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Static Recrystallization Behavior of Mg Alloy Single Crystals

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Abstract**—**Single crystals of Magnesium alloys such as pure Mg, Mg-1Zn-0.5Y, Mg-0.1Y, and Mg-0.1Ce alloys were successfully fabricated in this study by employing the modified Bridgman method. To determine the exact orientation of crystals, pole figure measurement using X-ray diffraction were carried out on each single crystal. Hardness and compression tests were conducted followed by subsequent recrysatllization annealing. Recrystallization kinetics of Mg alloy single crystals has been investigated. Fabricated single crystals were cut into rectangular shaped specimen and solution treated at 400°C for 24 hrs, and then deformed in compression mode by 30% reduction. Annealing treatment for recrystallization has been conducted on these cold-rolled plates at temperatures of 300°C for various times from 1 to 20 mins. The microstructure observation and hardness measurement conducted on the recrystallized specimens revealed that static recrystallization of ternary alloy single crystal was very slow, while recrystallization behavior of binary alloy single crystals appeared to be very fast.

Keywords**—**Magnesium, Mg-rare earth alloys, compression test, static recrystallization, hardness.

I. Introduction

MAGNESIUM and its alloys have been considered strong candidates for the next generation of lightweight and high-strength materials because Mg is the lightest metal that can be employed for structural use [1]. Magnesium alloys are becoming more attractive for engineering applications because of their particularly low density, excellent damping capacity, good recycling capacity and machinability [2]. However, the number of commercially available Mg alloys is still limited especially for application at elevated temperature [3]. The poor cold rolling response of Mg is generally attributed to its hexagonal crystallography and the basic symmetry of hexagonal close-packed (HCP) crystals has the effect of limiting the number of independent slip systems and making twinning an important deformation mechanism [4]. The general result in wrought polycrystalline aggregate is a more or less sharply developed texture which underlies a strong anisotropy in mechanical behavior. Knowing how the one influences the other is essential background for the use of such materials.

Magnesium is well known to undergo unique plasticity transition in the neighborhood of 200°C [5, 6]. Plastic working of Mg alloys is, therefore, generally performed at above 250°C. Considering productivity including manufacturing expenditure, however, it needs to develop a new alloy system or a new technology for the plastic working at lower temperature than 250°C [7]. In this regards, the origin of plasticity transition and the exact deformation mechanisms of Mg and its alloy are very important to be systematically understood. In the present work, single crystals of pure Mg, Mg-0.1Y, Mg-1Zn-0.5Y, and Mg-0.1Ce alloys were fabricated and their compressive deformation behaviors were measured. The static recrystallization kinetics of pure Mg and Mg alloy single crystals were studied by conducting compression test followed by annealing at 300°C for various time intervals.

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II. Experimental Procedures

Single crystals of Mg and Mg alloys used in this study were grown by directional solidification, using a modified Bridgman method. Ingots of pure Mg, Mg-0.1Y, Mg-1Zn-0.5Y, and Mg-0.1Ce alloys were prepared by conventional casting and machined into rods, 10 mm in diameter and 120 mm in length with one end pointed to fit into a split graphite mould. The mould, carefully tightened with Kanthal wire, was then placed in a vertical quartz tube. The tube was surrounded by three independently controllable heating elements and was water cooled in the bottom end to give proper temperature gradient for crystal growth. The maximum temperature was set at 760°C near the central zone of the furnace. After the rod was completely melted, the mould was driven downward by a motor at the rate of ~ 13 mm per hour. The apparatus used in this study is schematically illustrated in Fig. 1. Fig. 2 shows the split graphite mould used and a typical as-grown single crystal.

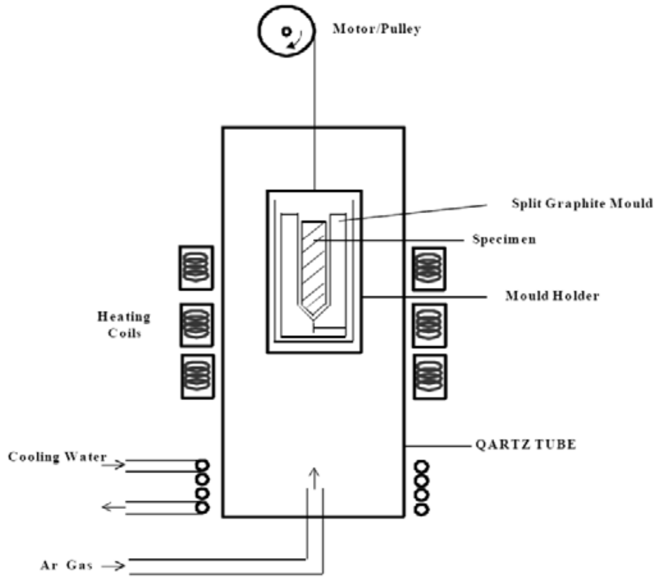

Fig. 1 Schematic illustration of the apparatus for single crystal growth

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Fig. 2 Graphite mould used in this study and as-grown crystal of pure Mg

A quick examination by etching in a NaOH solution occasionally revealed a few very small stray grains on the surface, but they were always removed from gauge section during machining process. Laue back reflection diffraction patterns were taken from a few spots of the bottom and top sections. The identity of the patterns confirmed them as single crystals. The pole figures of the single crystal specimens were also measured on planes normal to the solidification direction. (0002), $(10\bar{1}0)$, $(10\bar{1}1)$ and $(10\bar{1}2)$ pole figures were measured to analyze the orientations of single crystals grown in this study using X-ray reflection method up to a reflection angle of 70^o. Micro-hardness and compression tests were carried on the single crystals at room temperature.

Compression tests were conducted up to 30 % reduction at room temperature under the strain rate of 5×10^{-4} s^{-1}. Cylindrical specimens with a diameter of 8 mm and a height of 10 mm were used. After compression tests, after compression, these samples have been annealed for recrystallization at temperature of 300^{o}C for various times from 1 min to 20 min. Microstructures of annealed specimens were observed by optical microscopy (Olympus BX51M) and hardness was measured by micro Vickers hardness tester (Mitutoyo HM-112). Pole figures were also measured on the same plane to compare with the initial orientations of single crystals.

III. Results and Discussion

Fig. 3 shows initial microstructures of pure Mg, Mg-0.1Y, Mg-1Zn-0.5Y, and Mg-0.1Ce alloy ingots, revealing very coarse grain structure. In the case of alloys containing rare earth elements, some intermetallic precipitates are observed along grain boundary and in grain matrix. The (0002) pole figures of single crystals of pure Mg, Mg-0.1Y, Mg-1Zn-0.5Y, and Mg-0.1Ce alloys are illustrated in Fig. 4. As shown in Fig. 4, orientation of the single crystals appeared to be somewhat away from the basal orientation. Some traces from twins were also measured in the pole figures. The orientation of Mg-1Zn-0.5Y single crystal is very similar to that of pure Mg single crystal.

Optical microstructures of single crystals fabricated in this study were shown in Fig. 5, revealing some annealing twins. More amounts of twins were observed in the single crystals containing rare earth elements.

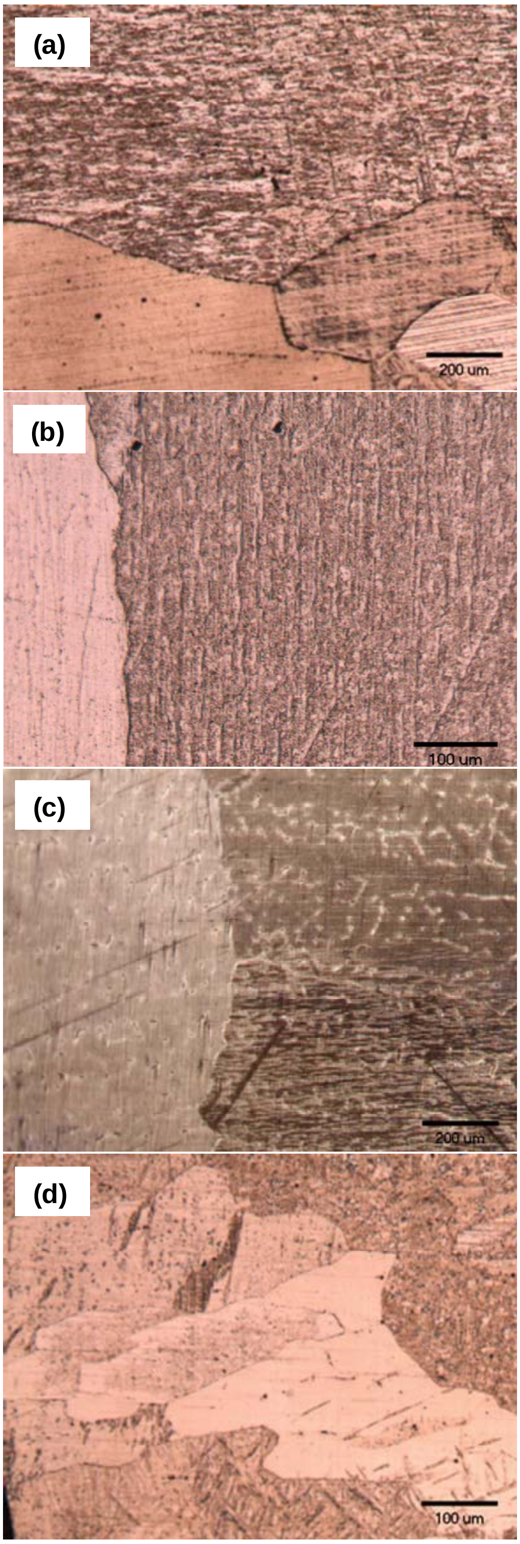

Fig. 3 As-cast microstructures of ingots of (a) pure Mg, (b) Mg-0.1Y, (c) Mg-1Zn-0.5Y, and (d) Mg-0.1Ce alloys

Fig. 6 shows compressive flow curves of the single crystals of pure Mg and Mg-Zn-0.5Y alloy grown in this study. It is very interesting to note from Fig. 6 that flow stress and strain hardening rate of pure Mg is much lower than those of Mg-1Zn-0.5Y, even though the orientation is very similar to each other. In the case of pure Mg single crystal, well known easy glide behavior is observed in the early stage of compression test. The yield strength of Mg-1Zn-0.5Y alloy single crystal is much higher than that of pure Mg single crystal, which indicates alloying effect is significant on the critical resolved shear stress (TRSS).

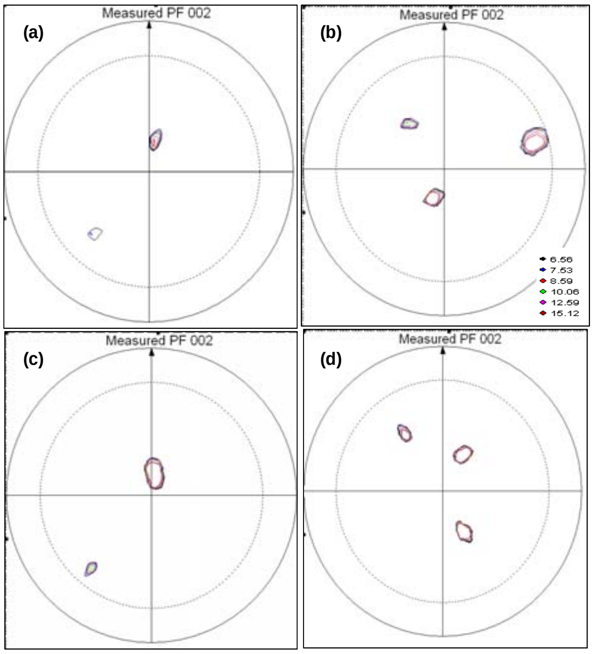

Fig. 4 (0002) pole figures measured on the single crystals of (a) pure Mg, (b) Mg-0.1Y, (c) Mg-1Zn-0.5Y, and (d) Mg-0.1Ce alloys

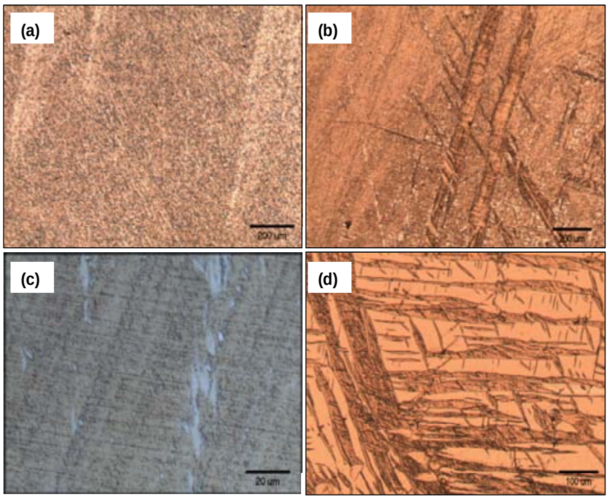

Fig. 5 Optical micrographs of the single crystals of (a) pure Mg, (b) Mg-0.1Y, (c) Mg-1Zn-0.5Y, and (d) Mg-0.1Ce alloys

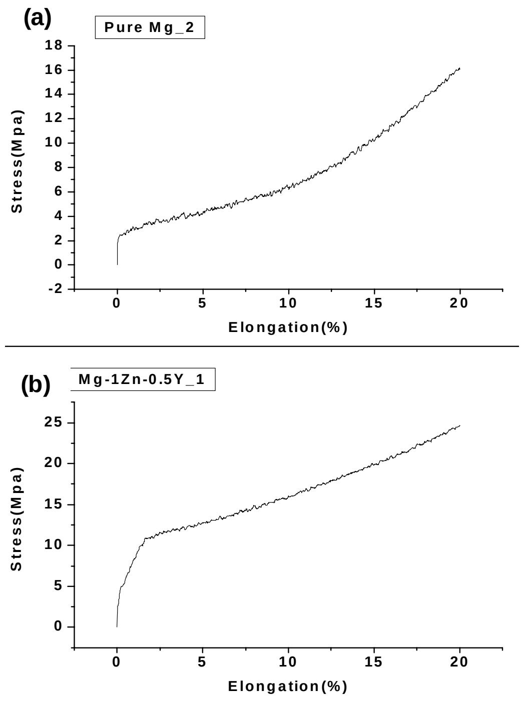

Fig. 6 Compressive flow curves of the single crystals of (a) pure Mg and (b) Mg-Zn-0.5Y grown in this study

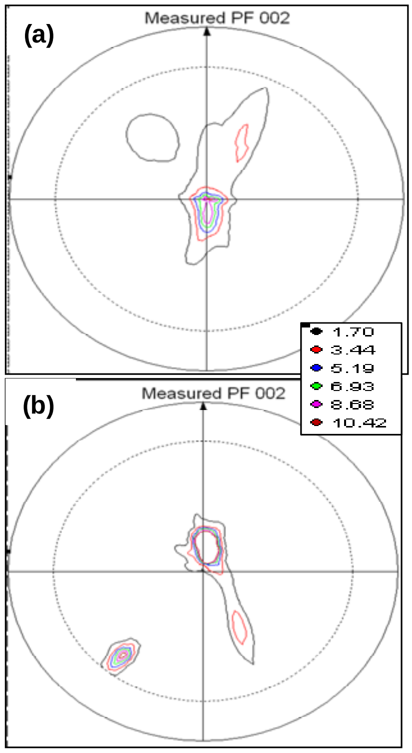

Fig. 7 (0002) pole figures measured after 30% compressive reduction on the pure Mg single crystal (a) and Mg-1Zn-0.5Y alloy single crystal

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Fig. 7 shows (0002) pole figures measured on the single crystals of pure Mg and Mg-Zn-0.5Y alloy compressed by 30% reduction at room temperature, of which the orientations are very similar to each other. In the case of pure Mg single crystal, orientation after compression test moved towards basal one, while that of Mg-Zn-0.5Y alloy single crystal appeared the same. The intensity of poles weakened somewhat. This is very important point to note because the basal texture deduced in the Mg and Mg alloy after wrought process causes poor formability. As shown in Figs. 7 and 8, off-basal texture can be achieved in Mg alloy by adding a small amount of rare earth elements.

The same results were obtained from the compressed single crystals of Mg-0.1Y and Mg-0.1Ce alloys as illustrated in Fig. 8. Even after 30% compressive reduction, off-basal orientation remained unchanged in both alloy single crystals.

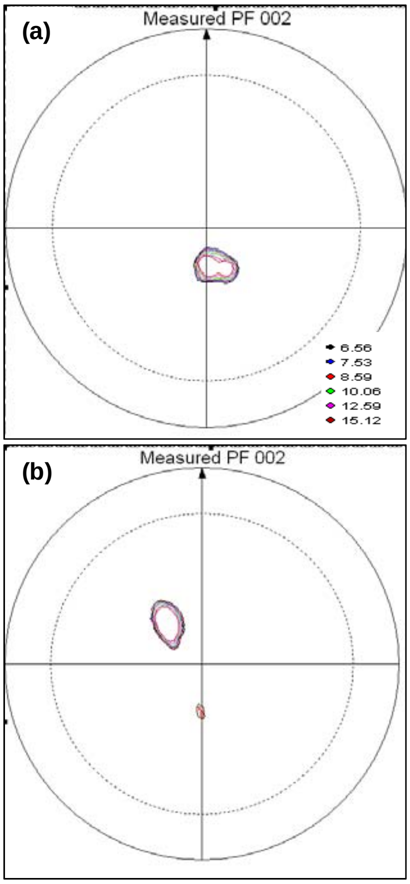

Fig. 8 (0002) pole figures measured after 30% compressive reduction on the single crystals of (a) Mg-0.1Y and (b) Mg-0.1Ce alloys

After compression by 30% reduction, all specimens were annealed at 300°C for various time intervals from 1 to 20 min to investigate recrystallization behavior and the microstructures were observed as shown in Fig. 9. While recrystallization of pure Mg was found to finish very fast, that of Mg-RE alloy appeared to be relatively slower. In binary alloys such as Mg-0.1Y and Mg-0.1Ce alloys, recrystallization occur faster that in ternary alloy. Even after 20 min at 300°C, no evidence of recrystallization could be observed in Mg-1Zn-0.5Y alloy single crystal. Hardness of Mg-1Zn-0.5Y alloy single crystal after annealing appeared to be much higher than those of binary single crystals as shown in Fig. 10. Hardness of Mg-1Zn-0.5Y alloy single crystal remained and dropped little compared with those of binary alloy single crystals.

IV. Conclusion

In this study, static recrystallization behavior of cold rolled pure Mg and Mg-RE alloy single crystals have been investigated. Single crystals of pure Mg, Mg-0.1Y, Mg-1Zn-0.5Y, and Mg-0.1Ce alloys were successfully fabricated in this study by employing the modified Bridgman method. To determine the exact orientation of crystals, pole figure measurement wase carried out. Hardness and compression tests were conducted and the results revealed that hardness and the strength strongly depended on the orientation. After compressive deformation of single crystals, the orientation of the pure Mg single crystal was found to rotate and to be parallel to the basal orientation, while that of Mg-Zn-0.5Y, Mg-0.1Y and Mg-0.1Ce alloy single crystals appeared off-basal, the same as before compression test. While recrystallization of pure Mg was found to finish very fast, that of Mg-RE alloy appeared to be relatively slower.

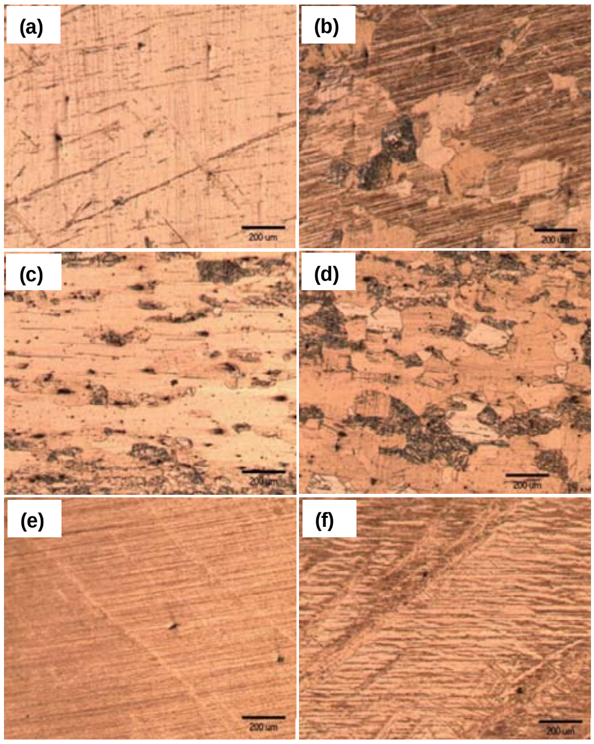

Fig. 9 Optical micrographs of the single crystals compressed and annealed at 300°C; Mg-0.1Y alloy for 1 (a), and 10 min (b). Mg-0.1Ce alloy for 1 (c) and 20 min (d). and Mg-1Zn-0.5Y alloy for 1 (e) and 20 min (f), respectively

Acknowledgment

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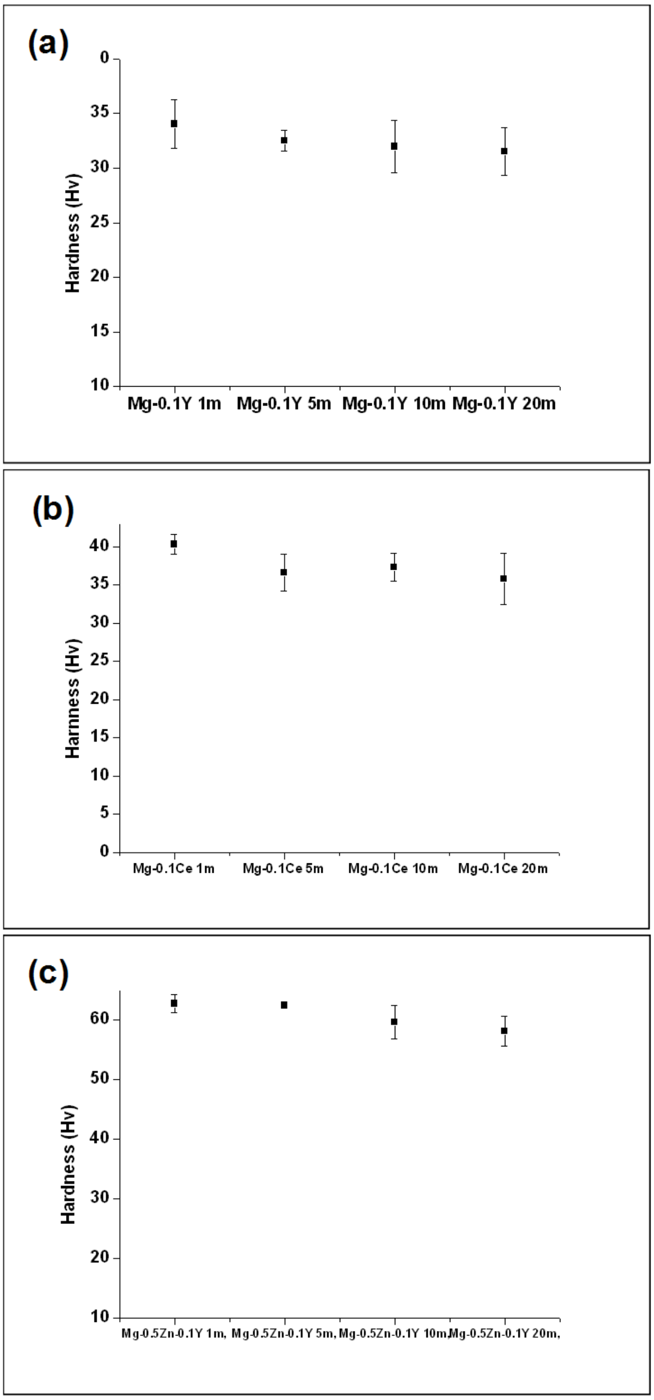

Fig. 10 Micro-hardness as a function of annealing time measured on the compressed and annealed single crystals of (a) Mg-0.1Y, (b) Mg-0.1Ce, and (c) Mg-1Zn-0.5Y alloys

REFERENCES

[1] W. Qiu, E-H. Han, L. Liu, Trans. Nonferrous Met. Soc. China, vol. 20, p. 481. 2010.

[2] S. M. Masoudpanah, R. Mahmudi, Mater. Sci. & Eng. A, vol. 526, p. 22. 2009.

[3] Y. Wang, S. Guan, X. Zeng, W. Ding, Mater. Sci. & Eng. A, vol. 416, p. 109. 2006.

[4] M. R. Barnett, M.D. Nave, C.J. Bettles, Mater. Sci. & Eng. A, vol. 386, p. 205, 2004.

[5] E. Schmid, Z. Elektrochem, vol. 37, p.447, 1931.

[6] A. Beck, “The technology of Magnesium and Its Alloys”, Huges, London, p. 22, 1940.

[7] N.-J. Park, S.-H. Han: Kor. J. Met. Mater. vol. 48, pp. 498-505, 2010.

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