Continuous Dynamic Recrystallization in Magnesium Alloy

Arthur Galiyev[1], Rustam Kaibyshev[1] and Taku Sakai[2]

[1]Institute for Metals Superplasticity Problems, Khalturina 39, Ufa 450001, Russia

[2]The University of Electro-Communications, Chofu, Tokyo 182-8585, Japan

Keywords: hot deformation; continuous dynamic recrystallization; grain boundaries; misorientations; magnesium alloy

Abstract. Dynamic process of new grain formation was studied in a coarse-grained Mg-Zn-Zr alloy deformed at elevated temperatures. The samples were mainly compressed in air to strains of 0.1 and 0.25 at a temperature of 523K and at strain rate of 2.8×10^{-3} s^{-1}. Crystal orientations of neighboring grains and subgrains were measured using a conventional Kikuchi-line technique. Hot deformation promotes the formation of a new grain structure and a gradual increase of the misorientations of (sub)grain boundaries up to high angle ones. These results lead that the development of new grains in the magnesium alloy can occur by continuous dynamic recrystallization (CDRX). The fact that CDRX occurred in the Mg alloy with a low stacking fault energy (SFE) may be associated with operation of a specific mechanism of plastic deformation in the zones of enhanced plastic activity at and near original grain and twin boundaries. The formation of new grains can be connected with extensive cross-slip of **a** dislocations in vicinity of pre-existing high angle boundaries which could provide the formation of subgrain structure and quick misorientation increase by cross-slip of screw **a** dislocations across subgrains and accumulation into subgrain boundaries. Such behavior is associated with decreased splitting width of **a** dislocations and growing of SFE of cross-slipped dislocations that is likely to provide the development of the "high SFE metal" type of DRX in magnesium alloy.

Introduction

Mg-Zn alloys are usually considered as materials with low stacking fault energy (SFE). Due to occurrence of dynamic recrystallization (DRX) taking place in materials with low SFE, uniform fine-grained structure can be generated under hot deformation of these alloys. This leads to significant enhance in strength and ductility of fine-grained magnesium alloys [1]. In this respect, studies on fine-grain evolution are of increasing importance for producing of high strength/toughness magnesium alloys. On the other hand, there have been limited studies of microstructural evolution during DRX due to a few experimental evidence and detailed physical understanding for the nature of DRX processes taking place in magnesium alloys. This makes difficult to use of the high commercial potential of magnesium alloys.

Recently, some steps of DRX development in magnesium alloy were investigated in [2]. It was shown that new grains in magnesium alloy were generated by several mechanisms of DRX and these resulted in essential distinctions of microstructural transformations. For example low temperature DRX and continuous DRX were found to occur in magnesium alloys during deformation at $T=0.46T_m$ and $0.57T_m$, respectively, where T_m is the melting point. Further analysis of these processes is of a great importance for deeper understanding of DRX nature and successful use of DRX for control of the microstructure.

The present work was aimed to investigate the microstructures formed in magnesium alloy during deformation at a temperature of $0.57T_m$ (523K). Special attention is focused on analysis of dynamic process of new grain formation.

Experemental procedure

The samples of magnesium alloy Mg-5.8%Zn-0.65%Zr were tested in the current study. Prior to deformation the material was solution heat treated at 723K for 6h. The average grain size was 85μm and no dislocation substructure was found in the grain interiors. Compression tests were carried out on an Instron mechanical testing machine equipped with a water quenching apparatus. The latter allowed to quench the samples in situ immediately after deformation. Test samples, 12 mm in length and 10 mm in diameter, were deformed in air to strains of 0.1 and 0.25 at temperature 523K under a strain rate of 2.8×10^{-3} s^{-1}. For study of microstructural transformations optical microscopy and transmission electron microscopy (TEM) were employed. Deformation microstructures were examined on sections parallel to the compression direction. In the special set of experiments misorientations of (sub)boundaries developed during hot deformation were determined. Individual orientations of neighboring grains and subgrains were measured using a conventional Kikuchi-line technique. The method was associated with indexing of transmission Kikuchi patterns produced by a finely focused electron beam on the sample and the local determination of the crystallographic orientations.

Experimental Results

Flow behavior. Figure 1 shows typical stress-strain curves of magnesium alloy at 523K and for comparison at 423K and 623K. It is clearly seen that the shapes of flow curves change clearly at each temperature. The curve at 523K shows a steady-state flow following a pronounced work softening. Such behavior is a typical DRX type of flow, which can be considered to occur in the region of thermally activated flow. This may be supported by a rapid decrease of the peak and steady-state flow stresses with increasing temperature, which can be expressed by a power-law equation with a stress exponent of 5.7 [2]. The apparent activation energy of about 93kJ mol^{-1} is close to those for magnesium pipe diffusion and Friedel-Escaig cross-slip. In further analysis we will consider the strain effect on microstructure changes in the samples deformed to 10% and 25% at 523K. These strains correspond to the regions in which dynamic softening processes can operate frequently.

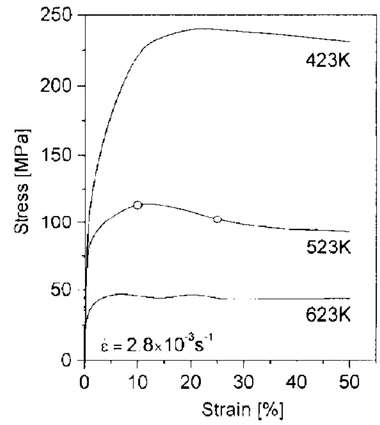
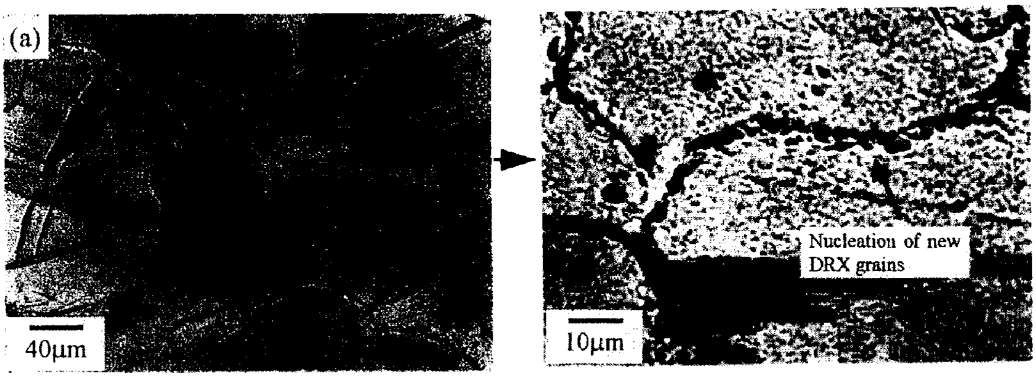


Fig. 1. Stress-strain curves of magnesium alloy. Open symbols indicate prestrains, where structure observation was carried out.

Optical microstructures. Figure 2 shows changes in the deformed microstructures at 523K. It can be seen in Fig. 2a that some grain boundaries become slightly serrated and twins are formed within few grains at a small strain 10%. More careful examination at higher magnification indicates that small equiaxed grains are frequently formed along the original grain boundaries. This indicates that DRX nucleation starts to operate before a peak strain at the boundaries of original grains.

Further straining to 25%, new equiaxed grains are developed at grain and twin boundaries, which are serrated, as seen in Fig. 2b. New grains usually form a necklace structure in coarse grains along pre-existing high angle boundaries [3]. Such a DRX structure is progressively developed from the boundaries to the centres of the deformed grains. The average grain size was about 2.5μm.



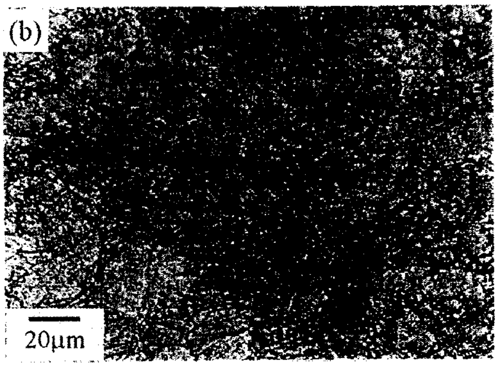


Fig. 2. Typical microstructures of samples deformed at 523K and $\dot{\varepsilon}$ =2.8×10^{-3} s^{-1}. a) ε=10%; b) ε=25%.

TEM observation. Figures 3 and 4 show typical substructures developed under deformation at 523K. It can be seen in Fig. 3a that a subgrain structure is formed in the early stage of plastic deformation. As misorientation angles of dislocation boundaries are indicated in Figs. 3 and 4, high angle boundaries (HABs) are already formed in the network of deformation induced boundaries at a small strain of 10%.

As strain increases, subgrains transform to grains with HABs, which include subgrains (Fig. 4). It can be concluded here that the networks of deformation induced boundaries gradually and continuously transform in recrystallized grains. This is supported by the fact that the size of subgrains (~2.2μm) is essentially similar to that of recrystallized grains (~2.5μm).

Orientation distribution analysis. Misorientation distributions in the areas of recrystallized grains were measured for strains 10% and 25%. Misorientation angles are represented in histograms in Fig. 5 and the rotation axes are displayed in standard triangles in Fig. 6. The misorientation

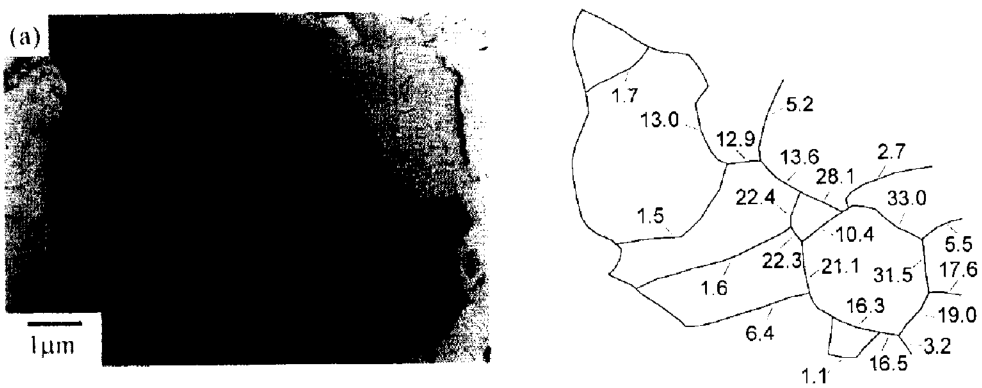


Fig. 3a. Development of subgrain structure in a sample deformed to a strain of 10% (T = 523K; $\dot{\varepsilon}$ =2.8×10^{-3} s^{-1}).

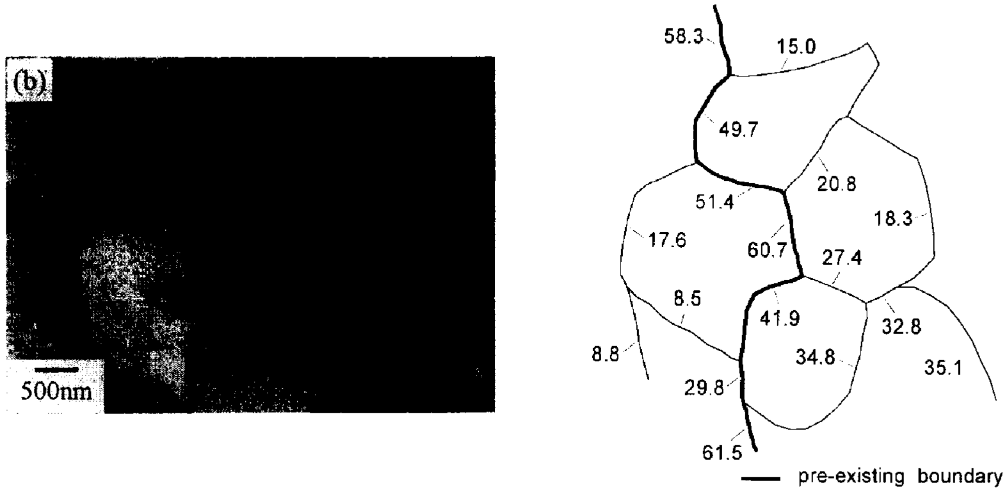


Fig. 3b. Development of new grains in a sample deformed to a strain of 10% ($T = 523$K; $\dot{\varepsilon} = 2.8\times10^{-3}$ s^{-1}).

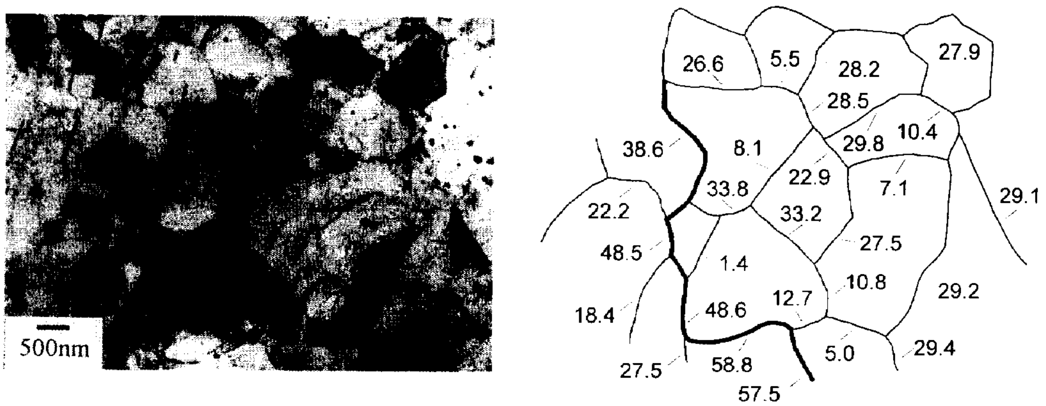


Fig. 4. Development of a new grain structure at a strain of 25% ($T = 523$K; $\dot{\varepsilon} = 2.8\times10^{-3}$ s^{-1}).

distribution at a strain of 10% reveals a preference for low angle boundaries with a wide spread to high angles (Fig. 5a). Here HABs are mainly pre-existing grain boundaries and the maximum at around 30-40° is associated with their presence in the networks of low angle boundaries. A strain increase to ε=25% leads to gradually decrease in the frequency of low angle boundaries and increase in the frequency of boundaries with higher misorientations in the mixed structure (Fig. 5b). It can be seen in Fig. 5b that a sharp maximum revealed at around 20-30° evidently results from transformation of low angle boundaries into HABs. Nevertheless, the fraction of low angle boundaries is remained relatively high. It is also worth noting that a number of boundaries with special misorientation as well as misorientations exceeded 40° are rather low. In the both cases misorientation distributions show no correspondence to the profile associated with randomly oriented grains [4]. Distributions of the rotation axes for strains of 10% and 25% are essentially similar and widely distributed in the standard triangle (Fig. 6).

Discussion

The present results show that continuous DRX occurs in a Mg-Zn-Zr alloy during deformation at 523K ($0.57T_m$). New grains are formed in the vicinity of pre- existing grain boundaries and also twin boundaries developed at lower strains. The flow curve exhibits a typical DRX type with a single peak followed by work softening.

Recently, Miura and Sakai showed [5, 6] that DRX, occurring during hot working of materials with low to medium stacking fault energy (SFE), can be generally nucleated by bulging of serrated grain boundaries. The other mechanism of DRX occurred in materials with low to medium SFE is associated with twin formation [7, 8]. However, the nucleation of DRX by bulging or twin formation was not revealed in magnesium alloy at 523K. The TEM observations show that DRX nucleation occurs in networks of deformation induced boundaries. From the comparison of distributions of misorientation angles in samples deformed to 10% and 25% (Fig. 5), it is quite evident that networks of low angle boundaries gradually transforms into HABs as deformation proceeds. This type of microstructural transformation suggests that the nucleation process is associated with continuous increasing misorientations in the networks of deformation induced boundaries, that is continuous dynamic recrystallization (CDRX) [9].

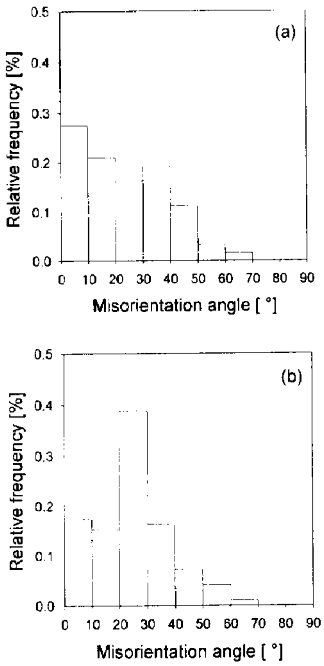


Fig. 5. Distribution of misorientation angles in the areas of newly formed recrystallized grains. (a) ε = 10%, (b) ε = 25%.

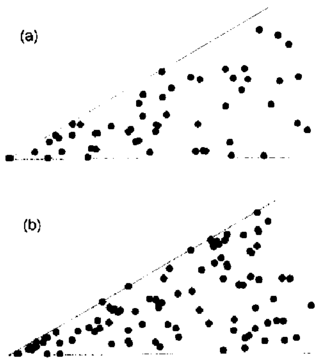


Fig. 6. Distribution of rotation axes in the areas of newly formed recrystallized grains. (a) ε = 10%, (b) ε = 25%.

It may be strange in this analysis that CDRX normally taking place in materials with high SFE occurs in the magnesium alloy, which is considered as a material with a low SFE due to the fact that SFE of **a** dislocation lying into basal plane is essentially low. To explain this disagreement, the results of previous analysis of operating deformation mechanisms [2] should be involved in the present consideration.

As shown in [2], the process controlling the plastic flow at 523K can be cross-slip of **a** dislocations on non-basal planes. This is supported by the value of the apparent activation energy close to that for the Friedel-Escaig cross-slip. The cross-slip in magnesium alloy is activated near pre-existing high angle boundaries where high stresses are concentrated and the critical resolved shear stress for non-basal slip can be attained. During deformation dislocations located near the boundaries recombine in the basal planes and transform into non-basal dislocations [10, 11]. This assumes that splitting width of dislocations in the basal planes decreases and these dislocations can cross-slip in the non-basal planes. In their turn, cross-slipped dislocations are constricted in the non-basal planes and characterized by high SFE. It is evident that cross-slip and dislocation rearrangement become much more active in the case of dislocations with reduced splitting width and high SFE.

The described ability of **a** dislocations to cross-slip at 523K is well consistent with development of CDRX. Due to cross-slip dislocations readily accumulate in dislocation boundaries in the vicinity of pre-existing high angle boundaries. Further gradual incorporation of dislocations into subboundaries with increasing strain leads to increase of their misorientations to HABs. The cross-slip of screw **a** dislocations across subgrains can also assist the evolution of grain structures. Thus, the operation of cross-slip can result in development of CDRX grain structure. In other words due to reduced splitting width of **a** dislocations and their transformation into high SFE cross-slipped dislocations at pre-existing high angle boundaries CDRX - which normally occurs in materials with high SFE – can also take place in this magnesium alloy. In reality, the cross-slip **a** dislocations can be developed in the significantly wider strip near pre-existing high angle boundaries [2]. As a result, new grains form a "necklace structure" of recrystallized grains developed to the centres of the deformed grains (Fig. 2b).

It can be concluded that the formation of new grain structure in the magnesium alloy at 523K can be a consequence of reducing splitting width of **a** dislocations and their transformation into non-basal high SFE dislocations near the pre-existing HABs. This leads to frequent operation of cross-slip and development of subgrain structure. Further gradual evolution of subgrain structure due to operation of cross-slip provides continuous nucleation of new grains and development of "necklace structure". This type of microstructural transformation indicates that the mechanism of new grain evolution in magnesium alloy at 523K can be CDRX.

Conclusion

Peculiarities of DRX process were studied in a Mg-5.8%Zn-0.65%Zr alloy at temperature 523K and at strain rate of 2.8×10^{-3} s^{-1}. Development of DRX was characterized by formation of new grains at and near original grain and twin boundaries. DRX occurred due to an extensive cross-slip of **a** dislocations near pre-existing high angle boundaries. The formation of new grains was associated with decreased splitting width of **a** dislocations and transformation of these dislocations into high SFE cross-slipped dislocations. The cross-slip of **a** dislocations provided formation of subgrain structure and quick misorientation increase by cross-slip of screw **a** dislocations across subgrains and accumulation into subgrain boundaries. This recrystallization mechanism can be CDRX.

References

[1] O. A. Kaibyshev: *Superplasticity of Alloys, Intermetallides, and Ceramics* (Springer-Verlag, Berlin 1992).

[2] A. M. Galiev, R. O. Kaibyshev and G. Gottstein: Acta Mater. Vol. 49 (2001), p. 1199.

[3] F.J. Humphreys and M. Hatherly: *Recrystallization and Related Annealing Phenomena* (Pergamon Press, Oxford 1996).

[4] H.S. Ryoo, S.K.Hwang, M.H. Kim, S.I. Kwun and S.W. Chae: Scripta Mater. Vol. 44 (2001), p.2583.

[5] H. Miura, H. Aoyama and T. Sakai: Jour. Jap. Inst. Met. Vol.58 (1994), p.267.

[6] T. Sakai and H. Miura: *Hot Workability of Steels and Light Alloys-Composites*, ed. by H.J. McQueen et al (TMS of CIM, Montreal 1996), p. 161.

[7] B. Bayle, Ph. Bocher, J.J. Jonas and F. Montheillet: Mater. Sci. and Tech. Vol. 15 (1999), p. 803.

[8] X. Wang, E. Brünger and G. Gottstein: Scripta Mater. Vol. 46 (2002), p.875.

[9] A. Belyakov, T. Sakai, H. Miura and R. Kaibyshev: Phil. Mag. Letters, Vol. 80 (2000), p. 711.

[10] B. Escaig: Phys. stat. sol. Vol. 28 (1968), p. 463.

[11] A. Couret, D. Caillard, W. Puschl and G. Schoeck: Phil. Mag. A Vol. 63 (1991), p. 1045.