FORMATION OF SUBMICROCRYSTALLINE STRUCTURE IN MATERIALS DURING DYNAMIC RECRYSTALLIZATION

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ABSTRACT

Mechanical properties and structural changes during plastic deformation in the region of low homologous temperatures (T=0.3-0.5T) of Mg-6%Zr alloy with matrix type structure and Ti-6%Al-3.2%Mo alloy with lamellar structure are investigated in this paper. It is shown that the development of dynamic recrystallization in both materials leads to the formation of submicrocrystalline structure with grain sizes of 50-500 nm. Differences in the effective mechanism of dynamic recrystallization are due to differences in the character of crystallographic slip.

INTRODUCTION

The dependence of the size of dynamically recrystallized grains on the value of the Zener-Hollomon parameter Z suggests the possibility of submicrocrystalline structure formation, the grain size amounting to tenths of micrometer. Thus, in nickel and copper microstructure with a grain size of 0.1μm is formed after deformations up to superhigh strains e=4-7 at low homologous temperatures of 0.2T_m (large values of Z) [1].

Two main questions arose after the analysis of recrystallization peculiarities at so low temperatures of deformation. 1. Since the mechanisms of plastic deformation affect the development of dynamic recrystallization [2], and with the sharp decrease of plastic flow temperature from the usual range for dynamic recrystallization (0.5-$0.9T_m$) are there any changes observed in the mechanisms of recrystallization as well? 2. What minimum grain size value can be obtained in the course of dynamic recrystallization? There seem to be no investigations of these problems in the literature.

The purpose of the present study is to investigate the structural changes occurring in materials during plastic deformation up to superhigh strains at low temperatures. A magnesium Mg-6%Zn-0.65%Zr alloy with matrix type structure and a two-phase titanium Ti-6%Al-3.2%Mo alloy with lamellar microstructure were used for the investigation. The magnesium alloy is known to be used [2] for easy identification of the effective slip systems, especially taking into account the transition to single slip on the basal plane with decreasing deformation temperature, while the comparison of the data obtained on the titanium alloy, where multiple slip prevails within a wide temperature interval, allows to establish the effect of the deformation mechanism changes on the development of recrystallization.

MATERIAL AND EXPERIMENTAL PROCEDURE.

A magnesium alloy ingot with an initial grain size of 85μm subjected to quenching from 450°C, was used to prepare the specimens. Dispersed globular particles of the Zr_3Zn_2 phase (their volume fraction amounting to 5%) were present in the microstructure. The specimens were deformed at room temperature ($0.3T_m$) on a Bridgman anvil-type unit [3]. The latter was used because of the low ductility of the alloy. The microhardness of specimens subjected to deformation with strains $e=0.5$-7 was measured.

The titanium alloy was annealed in the β-region and the β-transformed grain size amounted to 400μm. The α-plates were 1-2μm thick, while the β-plates were 0.1-0.2μm thick, the volume fraction of the latter phase being 9%. The specimens were deformed by upsetting on an Instron-type machine at temperatures between 500°C and 800°C (0.4-$0.6T_m$) with subsequent quenching after a 0.3 s delay.

Specimens for tensile tests were cut from work pieces forged at the same temperatures. The value of the strain rate sensitivity exponent $m = d\lg\sigma / d\lg\dot{\varepsilon}$ was determined by the rate-change method.

Optical and electron microscopy were used to analyze the alloy structure. Size and volume fraction of the recrystallized grains were calculated by the method of random secants. The azimuthal angle was used to estimate the misorientation degree of the Mg alloy nonrecrystallized grains [1, 4]. A topographical study was carried out on the Mg alloy specimens deformed in tension: they were cut out from the work pieces obtained on the Bridgman anvil-type machine.

RESULTS

a) Mg-6%Zn-0.65%Zr alloy

The alloy microstructure undergoes considerable changes in the course of deformation at room temperature. At the initial stage ($e=0.5$) the original grains are elongated in the deformation direction and the bending of boundaries is observed. A part of the grain boundaries is not observable metallographically. At the large strains $e=4$-7 the original grain boundaries are no longer detectable in the microstructure.

The sharp increase of dislocation density in the [0001]<1120> system has been discovered by electron microscopy. Dislocations of other systems have not been observed at any strains. At $e \geq 0{,}5$ the formation of flat pile ups of dislocations of high density ($\rho = 3\ 10^{11}\ cm^{-2}$) (Fig 1) is observed. They represent microregions of grain size 0.1-0.3 μm. Besides, dislocation-free sections, surrounded by boundaries which give an extinction contrast, are also seen. Their appearance was observed both in the near boundary regions and the grain interiors. The analysis of diffraction microscopic pictures enables us to establish them as recrystallized grains of 0.1-0.2 μm in size. An increase in strain leads to an increase in the recrystallized volume fraction (Fig. 2). If at e=1 it was equal to 4%, at e=4 - 37%, and at e=7-90%. The size of the new grains does not depend on the strain. Large internal stresses are present in the recrystallized regions. Misorientations in separate parts of the original grains were observed. It turned out that the orientation in non-recrystallized regions smoothly changed from one grain part to another though the total misorientation reached a few tens of degrees. The increase of strain turned out to result in the enhancement of misorientation within grains (Table 1). As seen, the values of the azimuthal angle of misorientation scattering in the non-recrystallized parts weakly changed with strain, and in this case they considerably differed from those of the recrystallized ones. The latter indicates that sharp changes of misorientation occur during recrystallization.

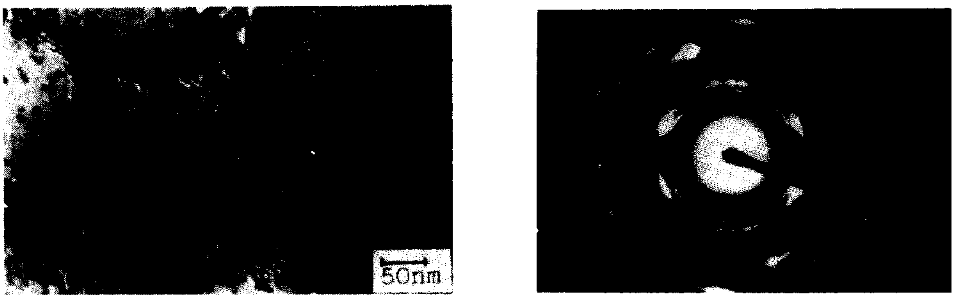

Figure 1. Fine structure and electron diffraction pattern of Mg-6%Zn-0.65%Zr alloy after deformation at room temperature up to e=0.5.

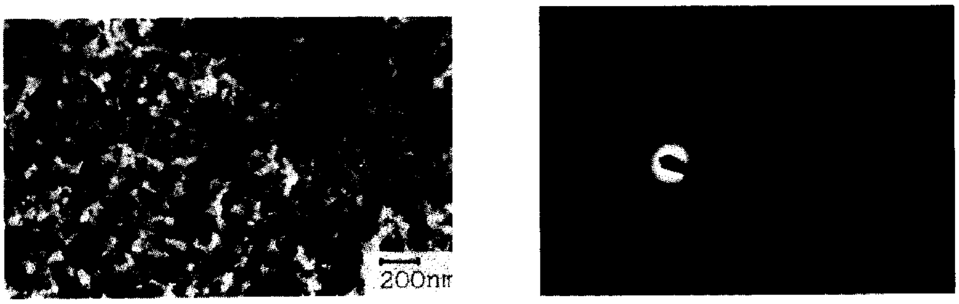

Figure 2. Microstructure and electron diffraction pattern of Mg-6%Zn-0.65%Zr alloy deformation at room temperature up to e=7.

Table 1. Strain effect on mean azimuthal angle of misorientation scattering.

Structure segment	Strains			
	e=0.5	e=1	e=4	e=7
nonrecrystallized	4.5	10-16	7-15	10-18
recrystallized			24(19-36)	homogeneous distribution

The surfaces of specimens cut out from ingots strained to e=1 on the Bridgman anvil were topographically investigated to the effect that in the non-recrystallized sections only basal slip was observed to take place (Fig. 3).

b) Ti-6%Al-3.2%Mo alloy

Fig. 4 shows σ-ε curves for the alloy subjected to deformation at 600-800^{O}C with an initial strain rate of 5 $10^{-4}s^{-1}$. With decreasing deformation temperature the value of the σ peak on the curves sharply increases, while the extent of steady state flow is reduced. At lower deformation temperatures the specimens failed when the strain reached 20%.

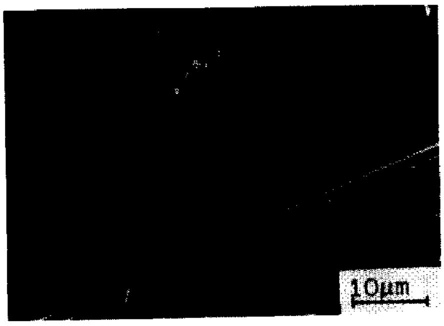

Figure 3. Deformation relief of surface in Mg-6%Zn-0.65%Zr alloy specimen after tensile straining at 20^{O}C.

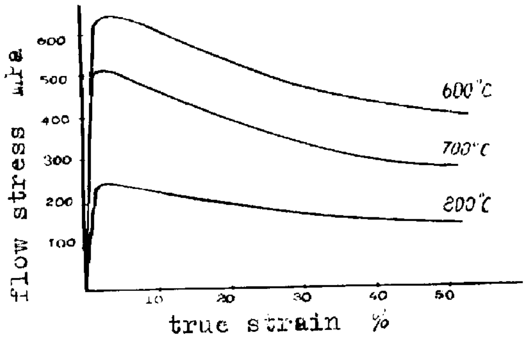

Figure 4. Stress strain curves of Ti-6%Al-3.2%Mo alloy at $\dot{\varepsilon}$ =5 $10^{-4}s^{-1}$ and T=600-800^{O}C.

Microstructural analysis showed that transformation of the initial lamellar microstructure into an equiaxed one occurred at all test temperatures. The formation of a submicrocrystalline structure with grain sizes of 0.1 and 0.4μm took place at deformation temperatures of 600 and 700^{O}C, respectively. The microstructure evolution was investigated at 700^{O}C. It was established metallographically that a texture was induced by rotation of the α- and β- phase plates up to ε=50% in the deformation direction. But, in the course of subsequent deformation the texture was blurred, and the formation of equiaxed precipitates was observed.

Changes in the fine structure of both phases were observed. The dislocation density increased with strain. At ε=15%, subboundaries with a grain size of 0.3-0.4μm were formed in the α-phase - both in the vicinity of the interphase boundaries and inside plates. After 50% straining, equiaxed grains of 0.3-0.5μm in size were discovered in the α-phase, their volume fraction increasing with ε. After 15% straining, transverse subboundaries were formed in the β-plates. At ε=50%, high angle, interphase and intergranular boundaries were formed, extinction contours on these surfaces indicating their nature (Fig. 5). At the place where the transverse boundaries emerge at the interphase surface grooves and, in many cases, the displacement of β -phase fragments relative to each other can be seen. At 75% strain only separate dislocations were observed in the grains.

Alloy specimens with an original grain size of 0.2μm were strained at 500^{O}C and $\dot{\varepsilon}$ =5 $10^{-4}s^{-1}$ up to ε =80% in order to produce still finer grains. As a result, a microstrucutre with a grain size of 0.05μm was formed (Fig. 6). Subgrains amounting to 0.05-0.06μm in size were observed at lower strains in the nonrecrystallized segments.

Specimens with a grain size of 0.6μm were tension tested at 550^{O}C and $\dot{\varepsilon}$ =1.0 $10^{-4}s^{-1}$. Values of m=0.3, δ=260%, and T=205 MPa were obtained.

Fig. 5. Microstructure of Ti-6%Al-3.2%Mo alloy after deformation at $\dot{\varepsilon}$ =5 $10^{-4}s^{-1}$ and T=700^{O}C up to ε=5%.

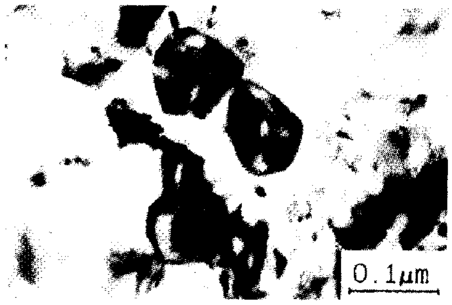

Fig. 6. Microstructure of Ti-6%Al-3.2%Mo alloy after deformation up to ε=80% at T=500^{O}C.

DISCUSSION

The data obtained indicate that dynamic recrystallization occurs in the course of plastic deformation at low homologous temperatures (0.3-0.5T) in both the magnesium and titanium alloys. But unlike the behaviour under high temperature testing conditions, in this case, considerably higher values of critical strain are required for the onset of dynamic recrystallization. Yet, the kinetics of this process are, on the whole, analogous to those of high temperature dynamic recrystallization. Unusual is the fact that the character of crystallographic slip (considerably different in the two alloys under investigation) affects the mechanism of dynamic recrystallization. Indeed, the main mechanisms of grain formation during high temperature dynamic recrystallization are the following: the local migration of the original high angle boundaries and the transformation of subboundaries into high angle ones, the latter process being the result of interaction with lattice dislocations. With decreasing deformation temperature, diffusion processes are impeded, and, evidently, the migration of original grain boundaries is hindered, hence, the bulge mechanism becomes less and less possible.

In this case, one can expect the second mechanism to operate, which is associated with an increase in the subboundary misorientation up to the high angle one, which (as the results obtained show) induces the formation of new grains in the titanium alloy. Meanwhile, unlike the latter, during low temperature dynamic recrystallization in the magnesium alloy, it is not multiple slip, but single slip on the basal plane that is observed, which is why the formation of subboundaries seems impossible. This can be related to the fact that with decreasing non-basal and basal planes increases from 4 to 100 [5, 6]. At $0.3T_m$, nonconservative and transverse dislocation slips are absent. That is why only flat pile ups of dislocations are formed: they create a continuous change of misorientation in the original grains. Since dislocations are practically immovable in such pile ups, increasing deformation results in growth of the tensor density of dislocations and, accordingly, in an increase in the misorientation in separate fragments of the original grains. From the investigation of the mean azimuthal angle of misorientation scattering in the recrystallized and nonrecrystallized segments (Table 1), it can be assumed that the formation of high angle boundaries is evidently due to the spasmodic restructuring of flat dislocation pile ups under the influence of powerful internal and external stresses.

Low temperature dynamic recrystallization induces the transformation of the titanium alloy lamellar microstructures into the equiaxed ones. In comparison with high temperatures of plastic flow no significant changes are observed in the mechanisms of transformation of the phase shapes [7]. Transverse subboundaries are formed in the β-phase during deformation. Interaction of the lattice dislocations with the transverse subboundaries, as well as, with semicoherent interphase boundaries leads to their transformation into general boundaries. Grain boundary sliding and diffusion processes which are under development on these general boundaries, are responsible for the formation of the equiaxed microstructure.

The results obtained indicate that microstructures with grain sizes in tenths and, even, hundredths of a micrometer can be obtained in materials by means of dynamic recrystallization. Low deformation temperatures and the resultant restriction of the migration of grain boundaries allow the size of dynamically recrystallized grains to be controlled easily. In this connection, conditions are created for the formation of microstructures comparable in grain size with the minimum possible subgrain value, i.e. nanocrystalline microstructures. As is known [8] nanocrystalline structures are distinguished for a number of high physical and mechanical properties. Among them the possibility of a sharp decrease in the super-plasticity temperature is of special interest. Thus, in titanium alloys with a grain size of 0.06μm it has been decreased to 550^{O}C, which is 300-350^{O}C below the lower super-plasticity boundary of an ordinary fine-grained material [9].

CONCLUSIONS

Low temperature plastic deformation up to high strains is accompanied by dynamic recrystallization in materials with both matrix type structure and lamellar structure. Mechanisms of low temperature dynamic recrystallization depend on the character of crystallographic slip. It is shown that the development of low temperature dynamic recrystallization results in the possibility of nanocrystalline structure formation.

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