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Dynamic stored energy gradient induced oriented growth of subgrains in rapidly hot-extruded tungsten heavy alloys

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ABSTRACT

Highly oriented lath-like subgrains decorated by numerous dislocation tangles and dislocation-free irregular grains were observed to prevail in a heavily hot-extruded fine-grained 93 W-4.9Ni-2.1Fe (wt.%) alloy. Except for such lath-like subgrains, there were also a considerable number of equiaxed strain-free grains visible in an extruded coarse-grained 93 W-4.9Ni-2.1Fe alloy. A mechanism in terms of a new concept designated as *dynamic stored energy gradient* was proposed to illustrate the formation of the lath-like subgrains that were actually created in extremely short time (only around 0.3 s) during hot extrusion. Due to the limited extrusion time, thermally activated diffusion of tungsten atoms was unlikely to play a decisive role in this process. It is found that there has been a significant driving force gradient at the growth front of the subgrains, which is believed to substantially promote directional annihilation of edge dislocation dipoles to develop the lath-shaped configuration of the subgrains in the extruded alloys.

1. Introduction

A great deal of research has been conducted on microstructural evolution of metallic materials during high-temperature working operations, such as extrusion, rolling, forging and so forth. During these processes, dynamic restoration including dynamic recovery (DRV) and dynamic recrystallization (DRX) were reported to actively involve, resulting in a series of microstructural partial reconstructions, for instance, annihilation and rearrangement of dislocations, development of subgrains, emergence of new recrystallized and strain-free grains, and formation of crystallographic textures [1–4]. The properties of materials after hot deformation are strongly dependent on the extent of the dynamic restoration imposed to the hot-worked microstructure. Thus, it is of great practical significance to get insights into underlying mechanisms of the DRV/DRX related microstructural evolution during hot working for manufacturing optimization. Over the past several decades, even though numerous theories have been proposed for modeling DRV/DRX and predicting microstructural development during hot working [5–10], the mechanisms of the dynamic restoration and resultant microstructural characteristics still have not been thoroughly understood.

Liquid-phase sintered tungsten heavy alloys (WHAs), a typical kind of duplex phase composite materials composed mostly of spherical tungsten particles plus a small amount of a low-melting point matrix phase such as a eutectoid of Ni, Fe and Co, have been extensively deformed at intermediate or elevated temperatures by rotary swaging [11], hydrostatic extrusion [1] and rapid hot extrusion [2] to further improve sintering density and introduce strain hardening. During these treatments, it was reported that both DRV and DRX were involved to bring interesting and abstruse modifications of microstructures. Particularly, lath-shaped or elongated subgrains parallel to the extrusion direction were generated throughout the tungsten phase, which is thought to have a strong effect on mechanical properties especially penetration performance of tungsten heavy alloys [1,2]. Nevertheless, relevant physical mechanisms on the formation of such highly oriented subgrains have seldom been stated explicitly in the literature and that it cannot be explained merely on the basis of the proposed models [5–10].

In the present study, liquid-phase sintered 93 W-4.9Ni-2.1Fe alloys with two grain sizes of 20 μm and 45 μm were rapidly extruded at 1100–1150 °C with a very high extrusion velocity more than 100 mm/s (the extrusion duration was less than 0.3 s) and an extrusion ratio of around 3.33:1. Large quantities of the lath-shaped subgrains with an average width of several hundred nanometers were observed by transmission electron microscopy (TEM) to exist in tungsten phase. The goal of this paper was to reveal the essence of the phenomenon of the lath-shaped subgrains oriented growth within so short extrusion time. A model in terms of the new concept termed as *dynamic stored energy gradient* was proposed to elucidate the possible physical mechanism. Furthermore, the influence of original grain size on the extent of DRV and DRX was discussed as well.

2. Experimental

The raw materials used to prepare the fine-grained (~20 μm) and coarse-grained (~45 μm) 93 W-4.9Ni-2.1Fe alloys were nanocrystalline

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W-Ni-Fe composite powders and commercial W, Ni and Fe powders respectively. Both powders were shaped into cylindrical compacts using cold isostatically pressing, followed by liquid-phase sintering at 1460 °C for 1.5 h in the case of the fine-grained alloy and at 1490 °C for 2 h in the case of the coarse-grained alloy. Subsequently, the sintered alloys were annealing-treated in vacuum at 1200 °C for 2 h. Rapid hot extrusion technique was employed to thermo-mechanically process the two alloys at 1100–1150 °C with a very high velocity of >100 mm/s (extrusion duration <0.3 s) and an extrusion ratio of around 3.33:1. After the treatment, tungsten particles were severely elongated along the extrusion direction [2]. The strain rate was estimated to be 10–20 s^{-1}. The microstructure of the as-extruded alloys was characterized by TEM (JEM-3010) in detail. More information on synthesis, characteristics and liquid phase sintering of the nanocrystalline composite powders, and specification of the rapid hot extrusion technique can be available in our previous works [2,12].

3. Results

Fig. 1 represents that abundant long lath-shaped subgrains have been produced in the tungsten phase of the fine-grained 93 W-4.9Ni-2.1Fe alloy after rapid hot extrusion, the width of which varies from 100 nm to 600 nm. Selected area electron diffraction (SAED), covering a small region containing two adjacent subgrains, shows that it consists of two sets of diffraction patterns slightly mismatched by a small angle of 6 to 7°. This suggests that the common boundary of the two grains should be a low-angle grain boundary (LAGB). Meanwhile, it also excludes a probability that the lath-shaped subgrains are "shear bands", because in a general case shear bands in a monocrystalline are of non-crystallographic orientation. Such organized microstructure is considered to be a main consequence of DRV, including some typical physical phenomena such as dislocation annihilation, regeneration and rearrangement. Even though there are a number of dislocation tangles randomly distributed in the subgrains, some dislocation-free areas are still visible. The crystallographic orientation between the dislocation-free area and dislocation-rich area was also identified by SAED. It is verified that they are misoriented approximately by 20°. Thus, these dislocation-free areas are most likely to be dynamically recrystallized grains due to the fact that grain boundaries produced after dynamic recrystallization usually are high-angle grain boundaries (HAGBs).

Unlike new equiaxed grains generated due to static recrystallization occurring in high temperature annealing, the recrystallized grains in this study are pretty irregular in configuration, as marked with yellow areas in the schematic profile of Fig. 2. In particular, the growth of the fresh recrystallized grains did not sweep away the boundaries so that they were confined inside each subgrain. All these microstructural features implicate that the driving force for DRX is anisotropic and it is not large enough to update all the strained structures in the extruded alloy. Additionally, as shown in Fig. 2, some subgrain triple junctions are visible in the microstructure. To lower the system energy, the triple junctions normally would have tended to be 120° relative to one another if the strain energy in the alloy had been homogeneous. However, the angles of the subgrain triple junctions in Fig. 2 evidently indicate that they are not under the condition of the lowest energy state. This further demonstrates that the driving force for DRV and DRX during hot extrusion has been inhomogeneous and the maximum should be in the growth direction of the subgains, i.e., the extrusion direction.

Compared to the slight DRX involved in the fine-grained 93 W-4.9Ni-2.1Fe alloy during hot extrusion, it seems that much more intense DRX took place in the coarse-grained alloy, which can be confirmed by the presence of equiaxed strain-free grains in Fig. 3(a). It can be seen that the boundaries of these grains are more distinguishable. Cell structures that have not yet evolved into new grains are also observed in Fig. 3(b). The microstructural distinction between the two alloys might suggest that the extent of DRV and DRX is grain-size dependent. In other words, for the fine-grained alloy, the development of microstructure during hot extrusion is dominated by DRV, whereas DRX is more obvious in the coarse-grained alloy.

4. Discussion

4.1. Possible physical mechanism

As mentioned above, one of the most interesting microstructural features is that all the subgrains are oriented towards a specific direction nearly parallel to the extrusion direction. The development of the subgrains is closely in relation to activities of screw and edge dislocations. The behavior of screw dislocations is dependent on stacking fault energy (SFE) and a high SFE enables them to be more mobile [13,14]. Tungsten is a well-known high SFE metal and hence DRV is prone to occur during hot deformation. Nevertheless, a complex non-planar core structure of screw dislocations increases the resistance to glide and consequently the required activation energy (2 eV) is 4 to 10 times higher than that of edge dislocations (0.2–0.5 eV) [15], giving rise to a much worse mobility of screw dislocations in comparison to edge dislocations. It should be further pointed out that the possibility for screw dislocation to cross slip is more or less identical in three dimensions (In b.c.c metals, the screw dislocation with $\mathbf{b} = 1/2 <111>$ can glide on three {110} planes and three {112} planes [13]). Due to these inherent characteristics, it is justifiable that screw dislocations preferably make more contributions to random dislocation structures

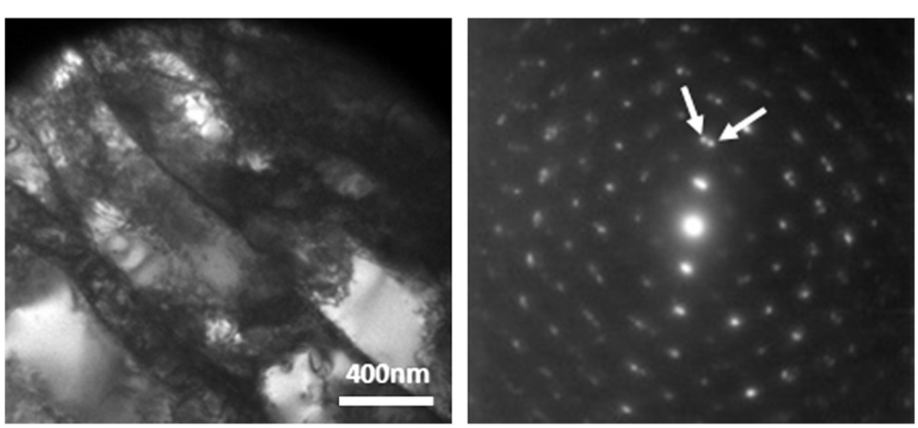

Fig. 1. TEM bright-field micrographs (a–d) of the fine-grained 93 W-4.9Ni-2.1Fe alloy after extrusion showing that there are numerous lath-shaped subgrains in tungsten phase. SAED at two adjacent subgrains indicates that the subgrain boundary is low-angle grain boundaries.