Evaluation of Non-Basal Slip Activity in Rolled Mg–Li Alloys*¹

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Tensile tests of rolled Mg–6.2 mol%Li and Mg–11.7 mol%Li alloys were carried out at room temperature to clarify effects of lithium addition on the relationship between mechanical properties and activities of slip systems. Ductility increased with increasing lithium content. 0.2% proof stress increased when 6.2 mol%Li was added. However, Mg–11.7 mol%Li showed low 0.2% proof stress, compared to pure magnesium. On the other hand, maximum stress decreased with increasing lithium content. Frequency of non-basal slips increased with increasing lithium content. Also, first order pyramidal $\langle c + a \rangle$ slip showed the highest frequency among non-basal slips in Mg–Li alloys. Critical resolved shear stresses for non-basal slips, which were reduced by lithium addition, increased ductility but decreased tensile strength of magnesium. [doi:10.2320/matertrans.L-M2021826]

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1. Introduction

Main slip system of magnesium is basal slip (BS). However, BS only offers two independent slip systems, and the number of basal slip is insufficient for von Mises criterion. For that reason, room temperature formability of magnesium is limited. Many magnesium alloys containing rare earth elements have been studied to improve the low ductility.^{1–5)} Sandlöbes *et al.*¹⁾ reported that Mg–0.84 mol%Y alloy polycrystals had five times higher ductility than pure magnesium and strength as same as pure magnesium at room temperature. In the Ref. 1), the high ductility was explained by the high activity of $\langle c + a \rangle$ dislocations due to yttrium addition. Rikihisa *et al.*²⁾ performed tensile tests of Mg–(0.6–1.3) mol%Y alloy single crystals in the directions of parallel to basal planes and reported that Mg–(0.6–0.9) mol%Y alloy single crystals yielded due to $\{10\bar{1}1\}\{11\bar{2}3\}$ first order pyramidal $\langle c + a \rangle$ slip (FPCS). Also, yttrium addition was found to increase the activity of FPCS based on results of tensile tests of Mg–0.9 mol%Y alloy polycrystals with basal texture. Therefore, they concluded that the high ductility of Mg–Y alloys resulted from the high activity of FPCS in Ref. 2).

Ando *et al.*⁶⁾ reported that Mg–(3.5–14.0) mol%Li alloy single crystals yielded due to second order pyramidal $\langle c + a \rangle$ slip (SPCS) in $[11\bar{2}0]$ tensile tests and that yield stress decreased with increasing lithium content. Also, Yoshinaga *et al.*⁷⁾ and Ahmadieh *et al.*⁸⁾ individually reported that the critical resolved shear stress (CRSS) for prismatic slip (PS) decreased with increasing lithium content. Therefore, the ductility of magnesium was expected to increase by improving activity of non-basal slips due to lithium addition. However, activated slip systems in Mg–Li alloy polycrystals

and effects of lithium addition on the slip system are unclear. The present study was carried out to clarify the effects of lithium addition on the relationship between mechanical properties and activities of slip systems in Mg–Li alloy polycrystals.

2. Experimental Procedures

Two types of Mg–Li alloys with different lithium contents were cast from 99.9% or more lithium (Nacalai Tesque Inc.) and pure magnesium using a high frequency induction heating vacuum furnace; the chemical composition of pure magnesium are shown in Table 1. Two Mg–Li alloys were identified to be Mg–6.2 mol%Li and Mg–11.7 mol%Li by ICP emission spectroscopy. Mg–Li alloys were hot-rolled and annealed to obtain mean grain size of approximately 50 μm . Mg–Li alloys were hot-rolled at 523 K with rolling reduction approximately 10% per pass and were subsequently water quenched; the total reduction was 72%. Mg–6.2 mol%Li alloys were annealed at 723 K for 1.5 ks, while Mg–11.7 mol%Li alloys were annealed at 748 K for 1.2 ks, in an argon atmosphere.

Figure 1 shows a schematic illustration of tensile specimen; tensile direction was parallel to rolling direction of

Table 1 Chemical composition of pure magnesium ingots. (mass%)

Zn	Ni	Si	Mn	Fe	Cu	Al	Mg
0.0607	0.1017	0.3875	0.0804	0.1178	0.1231	0.2911	Bal.

Fig. 1 Schematic illustration of tensile test specimen.

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Fig. 2 (0002) and $\{10\bar{1}0\}$ pole figures of (a) Mg–6.2 mol%Li and (b) Mg–11.7 mol%Li alloys before tensile tests.

sheets (RD). Tensile specimens were mechanically polished using emery paper (#400–4000), an MD-Nap (Struers), and diamond spray of 3, 1 and 0.25 μm (Struers). Specimen surfaces after mechanical polishing were chemically polished to mirror using a polishing cloth soaked in a solution ($\text{HNO}_3\text{:H}_2\text{O}_2\text{:C}_2\text{H}_5\text{OH} = 5\text{:}7\text{:}20$) and were corroded with a picric acid solution ($\text{HOC}_6\text{H}_2(\text{NO}_2)_3\text{:CO}_3\text{COOH:C}_2\text{H}_5\text{OH:H}_2\text{O} = 5\text{:}10\text{:}60\text{:}10$). For trace analysis, initial crystal orientations of the normal direction of specimens (ND) were analyzed using a FE-SEM (JEOL Ltd. JIB-4601F) and an EBSD camera (TexSEM Laboratories corp.) before tensile tests. Tensile tests were carried out at 298 K with the initial strain rate, $\dot{\varepsilon} = 8.4 \times 10^{-4}/\text{s}$. Slip lines were observed using a Nomarski type optical microscope (Nikon corp. ECLIPSE LV 150N) after tensile tests. Slip systems were identified at each strain, and Schmid factor for activated slip systems were determined using the initial crystal orientation analyzed by EBSD before tensile tests.

3. Results

Figure 2 shows (0002) and $\{10\bar{1}0\}$ pole figures of (a) Mg–6.2 mol%Li and (b) Mg–11.7 mol%Li alloys. Both Mg–Li alloys showed basal texture whose c-axis are oriented parallel to ND. Similar texture was reported in pure magnesium; the maximum intensity of texture was approximately 23.²⁾ The maximum intensity was 12 in Mg–6.2 mol%Li alloys and 11 in Mg–6.2 mol%Li alloys. Therefore, lithium addition was found to make basal texture weaker in magnesium. Also, the difference in the lithium additions barely affected the maximum intensity of the basal texture.

Figure 3 shows typical stress-strain curves of Mg–Li alloys. The results²⁾ on pure magnesium and Mg–0.9 mol%Y are also shown for comparison. The horizontal and vertical arrows in Fig. 3 indicate 0.2% proof stress and fracture

Fig. 3 Stress-strain curves of pure magnesium, Mg–6.2 mol%Li, Mg–11.7 mol%Li and Mg–0.9 mol%Y alloys.

Fig. 4 0.2% proof stress, maximum stress, fracture strain as a function of lithium content in Mg–Li alloys.

Fig. 5 Optical micrographs of slip lines and $\{10\bar{1}2\}$ twins on the surfaces of (a) Mg–6.2 mol%Li and (b) Mg–11.7 mol%Li. Slip lines were crystallographically analyzed and identified to the corresponding slip system: basal slip (BS); prismatic slip (PS); first order $\langle c + a \rangle$ pyramidal slip (FPCS); and second order $\langle c + a \rangle$ pyramidal slip (SPCS).

points. Two Mg–Li alloys show low work hardening rates until 6.0% tensile strain, compared to pure magnesium. Mg–6.2 mol%Li alloys broke at approximately 6.0% tensile strain, while Mg–11.7 mol%Li alloys showed almost constant flow stress with no break. Figure 4 shows changes in 0.2% proof stress, maximum stress and fracture strain, as a function of lithium content in Mg–Li alloys. 0.2% proof stress increased in 6.2 mol% lithium addition, but decreased in 11.7 mol% lithium addition. Also, maximum stress decreased, and ductility increased, with increasing lithium content.

Figure 5 shows optical micrographs of slip lines in Mg–Li alloys. Slip lines caused by BS, PS, FPCS and SPCS and $\{10\bar{1}2\}$ twins with lenticular morphology were observed in each Mg–Li alloy. BS slip lines appeared in many grains and occupied each grain. FPCS slip lines finer than BS slip lines

Fig. 6 Relationship between frequency of activated slips and nominal strain in pure magnesium, Mg–6.2 mol%Li, Mg–11.7 mol%Li and Mg–0.9 mol%Y alloys.

never occupied each grain but locally appeared near grain boundaries, as shown in Fig. 5(b). On the other hand, both PS and SPCS slip lines were short, and most of them were observed near grain boundaries only. The number fraction of grains where $\{10\bar{1}2\}$ twins were activated decreased with increasing lithium content; the frequency was 50% in Mg–6.2 mol%Li alloys ($\epsilon = 3.6\%$) and 8% in Mg–11.7 mol%Li alloys ($\epsilon = 3.0\%$). Here, Mg–11.7 mol%Li alloys with the low frequency showed high ductility. Conversely, in Mg–6.2 mol%Li alloys, the frequency was 50%; however the area fraction of $\{10\bar{1}2\}$ twins in each grain was approximately 15%, resulting in approximately 7.5% area fraction of the whole specimen. Also, the present Mg–Li alloys had basal texture, as shown in Fig. 2. Strain caused by $\{10\bar{1}2\}$ twinning occurs parallel to basal planes, i.e., compressive strain occurs parallel to the tensile direction (RD). Even assuming that the strain caused by $\{10\bar{1}2\}$ twinning affects the ductility, the shear strain of $\{10\bar{1}2\}$ twin in Mg is 13% and the normal strain parallel to the basal plane calculated to be 6.8%. Therefore, considering the area fraction of $\{10\bar{1}2\}$ twin, the amount of the strain caused by $\{10\bar{1}2\}$ twinning was estimated to be approximately 0.5%, and the effect of $\{10\bar{1}2\}$ twinning on the ductility is quite limited in Mg–6.2 mol%Li alloys.

Figure 6 shows the relationship between frequency of activated slips and nominal strain in pure magnesium,²⁾ Mg–Li alloys and Mg–0.9 mol%Y alloys.²⁾ Here, slip frequency was defined as the ratio of the number of grains with slip lines to the number of observed grains. The results on Mg–0.9 mol%Y alloys are also shown for comparison since the ductility was similar to that in Mg–11.7 mol%Li alloys. The frequency of BS was the highest and approximately 50% at a failure strain of approximately 4.0% in pure magnesium. Non-basal slips were also activated; however, each frequency was a few percent. Mg–0.9 mol%Y alloys with high ductility were reported to have high frequency of each slip system ($\epsilon = 12.5\%$),²⁾ especially, BS frequency was extremely high at 90% or more. Conversely, BS frequency was 30–40% in Mg–Li alloys and was lower than those in both pure magnesium and Mg–0.9 mol%Y alloys. Both FPCS and PS frequencies in Mg–Li alloys were higher than those in Mg–0.9 mol%Y alloys. Also, FPCS showed the highest frequency among non-basal slips in Mg–Li alloys. Both FPCS and PS

Fig. 7 Number fraction of grains where basal slips were activated as a function of Schmid factor for basal slips in (a) Mg–6.2 mol%Li and (b) Mg–11.7 mol%Li alloys.

Fig. 8 Resolved shear stresses (RSSs) for non-basal slips at a nominal strain of approximately 3.0% in Mg–6.2 mol%Li and Mg–11.7 mol%Li alloys.

frequencies increased with increasing lithium content but not SPCS.

Figure 7 shows changes in the number fraction of grains where basal slips were activated at any strain as a function of Schmid factor for basal slip at intervals of 0.1 in (a) Mg–6.2 mol%Li and (b) Mg–11.7 mol%Li alloys. Here, the black bars shows the number fraction of all grains analyzed by EBSD before the tensile tests; the highest Schmid factor for BS in each grain was used for the sorting. Therefore, tensile specimens before the tests had many grains with the small Schmid factor for BS because of its basal texture. The other bars show the number fraction of grains where BS was activated at each tensile strain. During the tensile test, BS activation started to occur in grains with relatively high Schmid factors for BS and then occurred in descending order, showing that BS activation depends on Schmid's law.

Figure 8 shows the resolved shear stresses (RSSs) for non-basal slips at a nominal strain of approximately 3.0% in Mg–Li alloys. Here, assuming uniaxial stress, RSS was calculated using Schmid factor for activated slip systems and

flow stress. Therefore, the RSS may be different from the actual shear stress loading on slip planes. Dashed lines in Fig. 8 indicate CRSS for SPCS in Mg–Li alloys; the CRSSs were calculated using the yield stress obtained from $[11\bar{2}0]$ tensile tests of Mg–Li alloy single crystals reported by Ando *et al.*^{6,9)} approximately 36 MPa in Mg–7.0 mol% Li alloys and 29 MPa in Mg–10.6 mol% Li alloys. Solid lines in Fig. 8 indicate CRSSs for PS: approximately 30 MPa in Mg–7.9 mol%Li alloys⁸⁾ and 24 MPa in Mg–12.9 mol%Li alloys.⁸⁾ Therefore, RSSs higher than CRSSs were found to apparently load on slip planes in most grains where SPCS and PS were activated in Mg–Li alloys. Here, CRSS for FPCS in Mg–Li alloys remains unclear, but must be higher than that for SPCS since Mg–Li alloy single crystals yielded due to SPCS^{6,9)} in $[11\bar{2}0]$ tensile tests. Therefore, in more than half the number of grains where FPCS were activated, FPCS would be activated at RSSs below the CRSS. Here, Ando *et al.*^{6,9)} reported that Mg–Li alloy single crystals yielded due to SPCS and showed work hardening in $[11\bar{2}0]$ tensile tests. Therefore, lines indicating CRSS for SPCS in Fig. 8 must be raised up when the strain of approximately 3% was considered. Hence, FPCS can be activated at RSSs below the CRSS in more grains. Also, slip lines caused by BS were observed in the grain next to the grain where FPCS was activated. Therefore, the stress concentration near grain boundaries by BS results in that FPCS is apparently activated at RSSs below the CRSS.

4. Discussion

Effects of lithium addition on mechanical properties and slip systems will be discussed below. 0.2% proof stress increased in Mg–6.2 mol%Li alloys but decreased in Mg–11.7 mol%Li alloys, compared to pure magnesium, as shown in Fig. 4. Figure 9 shows relationship between CRSSs and content of alloy elements in pure magnesium,^{10–12)} Mg–Li^{6–12)} and Mg–Y^{2,13)} alloys. When lithium was added, CRSS for BS increased in 6.6 mol% addition;⁷⁾ however, it barely changed even in 15.0 mol% addition.⁷⁾ Conversely, CRSSs for both PS and SPCS decreased with increasing lithium

Fig. 9 Relationship between CRSSs and content of alloy elements in Mg–Li and Mg–Y alloys.

content. Therefore, 0.2% proof stress of Mg–6.2 mol%Li alloys increases by the increase in CRSS for BS, while 0.2% proof stress of Mg–11.7 mol%Li alloys decreases by the significant decrease in CRSSs for non-basal slips.

Figure 10 shows schematic illustrations of texture, tensile properties and types of slip systems in rolled pure magnesium and magnesium alloys, based on the results in this study. Lithium addition increased ductility of magnesium but decreased the strength. Here, Mg–11.7 mol%Li alloys showed 20% ductility, and it was approximately the same as that in Mg–0.9 mol%Y alloys.²⁾ Rolled pure magnesium with strong basal texture shows high 0.2% proof stress since Schmid factor for BS is close to 0 as the unit cell shown in Fig. 10(a). On the other hand, the ductility is low due to the low activity of BS. Therefore, the tilt of basal planes from the tensile direction is key to increasing the ductility, as shown in Fig. 10(b).¹⁴⁾ However, when the basal planes tilt from the tensile direction, Schmid factor for BS increases, i.e., BS becomes more easily activated, resulting in that the ductility increases but strength decreases. Rikihisa *et al.*²⁾ reported that yttrium addition increases ductility without decreasing strength, as shown in Fig. 3. The cause of the high ductility resulted from that basal texture was weakened by yttrium addition²⁾ as shown in Fig. 10(c); activities of not

Fig. 10 Schematic illustrations of texture and tensile properties of rolled pure magnesium and magnesium alloys.

only BS but also non-basal slips increased as shown in Fig. 6. However, yttrium addition weakened basal texture but increased CRSS for both BS and non-basal slips, resulting in the strength did not decrease, as shown in Fig. 9. On the other hand, Mg–Li alloys showed strong basal texture, similar to that in rolled pure magnesium, and BS frequency was 30–40%, as shown in Fig. 6. Therefore, the increase in the ductility was caused by higher non-basal slip activity than those in pure magnesium and Mg–0.9 mol%Y alloys. CRSS for BS slightly increased by lithium addition, but the strength decreased since CRSSs for non-basal slips greatly decreased. Here, in both Mg–Li and Mg–Y alloys, the frequency of FPCS and PS was high among non-basal slips. Rikihisa *et al.*²⁾ reported that Mg–1.2 mol%Y alloys showed a lower ductility compared to Mg–0.9 mol%Y alloys and the frequency of PS was higher than those of FPCS and SPCS. Therefore, they concluded that FPCS activation is required to improve ductility. Frequencies of FPCS and PS in the present Mg–Li alloys are approximately the same; however, the area with slip lines caused by FPCS was wider than that by PS, as shown in Fig. 5. Therefore, the contribution of FPCS is high in deformation of Mg–Li alloys. Which slip system contributes to increasing ductility will be discussed in the near future.

5. Conclusions

Tensile tests of rolled Mg–6.2 mol%Li and Mg–11.7 mol%Li alloys were carried out to clarify the relationship between mechanical properties and activities of slip systems. Main results are as follows:

- (1) Ductility increased but tensile strength decreased, with increasing lithium content.
- (2) Frequency of non-basal slips increased with increasing lithium content. First order pyramidal $\langle c + a \rangle$ slip

showed the highest frequency among non-basal slips in Mg–Li alloys.

- (3) Frequency of basal slip in Mg–11.7 mol%Li alloys were similar to that in pure magnesium. Frequency of non-basal slips in Mg–11.7 mol%Li alloys was higher than that in Mg–0.9 mol%Y alloys, indicating that activation of non-basal slips improved ductility in Mg–11.7 mol%Li alloys.
- (4) Lithium addition decreased critical resolved shear stresses for non-basal slips, resulting in decreasing tensile strength.

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