Nanotwins Formation in Accumulative Roll-Bonded Brass

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Abstract: Accumulative roll-bonding (ARB) is a severe plastic deformation process that uses rolling to produce ultrafine grains in coarse grained metallic materials. In this study, ARB has been applied on 70/30 brass up to 6 cycles at ambient temperature and non-lubricated conditions to apply a true strain up to 4.8 Von Mises strain. Microstructures of ARBed brass samples were characterized by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The results indicated that during ARB cycles, the grain size decreased from micron-size to nano-size and mechanical twins were widely observed throughout the microstructure after cycle 1. However, after cycle 3, the twinning activity became significantly limited and deformation occurred via shear bands formation. After cycle 6, the measured average grain size was about 50 nm and nanotwins were observed originating from grain boundaries and gain boundary junctions. With the reduction in the grain size down to nanometer, the pole mechanism was not the dominant mechanism of nanotwin formation and nanotwins were mainly produced via partial dislocation emission from grain boundaries and grain boundary junctions.

1. Introduction

Generally, the ultrafine-grained (UFG) metallic materials produced by applying severe plastic deformation (SPD) have high strength but low ductility. The low ductility of UFG metallic materials is attributed to the lack of strain hardening due to small grain size and saturated dislocation structure. By lowering the stacking fault energy (SFE) of copper alloys, both dislocation accumulation and twin accumulation could be activated leading to higher ductility while simultaneously improving the strength [1]. A review of the literature shows that in face centered cubic (FCC) materials, deformation mechanisms such as the mechanical twinning vary significantly with the grain size. For example, in materials with a medium to high SFE and coarse grains (CG), the pole mechanism [2] is the fundamental twinning mechanism, whereas partial dislocation emission from grain boundaries (GBs) and GB junctions controls the mechanical twinning in most of nanocrystalline (NC) materials [3-4, 6]. Relatively, large plastic deformation prior to mechanical twinning is needed in both CG and NC materials [3].

In the present work, accumulative roll-bonding (ARB) process as one of the most promising methods to conduct SPD [7] was used to produce nano-grains in 70/30 brass. During SPD of brass, partial dislocation emission from grain boundaries would improve mechanical properties [8]. Although numerous investigations have been carried out on the microstructural evolutions of ARBed metals, limited studies have been on low SFE metals such as brass. The aim of current study, therefore, is to determine the mechanism of mechanical twinning in NC brass. Furthermore, the effect of grain size on the mechanism of mechanical twinning will be examined.

2. Experimental procedure

Strips with dimensions of 300 mm×30 mm×1 mm were cut from an annealed sheet of 70/30 brass alloy having an initial grain size of 25 μm. The cut strips were then degreased (in acetone), wire brushed, stacked on top of each other, and then roll bonded to reduce the thickness by 50% (corresponding to a Von Mises equivalent strain of 0.8 per cycle). Rolling was carried out at room temperature without lubricating. The diameter of roll and peripheral used speed were 127 mm and 6 m/min, respectively. The roll-bonded sample was then cut into two sheets of approximately initial dimensions and the whole procedure was repeated up to a total of six cycles so that an accumulated equivalent strain of 4.8 was reached. The microstructure studies were done using a Philips XL30 SEM and Philips CM200 TEM. The TEM specimens were prepared by mechanical and jet polishing with an electrolyte consisting of 25% phosphorus acid, 25% alcohol and 50% distilled water (by volume) that was cooled in an ice bath.

3. Results and Discussion

The TEM micrograph of deformed microstructure of one-cycle ARBed 70/30 brass is given in Fig. 1a. Deformation in brass alloys occurs initially by the glide of dislocations followed by the mechanical twinning. The corresponding selected area diffraction (SAD) pattern confirms the presence of mechanical twins. The SAD pattern analysis is given in Fig. 1b in which the small shift of some spots in diffraction pattern is caused by a great number of parallel mechanical twins produced during deformation. Additionally, a mixed structure consisting of regions with fine mechanical twins and regions with elongated cells are observed in Fig. 1. At first, the mechanical twins were not uniformly distributed in the individual grains and they appeared as clustered bands. The frequency of the mechanical twins in many of the grains increases to such an extent that the individual clustered bands can no longer be distinguished. The mechanical twins often occur on two deformation systems, but after 50% reduction in thickness, most of the mechanical twins in any grain are confined to a single system that tends to rotate towards the rolling plane with any further deformation [9].

The mechanism of mechanical twinning in coarse grained FCC metals is known to be the pole mechanism as established by Venables [2]. This phenomenon requires a high critical resolved shear stress that can usually be achieved in 70/30 brass. In Fig. 1, the mechanical twins divide the grains into twin/matrix lamellae. Lamellae are twin areas and austenitic matrix. The white areas are matrix and the black areas are twins.

As deformation proceeds, new mechanical twins were activated to accommodate deformation and the twin density increased gradually until it became saturated. When the twin density became saturated, it was difficult for new mechanical twins to nucleate and grow.

Therefore, strain shear banding was activated in some local areas of great stress such as high density twin areas to accommodate further deformation. Inside high density twin areas, the intense shear deformation would cause the transformation of twin/matrix lamellae into nano-grains (NGs) at higher cycles of ARB [10]. Figure 2 shows the shear bands formed in 70/30 brass after two ARB cycles. It should be noted that in low SFE alloys, e.g. brass, the microstructural features that appear during large deformation (such as the ARB) have been defined as slip/twin panels, microbands and shear bands, respectively. Generally, slip and twin panels as well as microbands are usually 0.1 to 0.2μm thick and traverse an entire grain. However, shear bands are independent of grain orientation and at high strains such as ARB, they traverse the entire thickness of the rolled sheet.
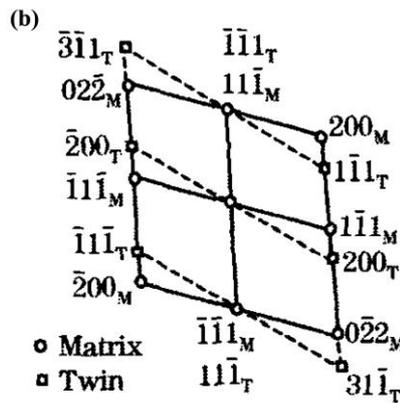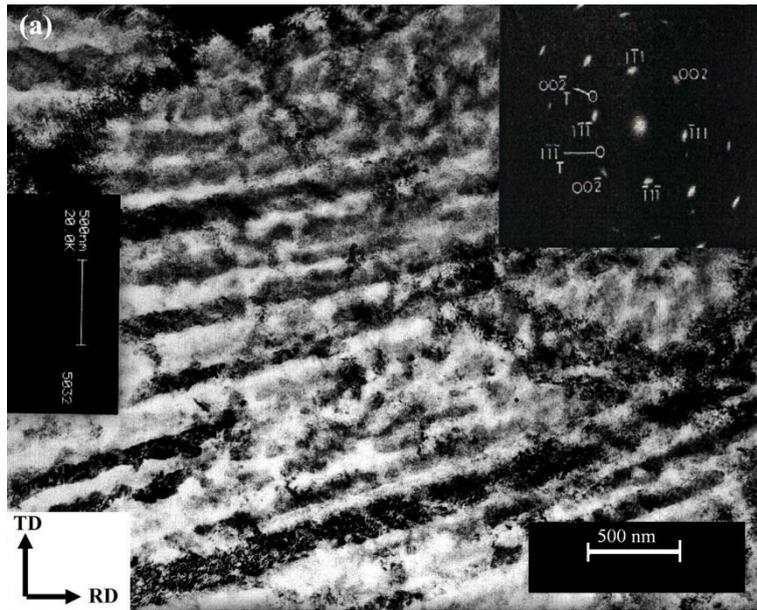
Fig. 1. a) TEM micrograph of the dislocations and mechanical twins in 70/30 brass after one ARB cycle (RD= Rolling Direction, TD= Transverse Direction) and b) diffraction analysis.
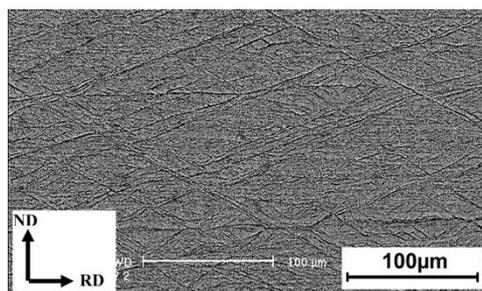

Fig. 2. Cross section SEM micrograph illustrating the shear bands
in 70/30 brass after two ARB cycles.

The microstructure of the three-cycle ARBed brass sample is shown in Fig. 3. The majority of grains were equiaxed, ultrafine with very few and localized twin/stacking faults. Limited number of twins/stacking faults observed in Fig. 3 implied that the twinning mechanism was strongly dependent on the grain size and became very difficult to occur with reduction in the grain size. The limited twin activity in the three-cycle ARBed brass sample would be explained by the formation of ultra-fine grains. According to the pole mechanism, the critical twinning stress follows the Hall–Petch relationship, in which the slope for twinning (K_T) is significantly larger than that for slip (K_S).

$$\sigma_t = \sigma_0 + \frac{K_T}{\sqrt{L}} \qquad\qquad (1)$$

where σ_t is the critical twinning stress, σ_0 is the material constant, and L is the homogenous slip length. With decreasing homogenous slip length, L, twinning stress, σ_t, increased to a power law ($\propto L^{-K}$, with $K = 0.89$) where L is directly related to the grain size and dislocation density [3]. Fig. 4 shows a TEM micrograph of the six-cycle ARBed sample. The average grain size in this sample was approximately 50 nm with a large number of nanotwins nucleated mostly at GBs or GB junctions and extended into the grains interior. Non-equilibrium nanotwins could be originated from GBs and GB junctions [3, 12]. While the twin boundaries visible in CG materials are normally atomically flat [13], nano-twins in Fig. 4 possess steps along their length. This difference with respect to twin boundary morphologies is likely due to the different twin formation mechanisms operating in coarse-grained and nanocrystalline materials.
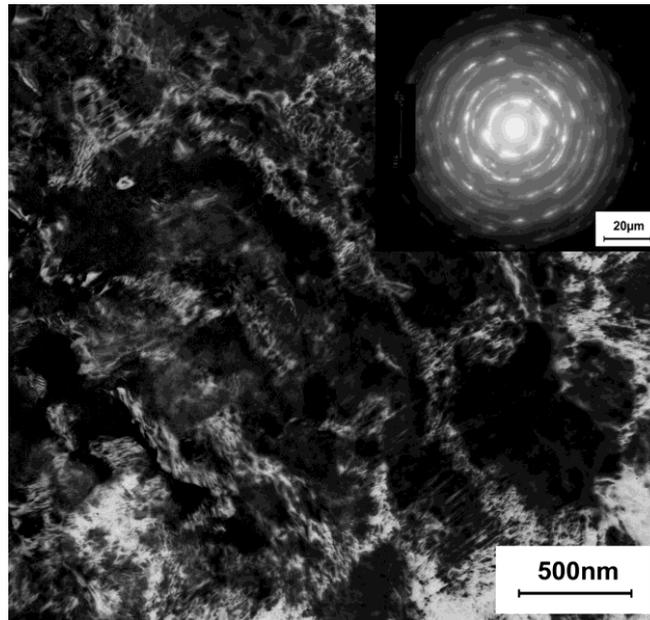

Fig. 3. TEM micrograph showing the limited twin density in 70/30 brass after three ARB cycles.
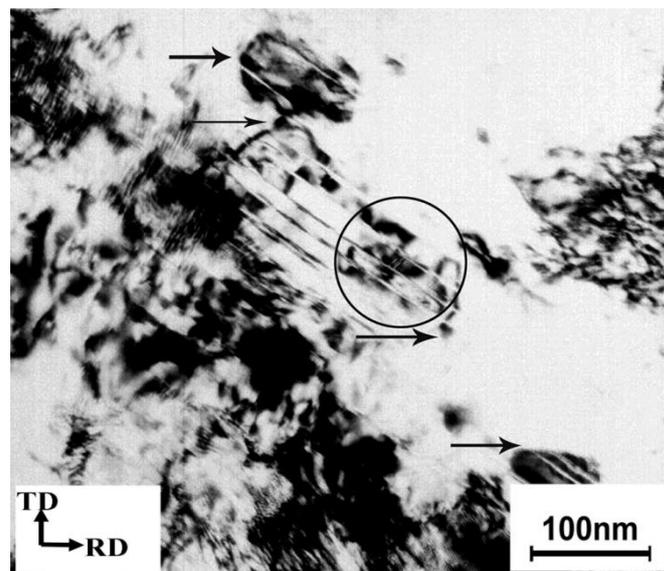

Fig. 4 TEM micrograph illustrating nanotwin formation in nano-grains, the circled region displaying nanotwins with a "stepped" morphology.

Accommodation of severe plastic deformation involves the activation of dislocations in addition to the nano-twins. Following the formation of a dense dispersion of nano-twins at some stage of the deformation, it is possible that dislocations cut through these twin interfaces during imposition of the strain [14]. In this process, it is then inevitable for a "stepped" morphology in Fig. 4 to be left behind in the nanotwins.

The emission of partial dislocations from grain boundaries plays a major role in the deformation of FCC metals with grain sizes of a few tens of nanometers [13, 15-19]. This consequently produces mechanical twins in nanocrystalline FCC metals. A critical grain sizes for mechanical twinning have been observed in nanocrystalline FCC metals above which no mechanical twins are formed by partial dislocation emission from the grain boundaries [15, 20]. For example, in pure Cu deformed by high-pressure torsion (HPT), deformation twins were observed only in grains smaller than 50 nm [15].

The critical ultra-fine grain size, d_c, at which the twinning mechanism transforms from the pole mechanism to the partial dislocation emission from GBs and GB junctions can be estimated from Eq. (2):

$$d_c = \frac{2\alpha\mu(nb - b_1)}{\gamma} \tag{2}$$

where n is a stress concentration factor [3]. At the initial stages of nucleation, n has a value between 2 and 4 [2] and γ is the SFE of the metal, and μ is the shear modulus. The parameter α reflects the character of the dislocation ($\alpha = 0.5$ and 1.5 for the edge and screw dislocations, respectively) and contains the scaling factor between the length of the dislocation source and the grain size. The Burgers vector of full dislocation and Shockley partial dislocation, respectively, are expressed by b ($\frac{1}{\sqrt{2}}a$) and b_1 ($\frac{1}{\sqrt{6}}a$), where a is the lattice parameter [3].

In the case of 70/30 brass, the parameters in Eq. (2) are given as follows: $\alpha = 1$, $n=2$ & $n=4$, $\gamma = 20$ mJm^{-2}, and $a = 3.68$ Å [3]. Using the parameters of 70/30 brass, d_c is estimated to be between 230 and 450 nm. This means that at grains smaller than 230 nm or 450 nm, nanotwins were not formed via the pole mechanism. In the six-cycle ARBed 70/30 brass with an average grain size of 50 nm, nanotwins were likely formed via partial dislocation emission from GBs and GB junctions. Previous experimental results on Cu [13, 15], Al [18-19] and Ni [21] also show that partial dislocation emissions occur only in grains smaller than 100 nm. Wang et al. [8] reported that the grain size for activating partial dislocation emissions from grain boundaries is a function of SFE and can be much larger than 100 nm for materials with low SFEs. Our observation implies that for brass with low SFE, there would be a grain size range in which two deformation mechanisms (partial dislocation emission from grain boundaries and full dislocation slip and multiplication) are able to operate simultaneously [8].

In this study, the grain size range in which the two deformation mechanisms are able to operate simultaneously was calculated to be 100-450 nm. The predominant mechanism in this range is, however, partial dislocation emission from grain boundaries.

Pre-existing nano-scale twin boundaries can effectively increase the strain rate sensitivity due to an interaction between twin boundaries and full dislocations, inhibiting the full dislocations from moving and disappearing at the grain boundaries. This significantly increases the dislocation storage capacity in the materials [22-23].

4. Conclusion

The results indicated that the average grain size of 70/30 brass after six ARB cycles was approximately 50nm with a large number of nanotwins. Also, it was found that at grain sizes of about 50nm, the twinning

mechanism transforms from the pole mechanism to the partial dislocations emission mechanism in which GB and GB junctions provide the necessary dislocation sources for the formation of nanotwin sources.

5. References

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تشکیل نانو دوقلویی در برنج نورد پیوندی – تجمعی شده

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چکیده: نورد پیوندی – تجمعی یکی از فرایندهای تغییر شکل پلاستیکی شدید است که برای تولید فلزات نانوساختار استفاده می‌شود. در این پژوهش، نورد تجمعی تا شش سیکل برای رسیدن به کرنش حقیقی مؤثر 4/8 در دمای محیط و بدون استفاده از روانکار روی برنج 70/30 اعمال گردید. ریزساختار نمونه‌های ساخته شده به این روش توسط میکروسکوپ‌های الکترونی روبشی (SEM) و عبوری (TEM) مورد بررسی قرار گرفت. نتایج بدست آمده نشان داد که پس از سیکل اول فرایند نورد تجمعی دانه‌ها از میکرو به نانو تغییر اندازه داد و دوقلوهای مکانیکی به طور گسترده در ساختار مشاهده گردید. پس از سیکل سوم فعالیت دوقلویی‌ها به طور قابل توجهی کاهش یافت و باندهای برشی در ساختار تشکیل شد. متوسط اندازه دانه پس از سیکل ششم فرایند، به حدود 50nm رسید و نانودوقلویی‌هایی که از مرزدانه‌ها و محل اتصال مرزدانه‌ها تشکیل شده بود، مشاهده گردید. با کاهش اندازه دانه به نانومتر، مکانیزم قطب، مکانیزم غالب تشکیل نانودوقلویی نبود و نانودوقلویی‌ها عمدتاً توسط تکثیر نابجایی‌های جزیی از مرزدانه‌ها و محل اتصال مرزدانه‌ها تولید گردید.

واژه‌های کلیدی: نورد پیوندی – تجمعی، نانودوقلویی، نانوساختار، برنج 70/30