Dynamic recrystallization of Zr–1Sn–0.3Nb alloy during hot compression

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Abstract. In this paper, using a hot-rolled and recrystallized Zr-1Sn-0.3Nb alloy sheet, five strain levels (10, 20, 30, 45 and 60%) of uniaxial compression experiments were performed at 700°C with a strain rate of 0.001 s⁻¹ using a Gleeble 1500D thermal simulator. The morphologies, distributions and misorientations of the grain boundaries at different strains were characterized by an electron backscatter diffraction (EBSD) technique. The EBSD maps indicated that dynamic recrystallization occurred during the high-temperature deformation. The processes, namely, grain boundary bulging, strain induced subboundaries around the bulged grain boundaries and their transformation from low misorientation to high misorientation, were considered as the formation mechanisms of recrystallized grains. At the early stage of deformation, low angle boundaries (LABs) formed in grains, the original grain boundaries become serrated, and LABs formed around the serrated boundaries. With increasing strain, the LABs transformed into high angle boundaries (HABs), leading to nucleation of new grains.

1. Introduction
Dynamic recrystallization (DRX) as one of the important mechanisms to obtain fine grains occurring under hot deformation is widely studied in cubic metals (e.g. aluminum and iron) [1-5]. Belyakov et al. [6] indicated that the DRX of the 304 type austenitic stainless steel occurred mainly through dynamic bulging of original grain boundaries under the conditions of high temperature, which can be accelerated by the progress of serrated grain boundaries and strain induced dislocation subboundaries. Furthermore, by studying the multiple compression at high strains of above 4 of a polycrystalline copper, Belyakov et al. [7] reported that the dynamic new grains generated by increasing misorientations of deformation-induced dislocation subboundaries with increasing strain, this process accompanied with dynamic recovery at 473K under a strain rate of 10⁻³ s⁻¹. However, few studies focusing on DRX in hexagonal close-packed (HCP) metals have been done. Yang et al. [8] reported that new grains generated at corrugated initial grain boundaries or the kink bands in grain interiors by increasing misorientation of kink bands boundaries to a saturation of around 43°, and the final grain size is almost the same as the average size of the regions fragmented by kink bands in deformed magnesium alloy AZ31 at 673K. By means of EBSD technique, Gerspach et al. [9] found that oriented nucleation occurred during recrystallization in the case of low strain (40%) cold cross-rolled Zr702 samples, whereas in the case of highly cold rolled Zr702 samples, non-oriented nucleation occurred during recrystallization. Additionally, by using in situ high voltage electron microscopy (HVEM), Zhu and his colleagues [10] observed that subgrains result from rearrangement of dislocation cells, and then grow to form nuclei, finally nuclei grow by high angle boundary migration. Perez-Prado et al. [11] pointed out that geometric dynamic recrystallization (GDRX) occurred during creep at...
temperatures from 400 to 800°C. This process can be described as follows: original grain boundaries become serrated resulting from subgrain formation, and with increasing deformation, “pinching-off” of the serrated grains occur, leading to fine grains.

All of the above works focused on the influence of a grain boundary on DRX in cubic metals and magnesium. There are few studies in zirconium alloys, and the dynamic softening mechanisms in zirconium alloys during hot deformation have not been well understood. In this work, cylindrical specimens of a Zr alloy were isothermally compressed to five strain levels (10, 20, 30, 45 and 60%), and the morphologies, distributions and misorientations of the grain boundaries at different strains were obtained by EBSD, in order to study the dynamic softening mechanisms.

2. Experimental

The as-received material was a hot-rolled and recrystallized Zr-1Sn-0.3Nb alloy plate of 9.5 mm in thickness. The chemical composition of the material is given in Table 1. Cylindrical compression specimens with 6 mm in diameter and 6 mm in height were cut from the plate, with the compression direction parallel to the normal direction (ND) of the original plate. In order to evaluate the evolution of grain boundary during deformation, uniaxial compression experiments were performed to five strain levels (10, 20, 30, 45 and 60%) at 700°C using a Gleeble 1500D thermal simulator at a strain rate of 0.001s⁻¹. The experimental process is shown in Figure 1. After hot compression, the deformed specimens were immediately quenched into water to retain the deformation microstructure.

| Sn   | Nb  | Fe  | Cr  | O   | Zr   |
|------|-----|-----|-----|-----|------|
| 1.06 | 0.36| 0.30| 0.10| 0.13| Bal. |

**Table 1.** Nominal composition of the Zr-1Sn-0.3Nb alloy (wt.%).

For microstructural analyses, the specimens were cut along the direction of the compression axis, and prepared by mechanically polished and electropolishing. The microstructure was characterized using a field emission gun scanning electron microscope (TESCAN MIRA 3 XMU) equipped with a backscatter electron detector and an EBSD analysis system (AZtec, Oxford Instruments). The step size of EBSD scan was 0.2μm. The initial microstructures of the hot-rolled and recrystallized Zr-1Sn-
0.3Nb alloy plate is shown in Figure 2, where the black lines and the gray lines represent HABs (> 15°) and LABs (< 15°), respectively.

3. Results and Discussion

3.1. High-temperature deformation behaviour

The true stress-strain curve is shown in Figure 3. It can be seen from Figure 3 that the flow stress decreases after reaching the maximum, which is caused by high temperature dynamic softening, and then increases slowly after a minimum. The stress increase is caused by grain refinement resulting from DRX, which will be discussed in the next section in detail.

![Figure 3. Compression true stress-true strain curve of zirconium alloy deformed to various strains at 700°C and at 0.001 s⁻¹.](image)

3.2. Effects of compression reduction on microstructural evolution

The EBSD maps of specimens at different reductions in thickness are shown in Figure 4, in which different colours represent different crystal orientations indicated in the inverse pole figure. It can be seen from Figure 4 that the reduction in thickness significantly influences the morphology of grains and grain boundaries. Compared with the original microstructure in Figure 2, the grains are elongated at the reduction of 10%. Meanwhile, the grain boundary becomes serrated, and a number of LABs appear inside the grain with a short morphology. With increasing strain, more and more fine grains are produced along the elongated grain boundaries. Such a phenomenon may be ascribed to the formation of long LABs, separating the grain into several parts. Subsequently, the long LABs caused by the reaction of short LABs transform to HABs. As the reduction increases to 60%, the grains become fine and uniform, and the serrated grain boundaries are replaced by a smooth morphology. Based on the above analysis, it can be concluded that the grain refinement can be attributed to DRX, including the formation of LABs and HABs, as well as their migration.
Figure 4. EBSD maps for samples deformed to (a) 10%, (b) 20%, (c) 30%, (d) 45%, (e) 60%. The colour is defined by the inverse pole figure. With increasing reduction, more and more fine grains are produced along original grain boundaries. As the reduction increases to 60%, the grains become fine and uniform, and the serrated grain boundaries are replaced by a smooth morphology.
3.3. **Dynamic nucleation and progress of new grains**

Figure 5a shows the EBSD map of the specimen after 20% compression. The point to point misorientation (θ) and the accumulative misorientation (Σθ) were measured along the lines L1 and L2 indicated in Figure 5a and are shown in Figure 5b and 5c. The θ represents the misorientation between two adjacent scanned points (0.2μm distance), and the Σθ represents the misorientation between the first point and a point-n. On the line L1, θ ranges from 0 to 1°, while the Σθ continuously increases from 0° to 11° along the direction indicated by the arrow, demonstrating the existence of an orientation gradient. Line L2 crosses several LABs, and θ ranges from 0 to 12°. Σθ changes discontinuously at the same positions which correspond to low misorientation peaks. It can be concluded that the generation of subgrains was caused by the orientation gradient.

Figure 6a shows the EBSD map of the sample after 30% reduction. The grains became flattened and the grain boundaries became serrated. Figure 6b shows the distribution of θ and Σθ along the line L1 in Figure 6a, Σθ continues increasing from 0° to 4.5° along the arrow direction, demonstrating the existence of an orientation gradient. In comparison with Σθ in Figure 5b and 5c, Σθ in Figure 6b is lower, signifies that the orientation gradient presence in this region is lower than that in the region crossed by line L1 in Figure 5a. Strain induced subboundaries enclosing the bulged grain boundary in this area did not evolve into HABs, which is partly due to the low orientation gradient. Another typical case of nucleation of new grains near grain boundary is illustrated in Figure 6a indicated by line L2. Miura et al. [12] pointed out the serrated grain boundaries and a non-uniform strain gradient along the boundaries were induced by grain boundary shearing. High temperature deformation is easier to make a grain boundary serrated than cold deformation. LABs formed around the bulged grain boundaries separate the bulge boundaries from the initial grain, leading to nucleation of new grains. In addition to the serration of grain boundaries, the strain induced LABs is also important.
The phenomenon that “pinching-off” of serrated grains to form fine grains in zirconium alloys proposed by Perez-Prado et al. [11] was not found in this work. Perez-Prado et al. [11] claimed that, with increasing strain, original grain boundaries become serrated, original grains elongate and became thinner in the direction perpendicular to the tensile direction. Finally, when the thickness decreases to a threshold value, grain boundary serrations come in contact with each other, grain pinching-off can occur. As a result, fine grains are formed. In this study, strain induced subboundaries in the regions which have an orientation gradient near original bulged boundaries evolve into HABs with increasing strain, finally leading to nucleation of new grains in these regions. This phenomenon is similar to the rotational recrystallization (RRX) in which the subgrains around original grain boundaries transform into HABs, partly due to the presence of high local stresses [13]. Loge et al. [14] claimed that, at temperatures higher than 600°C, DRX occurs at a high strain rate, while dynamic recovery predominates at low strain rates in Zircalloy-4.

As a conclusion in this study, the structural evolution is characterized by various types of strain induced subboundaries evolved around serrated original grain boundaries. This process can be described as follows: firstly, LABs are formed in grains by rearrangements of dislocations in the initial stage of deformation. Secondly, with increasing strain, the original grain boundaries become serrated, a strain gradient forms around grain boundaries because of grain boundary shearing. Thirdly, strain induced LABs form around the serrated boundaries resulting from the strain gradient. Finally, as the misorientation increases by the accumulation of the dislocations, the LABs transform into HABs. Hence, the recrystallized grains form in the deformed grains. This process is illustrated in Figure 7.
4. Conclusions

Based on the EBSD observation and analysis, the deformation behavior and structural evolution of a zirconium alloy compressed at 700°C at a strain rate of 0.001s⁻¹ were studied. The main results can be summarized as follows:

1. With increasing strain, more and more fine grains are produced along the boundaries of elongated grains.
2. The structural evolution is characterized by various types of strain induced subboundaries evolved around serrated original grain boundaries. At the early stage of deformation, LABs form within grains, and the original grain boundaries become serrated. The LABs formed around the serrated boundaries, gradually transform into high angle grain boundaries, leading to nucleation of new grains.

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