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Hardening-softening transition in pre-annealed and slightly deformed $Ni_{70}Fe_{30}$ nanoalloy

H.T. Ni [a, *], J. Zhu [a], X.Y. Zhang [b]

[a] College of Materials and Chemical Engineering, Chongqing University of Arts and Sciences, Yongchuan, Chongqing 402160, China
[b] College of Materials Science and Engineering, Chongqing University, Chongqing 400044, China

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ABSTRACT

The Effect of slight deformation on microstructure evolution and microhardness variation in pre-annealed $Ni_{70}Fe_{30}$ nanoalloy has been investigated. An obvious hardening-to-softening transition is observed at the rolling strain of ~6%. Further x-ray diffraction analysis and transmission electron microscopy observation reveal a accumulation of dislocation, stacking fault and twin fault before the sample deformed to ~6%, which is considered the dominant contribution to strain hardening. Moreover, despite of rolling the sample at a relative small strain level, obvious grain growth takes place overall deformation process. With the increasing grain size, $Ni_{70}Fe_{30}$ nanoalloy enters into strain softening region due to a decrease in the quantity of defect densities when the rolling strain exceeds ~6%.

1. Introduction

The microstructure and mechanical behaviors of nanocrystalline (NC) metals and alloys have been extensively studied for their potential engineering applications. It has been well known that NC metals exhibit a series of different behaviors compared to coarse-grained and ultrafine-grained counterparts [1–5]. According to the basis of the classical dislocation theories, it is speculated NC metals exhibit a weak strain hardening behavior due to unsustainable dislocation networks within nanograins [6–8]. However, a recent study has suggested the presence of Lomer-Cottrell (L-C) locks introduces barriers for dislocation motion within nanograins, resulting in a strong strain hardening in NC Ni [9]. It is still of interest to investigate the influence of such peculiar dislocation activity on macroscopic mechanical response of NC metals. Particularly for NC alloys, the addition of alloying elements could further increase strength through solid solution strengthening and improve strain hardening ability of pure NC metals. Moreover, besides strain hardening, strain softening phenomenon has also been observed after large deformation of NC metals and alloys. Based on microstructure analysis, it is suggested strain softening behavior can be attributed to several factors such as defect annihilation, grain growth, grain rotation and even grain size distribution [10–15]. Some studies have reviewed the crossover from hardening to softening in NC metals and alloys, e.g. Ni, $Ni_{20}Fe_{80}$ [14–17]. It is shown that dislocation accumulation and annihilation is one of the most important reasons for the crossover behavior in deformed NC metals. In addition to this, de-twinning has also played a role in Ni-Fe alloys with a relatively low stacking fault energy [17].

The initiate state of the as-received NC metal should be also considered in the microstructure and property evolution. It has been found that deformation can lead to an obvious decrease in the densities of both dislocations and twins during cold rolling of electrodeposited NC $Ni_{82}Fe_{18}$ alloys with an initial highly excited state [18]. In our previous work, the relationship between microstructure and microhardness of electrodeposited NC $Ni_{70}Fe_{30}$ alloy have been studied preliminarily [19]. Specifically, both dislocation density and grain size increase with the increasing deformation strain, which appears to be inconsistent with other experiments. It is therefore important to understand the intrinsic factors that affect the microstructural evolution and corresponding mechanical response. It seems that low stacking-fault energy allows $Ni_{70}Fe_{30}$ alloy to harden easily. On the other hand, annealing is an effective way in tuning the microstructure. Once again it is necessary to ask the effect of initial microstructure on strain hardening ability of NC metals and alloys. In light of these results, low temperature

* Corresponding author.
E-mail address: htniok@163.com (H.T. Ni).

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annealing process has been conducted for the pretreatment of $Ni_{70}Fe_{30}$ nanoalloy. We have endeavored to explorer the crossover from hardening to softening in slightly rolled $Ni_{70}Fe_{30}$ nanoalloy based on the quantitative microstructure parameters from x-ray diffraction (XRD) analysis. Considering the XRD results is feasible and valid in the statistical sense, the microstructure of $Ni_{70}Fe_{30}$ nanoalloy is further investigated by transmission electron microscopy (TEM) observation and analysis. Note that some previous studies have reported the grain growth and defect evolution in NC metals based on in-situ TEM technique [20–22], much attention is focused on the defect structure in post-deformed sample, which will help to remedy deficiency in XRD analysis.

2. Material and methods

Bulk $Ni_{70}Fe_{30}$ nanoalloy sheet of about 250 μm thickness were prepared by electrodeposition technique. The chemical composition of the nanoalloy analyzed by x-ray fluorescence spectroscopy was ~70% Ni and ~30% Fe in weight. Prior to deformation, the as-received sheet was thermally annealed at 150 °C for 30min to reduce residual strain and intrinsic defects without grain growth. Two rectangular pre-annealed samples, with the same dimensions 6 mm (width) × 8 mm (length), were subjected to repeated rolling deformation using a twin-roller (with a diameter of 180 mm) apparatus at room temperature. To provide a measurable reduction in thickness, such slight cold rolling process was repeated up to 100 times. For simplicity, two samples under different preset rolling forces were referred to as sample A and sample B. During cold rolling processes, the rolling strains of each sample were calculated by $\varepsilon = \left|2/\sqrt{3}\ln(1+\delta)\right|$, where δ is the rolling reduction. After deformation, the samples for various microstructure and mechanical property tests were cut into 4 mm × 5 mm by edge trimming. Considering continuous rolling deformation and the size limitation of small sample, the mechanical responses of the samples were investigated by Micro-Vickers hardness, with a load of 9.8 N and a loading time of 10 s. The reported hardness values were averaged over eight measurements taken at different points. The microstructures of the pre-annealed and deformed samples were investigated by TEM and XRD. TEM observations were made on a Zeiss Libra 200FE instrument operated at 200 KV. XRD studies were performed using a Rigaku D/MAX 2500 PC diffractometer operating in a step scan mode with Cu-Ka radiation. Quantitative defect analysis was determined by x-ray line profile analysis [23–26].

3. Results and discussion

Fig. 1 shows the microhardness results of samples A and B. The hardness of the pre-annealed $Ni_{70}Fe_{30}$ nanoalloy is ~535 H V. After cold rolling deformation, the nanoalloy exhibits a combination of strain hardening followed by a strain softening. At very small rolling strains, the hardness increases rapidly to ~600 H V when the rolling strain reaches 6%. However, at higher strains, the hardness starts to decline during further rolling deformation and the hardness of 10% deformed sample drops to ~570 H V. This unstable hardness stage indicates that deformation structure has not yet achieved equilibrium during the small strain deformation. According to the fitting curve, the hardening-softening transition occurs at the rolling strain of ~6%, which will be referred to as critical strain.

To assess the correlation between the hardness and microstructure, the dislocation density is evaluated by XRD. Fig. 2 shows the dislocation density evolution during cold rolling of $Ni_{70}Fe_{30}$ nanoalloy. In the pre-annealed state, both sample A and sample B have a relatively low dislocation density ($4.5 \times 10^{14}/m^2$). Compared to the quantity of dislocation densities in the as-received state [19],

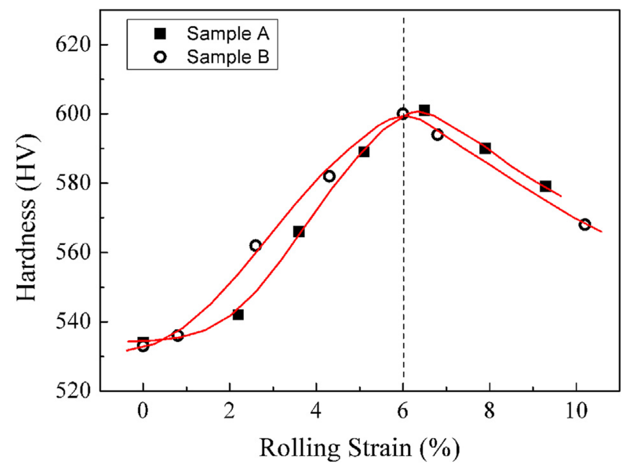

Fig. 1. Vickers microhardness of deformed $Ni_{70}Fe_{30}$ nanoalloy as a function of rolling strain.

it is found dislocation density may effectively be decreased by pre-annealing treatment. During cold rolling, there is a visible increase in dislocation density with increasing strain for both samples. When the rolling strain reaches critical strain, the dislocation density increases to ~$10^{15}/m^2$. Notably, the dislocation density of deformed sample B is somewhat higher than that of deformed sample A, which could probably be confirmed by the micro-hardness shown in Fig. 1. A more detailed analysis on the fitted micro-hardness data shows the hardness of sample B is indeed higher than that of sample A within critical strain, especially at the initial stage of rolling process, excluding the effect of measurement error. Nevertheless, at the latter stage of deformation for samples A and B, the dislocation density seems to saturate and even decrease. Despite of the downward trend, it is found the dislocation density of finally deformed sample A is still higher than that of its pre-annealed state.

Considering the SFs accompanied by partial dislocations, the density of stacking faults (SFs) including deformation SFs and twin-type SFs were analyzed and evaluated from XRD peak shift and broaden. Fig. 3a shows the evolution of deformation fault probability of samples A and B with increasing rolling strain. The undeformed sample has a very low deformation fault probability (~0.002), indicating a low density of deformation SFs. After rolling

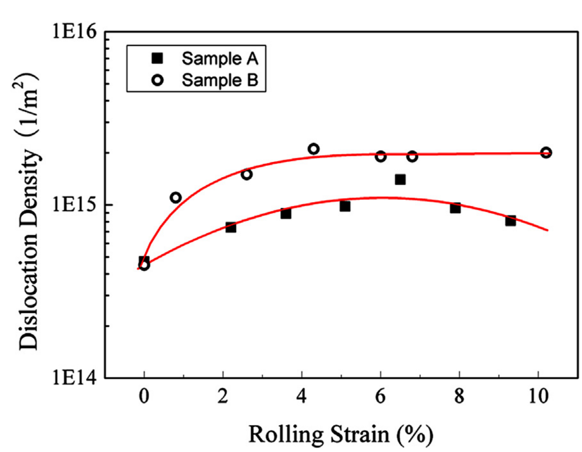

Fig. 2. Dislocation density of deformed $Ni_{70}Fe_{30}$ nanoalloy as a function of rolling strain. The corresponding fitted curves shows dislocation density of deformed sample B is higher than that of deformed sample A.