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論 文 内 容 要 旨

Chapter 1. Research backgrounds and objectives

Damping capacity of magnesium (Mg) is generally dominated by basal slip, which absorbs the external vibration energy through reciprocating motion of dislocations. The high damping capacity of pure Mg can usually be ascribed to the extremely low critical resolved shear stress (CRSS) of basal slip (~0.6 MPa) as compared to that of non-basal slip systems (>38 MPa). However, damping capacity of Mg is significantly reduced once it is alloyed in order to improve its mechanical properties.

Several techniques have been applied to modify the damping capacity of Mg alloys by such as adding Ni, Cu, or heat treatment. However, these methods generally either deteriorate the mechanical properties or lead to other problems, such as complications in manufacturing. Novel methods to substantially enhance the damping capacity of Mg alloys, without compromising mechanical properties, would be highly valuable for engineering applications as well as in fundamental scientific research.

Twinning is an important deformation behaviour due to limited slip systems in Mg alloys as close-packed hexagonal structure (hcp) metals. The twin boundary mobility has attracted many researchers' attention. From previous research, the motion of twin boundaries contributes to the high damping capacity of Mn-Cu alloy. This influence is more significant after the removal of residual stress by annealing. However, whether the motion of twin boundaries contributes to the damping capacity of Mg alloys has never been verified. It is well known that $\{10\bar{1}2\}$ tensile twin boundaries in Mg are movable and they can shrink or grow even at a stress much lower than the nucleation stress. CRSS for the growth of $\{10\bar{1}2\}$ twin is estimated to be ~2-3 MPa for AZ31 alloy, which is slightly higher but very close to that of the basal slip system (approximately 2 MPa).

However, no clear evidence has been provided, and it remains unknown how twins can affect the damping capacity of Mg alloys, and also to what extent the damping capacity can be improved.

The solute atoms and resultant precipitates impose a tremendous impact on the motion of twin boundaries. Twin boundaries can be stabilized by the occurrence of precipitates and segregation of solute atoms into twin boundaries after annealing. To clarify the mechanism of twin-boundary motion so as to further optimize properties of Mg alloys, it is crucial to fully understand the effect of solute elements and their resultant precipitates on both twin growth and detwinning behavior.

In summary, on one hand, twin boundary can be stabilized by alloying and annealing to enhance the yield strength of Mg alloys. On the other hand, improving the twin boundary mobility by annealing has potential to improve the damping capacity of Mg alloys. However, there was no systematic research about the effect of alloying and annealing on twin boundary mobility.

The objectives of our research are to study the influences of alloying elements on twin growth and detwinning behaviour of Mg and its alloys systematically. The effects of annealing on twin boundary mobility of Mg and its alloys are also systematically studied. Whether the motion of twin boundaries contributes to damping capacity of Mg alloys are to be clarified.

Chapter 1 describes the above-mentioned background and objectives of this study.

Chapter 2. Influences of alloying elements on twinning behaviour in Mg and its alloys

Chapter 2 describes the effects of alloying elements on the twinning behavior. Micro-scaled and nano-scaled precipitates embedded or along twin boundaries observed by SEM and TEM are considered to impede the twin growth. Consequently, thinner twins formed in Mg alloys compared to pure Mg due to the hardening effect on the twin growth by solute atoms and precipitates. Besides, the yield stress for twinning is almost independent of strain rate and temperature while slips are sensitive to strain rate and temperature. As a result, the twinning behavior is suppressed due to increased activity for slipping with the increase of temperature. Twinning can easily be activated at high strain rate due to a relatively high CRSS for slips. Our results revealed that the influences of temperature and strain rate on twinning behavior are relieved by the strengthening effect by alloying elements.

Chapter 3. Influences of alloying elements on detwinning behaviour in Mg and its alloys

Chapter 3 describes the systematic twinning and detwinning behaviors of $\{10\bar{1}2\}$ tensile twins in pre-compressed pure Mg and Mg alloys AZ31 and AZ91 investigated by combining compressive tests, reverse tensile tests, in-situ electron-backscattering diffraction, and high-resolution transmission electron microscopy. Fig.1 shows microstructural evolution as a function of tensile strain of pre-compressed samples, which is observed by in-situ EBSD. The results revealed that the twin growth is restricted by solute atoms and the resultant precipitations, giving rise to thinner $\{10\bar{1}2\}$ tensile twins during pre-compression. Moreover, the solute atoms and their resultant precipitates disrupt the synchronous motion of atoms in twin boundaries during twinning formation, yielding a large amount of semi-coherent twin boundaries (incoherent twin boundaries). Detwinning is also retarded by alloying elements. The tendency of detwinning via reverse motion of twinning boundary is weakened, and the secondary twins are formed remarkably in the interior of the primary twins in the Mg alloys (as shown by black arrows in Fig. 1) with a high solute-element concentration owing to the low mobility of the semi-coherent twin boundaries and the impedance of reverse motion of twin boundaries by precipitates. The selection mechanism of secondary twin variants during reverse deformation is discussed in our research. The reverse motion of twin boundary (detwinning) can be regarded as a special secondary twinning with the opposite twin variants. The compatibility strain was considered to be the main factor determining the selection of secondary twin variants during reverse tensile deformation. The secondary twins tend to be formed where the twin variant matches closely between the primary and the selected secondary twinning plane, thus minimizing the compatibility strain. This is the reason why detwinning generally occurs via the reverse motion of twin boundaries, with lowest compatibility. However, the reverse motion of twin boundaries can be impeded by solute atoms and precipitates in Mg alloys. In this case, secondary twin variants with preferable Schmid factor form instead of the opposite twin variant (the reverse motion of twin boundaries).

In addition, more low-angle boundaries were formed from the pre-existing twin boundaries after detwinning with larger content of alloying elements. It was considered to be closely related to twin boundary structures before detwinning. Our results revealed that more semi-coherent twin boundaries formed due to precipitates and solute atoms in Mg alloys compared to pure Mg. Low angle boundaries could be formed when the component of coherent twin boundaries move away from the semi-coherent twin boundaries.

The ultimate tensile strength of pre-compressed Mg and Mg alloys was found to be increased compared to solution treated (ST) samples, especially in AZ91 alloy, which is attributed to the hardening effect caused by the formation of many low-angle and secondary twin boundaries and also to the reduced Schmid factor for the basal slip in Mg alloys during detwinning process.

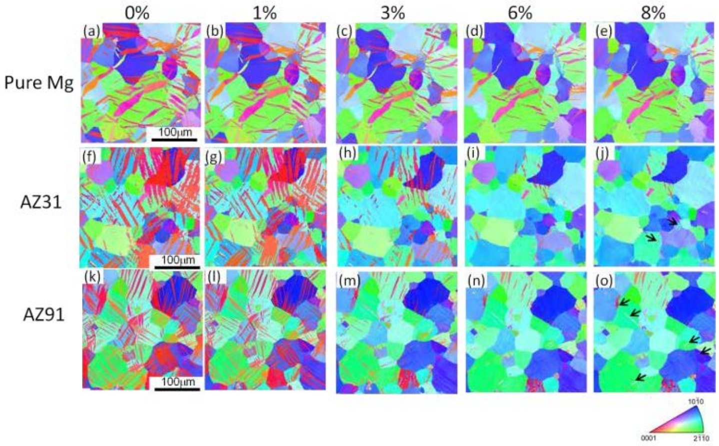

Fig.1. Inverse pole figure maps of pre-compressed pure Mg, AZ31 alloy, and AZ91 alloy after tension to a strain level of 0, 1, 3, 6, and 8%.

Chapter 4. Influences of annealing on twin boundary mobility in Mg and its alloys

In Chapter 4, the effects of annealing on twin boundary mobility in Mg and its alloys were evaluated. The friction stress and back stress were considered to affect twin boundary mobility. The friction stress represents the required stress for the twin boundary motion, which is not influenced by the residual stress distribution in both sides of twinning boundary but is closely related to twinning boundaries characteristic such as alloying element concentration, coherency, and the presence of precipitates etc. The non-uniformity of stress distribution on two sides of twinning boundaries contributes to the back stress. Both friction stress and back stress were calculated based on the further-compressive yield stress and reverse tensile stress of pre-compressed samples.

The back stress could be relieved by annealing due to the elimination of dislocations and stress field nearby twin boundaries, especially around twin tips. The non-uniformity of stress distribution was relieved by annealing and twin boundary mobility was improved consequently. The friction stress for motion of twin boundary was determined by dislocation density, segregation of solute atoms and precipitation within twin and/or along twin boundaries simultaneously. Firstly, the decrease in dislocation density after annealing is beneficial for the twin boundary motion, resulting in the decreased friction stress. Secondly, the significant segregation hardening contributed to the increase of the friction stress, especially in AZ91 alloy. The segregation of alloying elements Al and Zn into twin boundaries after annealing in Mg alloys AZ31 and AZ91 was verified by experiments for the first time. Lastly, the increased amount of precipitates after annealing, which is significant in AZ91 alloy, is also considered to contribute to the increase in friction stress. Our results revealed that the dislocation density mainly dominated the twin boundary mobility in pure Mg and AZ31 alloy. The twin boundary mobility was increased due to decreased dislocation density after annealing. However, the segregated solute atoms into twin boundaries and increased amount of segregates after annealing mainly dominated the twin boundary mobility in AZ91 alloy. The twin boundary mobility increased after short-time annealing (at 250 °C less than 600s). However, it decreased after long-time annealing (at 250 °C for 5000 s) due to significant segregation strengthening and precipitation strengthening. In summary, annealing has different effects on the twin boundary mobility in Mg and its alloys.

Chapter 5. Influences of mobile twin boundaries on damping capacity in Mg alloys

In Chapter 5, we firstly verified that the motion of twin boundaries contribute to the increased damping capacity of Mg alloys. The twin boundary mobility was proved to be closely related to the damping capacity of Mg alloy containing twins. Mg alloys AZ31 and AZ91 samples containing twin boundaries show higher damping capacity than those without twin boundaries. Furthermore, the damping capacity of pre-compressed AZ31 alloy is increased after annealing at 250 °C, which is coincident with the improved twin boundary mobility after annealing. The damping capacity of pre-compressed AZ91 alloy is increased after annealing for a short time (600 s) but decreased after annealing for a longer time at 250 °C (5000 s), which is also consistent with the change of twin boundary mobility of AZ91 alloy with annealing time. To verify whether the stress applied on individual twins during the damping test reach the critical stress for the activation of twin boundaries, the applied stress on two individual twins at the strain amplitudes (0.25% and 0.1% respectively) during damping test were calculated. The results revealed that the applied stress on the twin boundary during damping test is beyond the CRSS for motion of twin boundary, which provides theoretical basis for the contribution of mobile twin boundaries to the increased damping capacity.

To distinguish the contribution to damping capacity by the motion of twin boundaries from that by the motion of basal slip, the effect of frequency on damping capacity of AZ31 alloy was also examined. Two peaks around 50 Hz and 80-90 Hz are found in the changing curves of damping capacity as a function of frequency in pre-compressed samples with twins. It can be speculated that the peak around 50 Hz was ascribed to the motion of twin boundaries. The peak around 80-90 Hz was ascribed to the motion of dislocations when allowing for that the motion speed of twin boundaries is a little lower than that of dislocations.

Besides, the tensile strength of Mg alloys AZ31 and AZ91 are slightly enhanced after introducing twin boundaries. Therefore, Mg alloys with improved damping capacity without sacrificing mechanical properties are obtained by introducing $\{10\bar{1}2\}$ twins and subsequent annealing for appropriate time as shown in Fig.2.

Chapter 6. Conclusions

The conclusions of this study are summarized in Chapter 6. The contribution by the motion of twin boundaries to the damping capacity of Mg alloy AZ31 and AZ91 was verified for the first time. This provides a novel method to facilitate Mg alloys with both high strength and high damping capacity. The effects of solute elements on the twinning and detwinning behavior were systematically clarified.

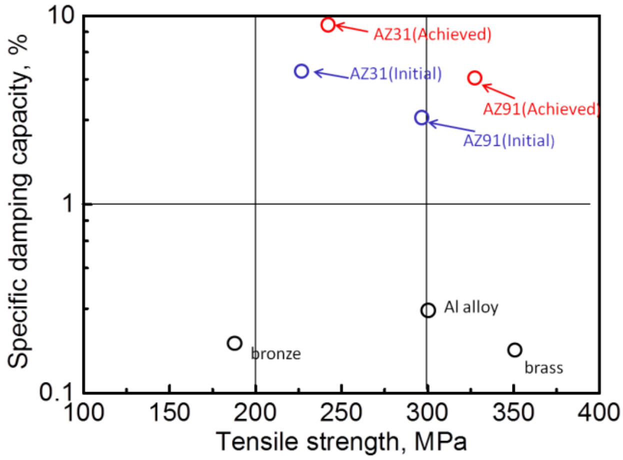

Fig. 2. Damping capacity and tensile strength of obtained AZ31 and AZ91 alloys (Achieved) compared to as-received samples (Initial) and other alloys.

論文審査結果の要旨

本論文は、高い比強度と優れた制振特性とを両立させたMg合金の開発を目的とした双晶界面移動度の制御に関する研究である。一般に強度と疲労特性を向上させると制振特性が低下する。それに対して本論文では、可逆的に移動する双晶界面を材料中に導入することで制振特性の向上が可能である点に着目し、高比強度と優れた制振特性を両立させるために双晶界面移動度の制御指針を検討している。本論文は全6章で構成されている。

第1章の序論では過去の研究—Mgとその合金の変形挙動、twinningとdetwinning、双晶界面の移動度—のレビューを行い、研究背景と研究目的を論じている。

第2章ではtwinningに及ぼす合金化の影響について論じている。本論文では溶体化処理した純Mg、AZ31合金、AZ91合金を圧縮変形させることで双晶を導入している。合金化を行うことでより微細な双晶が数多く導入されること、加工温度が高いほどtwinning挙動の歪速度依存性が大きいこと、合金化により歪速度依存性が小さくなることを明らかにしている。Twinning挙動の加工温度・歪速度依存性が溶質原子と析出物の効果により説明できることを示している。

第3章ではdetwinningに及ぼす合金化の影響について論じている。双晶界面の可逆的な移動によりdetwinningが生じる純Mgに対して、AZ31およびAZ91合金では2次双晶を形成しやすくなり、また双晶界面の存在していた場所に小傾角粒界が形成されることを明らかにしている。析出物による双晶界面移動が抑制されるため合金化により2次双晶の形成が容易になること、選択される2次双晶バリアントは整合歪とシュミット因子で説明でき、整合歪が主要な因子であることを示している。また、析出物と溶質原子の効果によりtwinning時に双晶界面から放出される転位の数が増加し、detwinning時に双晶界面に吸収される転位の数が抑制されるため、合金化により小傾角粒界形成されやすくなると考察している。

第4章では双晶界面の移動に及ぼすアニールの影響について論じている。双晶の成長を抑制しdetwinningを促進する逆応力は双晶形成時に生じ、アニールによって緩和されることを明らかにしている。また、双晶界面移動に対する摩擦応力は転位密度・双晶界面への溶質原子(AlとZn)の偏析・双晶界面に沿った析出物によって決まり、摩擦応力のアニール時間依存性の調査から純MgとAZ31では転位密度が、AZ91では双晶界面での偏析と析出物が双晶界面の移動度を決定していることを明らかにしている。

第5章では2〜4章で得られた知見を基に、可動双晶を導入したAZ31合金およびAZ91合金の制振特性を調査している。双晶を導入することで制振特性を向上可能なことを明らかにした。アニールによる制振特性の向上は双晶界面移動度の増加によって説明できることを明らかにしている。

第6章は本論文の結論である。

以上から、本論文は、マグネシウム合金において合金化元素と熱処理条件の最適化により双晶界面移動度の制御因子を明らかにし、高比強度・高制振特性を有するMg合金の開発に資する「可動双晶の導入」という新規な合金設計手法に関する論文であり、材料システム工学の発展に寄与するところが少なくない。

よって、本論文は博士(工学)の学位論文として合格と認める。