magnesium alloys containing rare earth metals are expensive.

Recently, it was reported that addition of Ca is effective for improvement of high temperature strength and creep resistance of magnesium alloys.^{7–10)} Nie and Muddle¹¹⁾ pointed out the importance of a fine distribution of Mg₂Ca precipitates caused by precipitation-hardening by aging of Mg–Ca alloys. Also, Piatti and Stoto¹²⁾ showed that superplastic behavior is attained in a Mg–Ca eutectic alloy. Thus, addition of Ca significantly improves mechanical properties of magnesium alloys.

The Mg₂Ca phase has the melting point of 988 K and is stable at elevated temperatures. Hence, improvement of high temperature strength by addition of Ca is attributed to the stable Mg₂Ca precipitates. However, Miyazaki *et al.*¹³⁾ showed that the strength of Mg–Ca based alloys decreased largely at 573 K. Further research is needed to understand mechanisms of modification of mechanical properties by addition of Ca.

In many studies related to Mg–Ca alloys,^{7–11,13)} ternary Mg alloys such as Mg–Al–Ca and Mg–Zn–Ca have been investigated. For these alloys, the precipitation behavior is too complicated to understand effects of addition of Ca on mechanical properties of Mg. Also, room temperature properties have received little attention for the Mg–Ca alloys.¹⁰⁾ In the present paper, tensile properties from room temperature

to 673 K of a binary Mg–0.9 mass%Ca alloy are investigated and their properties are compared with those of pure Mg to investigate effects of addition of Ca on mechanical properties of Mg.

2. Experimental Procedures

Mg–0.9 mass%Ca was prepared from 99.9 mass% pure Mg ingot and 99.9 mass% pure Ca grains by induction furnace melting in a Fe mould under an argon atmosphere. Any additional heat treatments were not applied to the as-ingot Mg–0.9%Ca alloy. For comparison, pure Mg was also prepared under the same processing conditions. Contamination with Fe from the mold was low level (less than 0.01 mass%) as a result of ICP emission spectrochemical analysis. Tensile specimens with a gauge length of 10 mm and a gauge diameter of 2.5 mm were machined from the central part of the as-ingots with a diameter of 30 mm and a height of 50 mm. Tensile tests were carried out from room temperature to 673 K at an initial strain rate of $1.7 \times 10^{-3} \text{ s}^{-1}$ by an Instron-type electromechanical machine. The furnace temperature was controlled within $\pm 1 \text{ K}$. Grain boundary sliding in the specimens tested at 473 K was investigated by scanning electron microscopy.

3. Results and Discussion

Microstructures of the as-ingot Mg–0.9%Ca alloy and pure Mg are shown in Fig. 1. The X-ray diffraction analysis revealed that the second phase in the Mg–Ca alloy is Mg₂Ca. The Mg₂Ca phase was dispersed as lamella in the interior of Mg grains. The lamellas in Mg grains were dispersed in groups of different discrete orientations. Also, the Mg₂Ca phase was observed near grain boundaries as well as in the grains. The Mg–Ca alloy showed the grain size of 459 μm , which was almost the same as the grain size of pure Mg (=476 μm). It is therefore demonstrated that the grain size is not affected by addition of Ca.

The variation in ultimate tensile strength as a function of testing temperature for the Mg–0.9%Ca alloy and the pure

*Graduate Student, Graduate School of Himeji Institute of Technology.

Fig. 1 Microstructures of (a) Mg-0.9%Ca alloy and (b) pure Mg.

Fig. 2 The variation in ultimate tensile strength as a function of testing temperature for Mg-0.9%Ca alloy and pure Mg.

Mg is shown in Fig. 2. At room temperature, the strength of the Mg-Ca alloy was almost the same as that of the pure Mg. It is of interest to note that the strength of the Mg-Ca alloy did not decrease up to 473 K; the strength of the pure Mg decreased with temperature. Furthermore, the strength of the Mg-Ca alloy at 473–673 K was more than twice higher than that of the pure Mg. Clearly, addition of Ca increased the high temperature strength.

The variation in 0.2% proof stress as a function of testing temperature for the Mg-0.9%Ca alloy and the pure Mg is shown in Fig. 3. The Mg-Ca alloy showed 2.0–3.7 times higher 0.2% proof stress than the pure Mg in the temperature range investigated. It should be noted that addition of Ca significantly increased the 0.2% proof stress not only at elevated temperatures, but also at room temperature.

The variation in elongation as a function of testing temperature for Mg-0.9%Ca alloy and pure Mg is shown in Fig. 4. The Mg-Ca alloy showed lower elongation than the pure Mg in the testing temperature range except 673 K. Clearly, the Mg₂Ca phase had a harmful effect on ductility. Observation of fracture surfaces of the specimens tested at room temperature revealed that intragranular fracture occurred for both the Mg-Ca alloy and the pure Mg. The Mg₂Ca phase dispersed in Mg grains were the sites for crack nucleation for the Mg-Ca alloy. However, the Mg-Ca alloy showed larger elongation

Fig. 3 The variation in 0.2% proof stress as a function of testing temperature for Mg-0.9%Ca alloy and pure Mg.

Fig. 4 The variation in elongation as a function of testing temperature for Mg-0.9%Ca alloy and pure Mg.

than the pure Mg at 673 K. To understand this phenomenon, further research is needed.

In the previous study,¹⁴⁾ Mg alloy having spherical particles less than 1 μm exhibited a large decrease in strength at 473 K. In the present investigation, however, there was not such a large decrease in strength at 473 K for the Mg-Ca alloy which contained the lamella structure in the grains. This points out importance of the lamella structure for improve-

Fig. 5 The side surfaces of the specimens tested at 473 K, (a) Mg–0.9%Ca alloy and (b) pure Mg.

ment of high temperature strength in Mg alloys. In TiAl alloys,¹⁵⁾ it is known that the lamella structure is effective for higher high-temperature.¹⁶⁾ The same mechanism may be responsible for improvement of high temperature strength in Mg alloys. Further research is needed to clarify details of the mechanisms of improvement of mechanical properties by the lamella structure in Mg alloys.

The grain boundary diffusion coefficient is large for Mg, for example, in comparison with Al,¹⁴⁾ suggesting that grain boundary sliding occurs easily for Mg based materials. When grain boundary sliding occurs, the strength largely decreases even for Mg alloys containing stable phases.¹⁷⁾ Hence, it is important to limit grain boundary sliding for high strength at elevated temperatures in Mg based materials, and it is worthwhile to investigate grain boundary sliding for understanding of mechanisms of high strength at elevated temperatures.

The side surfaces of the fractured specimens tested at 473 K for the Mg–Ca alloy and the pure Mg are shown in Fig. 5, where the sites where the diameter is about 2.4 mm are observed. The surface was bumpy for the pure Mg. The bumpy surface for the pure Mg is attributed to deformation related to grain boundaries such as grain boundary sliding or local deformation around grain boundaries. In the previous study,¹⁷⁾ grain boundary sliding was observed for Mg alloy with the large grain size of 60 μm. The present investigation revealed that deformation related to grain boundaries occurred even for Mg with the very large grain size of about 500 μm. This may be related to the large grain boundary coefficient for Mg.¹⁴⁾ However, the bumpy surface was not observed for the Mg–Ca alloy. Clearly, the deformation mechanism of the Mg–Ca alloy is different from that of the pure Mg. The Mg₂Ca phase was dispersed near grain boundaries as well as in the grains. The Mg₂Ca phase dispersed near grain boundaries hinders deformation related to grain boundaries for the Mg–Ca alloy. Therefore, it is suggested that both strengthening by the stable Mg₂Ca lamellas in the grains and limitation of deformation related to grain boundaries by the Mg₂Ca phase at grain boundaries contribute to improvement of high temperature strength for the Mg–Ca alloy.

4. Conclusions

Tensile properties from room temperature to 673 K of a Mg–0.9 mass%Ca alloy were compared with those of a pure Mg. The results are concluded as follows.

(1) The Mg₂Ca phase was dispersed as lamella in the interior of Mg grains. Also, the Mg₂Ca phase was observed at grain boundaries.

(2) The ultimate tensile strength of the Mg–Ca alloy was almost the same as that of the pure Mg at room temperature. However, the Mg–Ca alloy exhibited more than twice higher strength than the pure Mg in an elevated temperature range of 473–673 K.

(3) In addition, the Mg–Ca alloy showed 2.0–3.7 times higher 0.2% proof stress than the pure Mg.

(4) However, the Mg–Ca alloy showed lower elongation than the pure Mg in the temperature range except 673 K. The Mg₂Ca phases had a harmful effect on ductility.

(5) It was suggested that strengthening by stable lamella Mg₂Ca in the grains and limitation of deformation related to grain boundaries by Mg₂Ca phase at grain boundaries contribute to improvement of high temperature strength for the Mg–Ca alloy.

REFERENCES

- 1) E. Aghion and B. Bronfin: *Mater. Sci. Forum* **350–351** (2000) 19–28.
- 2) B. Landkof: *Magnesium Alloys and Their Applications*, ed. by K. U. Kainer (WILEY-VCH Verlag GmbH, Weinheim, 2000) pp. 168–172.
- 3) B. L. Mordike and W. Henning: *Proceedings of the International Conference on Magnesium Technology*, ed. by C. Baker (The Institute of Metals, London, 1987) pp. 54–59.
- 4) H. Karimzadeh, J. M. Worrall, R. Pilkington and G. W. Lorimer: *Proceedings of the International Conference on Magnesium Technology*, ed. by C. Baker (The Institute of Metals, London, 1987) pp. 138–141.
- 5) T. Mohri, M. Mabuchi, N. Saito and M. Nakamura: *Mater. Sci. Eng. A257* (1998) 287–294.
- 6) R. Kariya, H. Iwasaki, T. Mori, T. Mohri, M. Mabuchi, M. Nakamura, T. Asahina and K. Higashi: *Mater. Sci. Forum* **350–351** (2000) 93–96.
- 7) A. Luo and M. O. Pekguleryuz: *J. Mater. Sci.* **29** (1994) 5259–5271.
- 8) R. Ninomiya, T. Ojio and K. Kubota: *Acta Metal. Mater.* **43** (1995) 669–674.
- 9) T. Horie, H. Iwahori, Y. Awano and A. Matsui: *J. JILM* **49** (1999) 272–276.
- 10) W. Qudong, C. Wenzhou, Z. Xiaoqin, L. Yizhen, D. Wenjiang, Z. Yanping, X. Xiaoping and M. Mabuchi: *J. Mater. Sci.* **36** (2001) 3035–3040.

- 11) J. F. Nie and B. C. Muddle: *Scr. Mater.* **37** (1997) 1475–1481.
- 12) G. Piatti and T. Stoto: *J. Mater. Sci. Lett.* **3** (1984) 60–64.
- 13) T. Miyazaki, J. Kaneko and M. Sugamata: *Mater. Sci. Eng.* **A181/A182** (1994) 1410–1414.
- 14) M. Mabuchi and K. Higashi: *Acta Mater.* **44** (1996) 4611–4618.
- 15) Y.-W. Kim: *JOM.* **46** July (1994) 30–39.
- 16) J. S. Huang and Y.-W. Kim: *Scr. Metal.* **25** (1991) 1901–1906.
- 17) M. Mabuchi, Y. Chino and H. Iwasaki: *Mater. Trans.* **43** (2002) 2063–2068.