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Microstructure evolution and mechanical property of nanocrystalline Ni–20Fe alloy during cold rolling

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ABSTRACT

We report the microstructure evolution and mechanical property during room-temperature rolling of nanocrystalline Ni–20Fe alloys. The results show that massive dislocations and deformation twins in the deformed samples. Quantitative x-ray analysis reveals deformation induced grain rotation and grain growth. The dislocation density increases firstly and then tend to saturation after an equivalent strain (ε_{VM}) of ∼0.10 during the deformation. Correspondingly, the hardness increases when the ε_{VM} increases from 0 to ∼0.10, in spite of the increase in grain size. However, once the ε_{VM} exceeds 0.10, the hardness starts to decrease. It is suggested that the role of crystal defect as well as grain size should be considered to better understand the mechanical property of nanocrystalline Ni–20Fe alloy. At small strains ($\varepsilon_{VM} < 0.10$), the increase in hardness is a direct consequence of the high-density crystal defects; while at large strains ($\varepsilon_{VM} > 0.10$), since the crystal defects are saturated, the hardness decreases with further increase in grain size.

1. Introduction

The evolution of crystal defects in the metals is of obvious fundamental importance to understand the mechanical property. It is worth noting that, however, the presence of grain size effects on crystal defects in nanocrystalline (nc) metals. Most of the previous studies of nc metals have been devoted to mechanical behaviors based on different deformation mechanisms [1–8]. It has been found that stacking fault energy (SFE) is an important factor influencing deformation mechanisms and mechanical behaviors [9,10]. In the classical case, the relationship between material strength and grain size can be expressed as the Hall–Petch (HP) relation. Some investigations on nc metals show the cross-over between the normal and inverse HP effects seems to be related to SFE, suggesting the transition of deformation mechanisms [10,11]. For high SFE metals such as Al, the cross-over could be attributed to the transition from perfect dislocation slip to grain-boundary-mediate deformation, whereas in low SFE metals as Cu, the deformation mechanism goes from partial dislocation slip to grain-boundary-mediate deformation [10,11]. Furthermore, the stress-strain behavior of nc face-centered cubic metals during tensile deformation has shown that decreasing the SFE increases the strain hardening rate of the nc metals, especially at smaller grain size [12].

Following earlier studies, nc Ni with an grain size of ∼20 nm can still have strong strain hardening with increasing rolling strain but subsequently softening at higher strain [13]. Such unique mechanical behavior is treated to be caused by defect evolution as well as grain growth. If the crystal defects are more effective in contributing the strain hardening, the strain hardening and/or strain softening is expected to be more dependent on the SFE. Recognizing that a reduced SFE in the alloys would retard dislocation cross slip, recent studies have focused on the nc alloy systems like Cu–Al, Ni–Fe and so on [14,15]. In this study, we have carefully examined the nc Ni–20 wt% Fe (Ni–20Fe) alloys with an average grain size of ∼20 nm, as many studies allude to SFE effect on dislocation activity during plastic deformation.

2. Material and methods

The experiment for the current study was performed on a sheet of electrodeposited nc Ni–20Fe binary alloy acquired from the Integran Technologies Inc. The as-received sample was 25 mm square but only 200 μm thick. Samples were cut into several pieces in size of 10 × 10 mm^2, and then rolled at room temperature to various von Mises equivalent strains (ε_{VM}), calculated as $\varepsilon_{VM} = \left|2/\sqrt{3}\ln(1+\delta)\right|$, where δ is the rolling reduction. The microstructure of the samples was investigated using transmission electron microscopy (TEM) and X-ray diffraction (XRD). TEM specimens were thinned by double-jet electropolishing using a solution of nictric acid and mechanol (v:v = 1:4) at −30°C. TEM observation was employed on ZEISS LIBRA 200FE operated at 200 kV. XRD studies were performed on the as-received and the deformed samples on a Rigaku D/MAX 2500 PC diffractometer (18 kW) operating in a step scan mode with Cu Kα radiation. The mechanical properties of the specimens were measured in a Vickers hardness tester using a load of 100 g for a dwell time of 10 s during testing.

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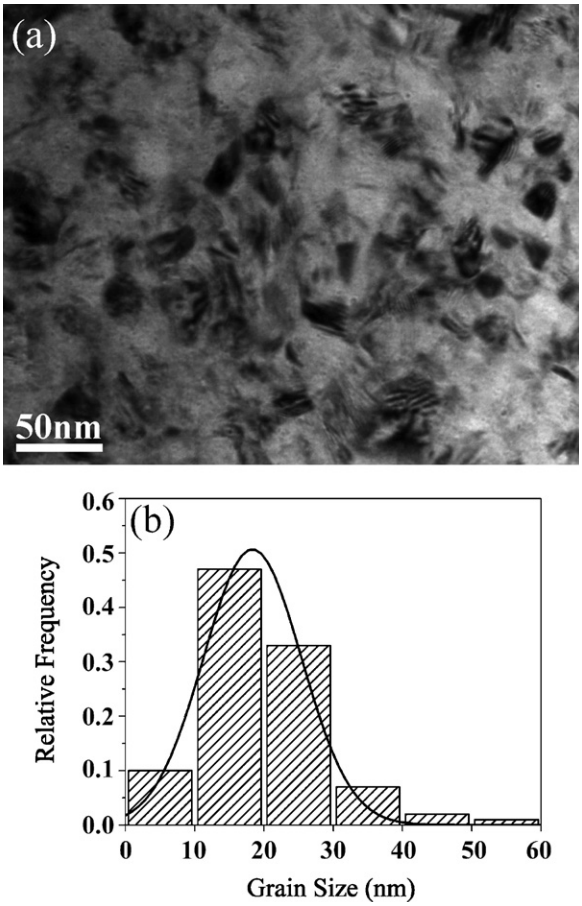

Fig. 1. (a) The bright-field TEM image of as-received nc Ni–20Fe alloy. The statistical grain-size distribution from corresponding dark-field TEM image is shown in (b).

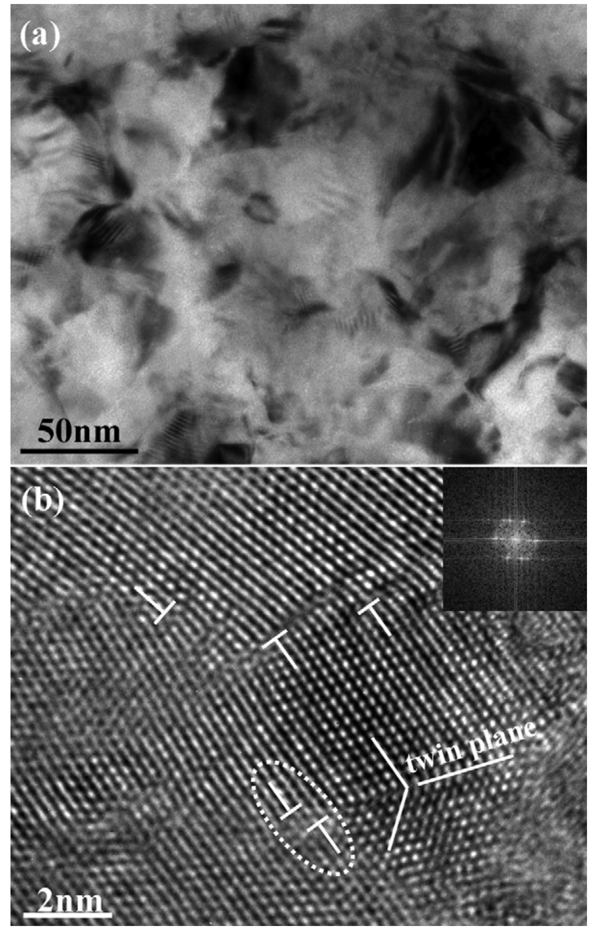

Fig. 2. (a) Typical bright-field TEM image of deformed nc Ni–20Fe alloy (ε_{VM} = 0.13). (b) High-resolution TEM image showing dislocations and twins in deformed sample. Dislocations marked by *T* and dislocation dipole marked by dashed circle. The fast Fourier transformation for twin relationship is shown in the upper right corner.

The quantitative parameters of the microstructure such as dislocation density and grain size were determined by X-ray line profile analysis [16,17]. The characteristic parameters of each individual peak profile, especially the peak position and the full width at half maximum, were used to evaluate the statistical microstructural information. A general least-squares technique for x-ray line broadening analysis was preformed to separate multiple-broadening effects due to small grain size and microstrain [17].

3. Results and discussion

Fig. 1 shows the TEM results on the microstructure of as-received nc Ni–20Fe alloy. The bright-field TEM image reveals that the grain shapes are equiaxed in the as-received state (as seen in Fig. 1a). The corresponding grain-size distribution based on TEM observations are given in Fig. 1b. It can be seen that the sample has a relatively narrow grain-size distribution. Most of grains have a size ranging from 10 nm to 30 nm.

Fig. 2a shows a typical bright-field TEM image for Ni–20Fe sample after cold-rolling to a stain of 0.13. No macro- or micro-cracks are found in the deformed sample. Particularly, there is an obvious grain growth after rolling deformation. The grains increase from around 20 nm in the as-received sample to around 30–50 nm. The high-resolution TEM is also taken to carefully examine the crystal defects within the deformed grains. Fig. 2b reveals high density of dislocations in a grain of ~35 nm. They are present in the form of individual dislocation or dislocation dipoles. Besides the massive dislocations, deformation twin is also observed.

Fig. 3 shows the XRD results of as-received and deformed samples. As shown in Fig. 3a, no Fe peaks are detected from XRD scans, indicating Ni and Fe atoms have formed single-phase solid solution alloy. Furthermore, it is clearly observed that the x-ray peaks are narrowed after deformation, implying that grain coarsening occurs during cold rolling. This grain coarsening phenomenon has also been observed in the tensile deformed Ni–Fe alloy [18]. Quantitative X-ray analysis shows there are many crystal defects in the deformed samples, which is consistent with TEM observations. Fig. 3b displays the changes in grain size and dislocation density for samples rolled to various strains. For the as-received state, the initial average grain size and dislocation density are about 20 nm and $1.09 \times 10^{16}\ m^{-2}$, respectively. However, upon cold rolling within strain of 0.10, there is a general increase in dislocation density. With continued deformation above ~0.10, the dislocation density starts to increase slowly and gradually change little. This behavior can be predicted from molecular dynamics simulation results of nc metals [19]. It has been suggested when partial dislocation starts to propagate a stacking fault defect will be left behind. After a grain is transected by a number of stacking faults, further dislocation propagation no longer occurs. Therefore, the dislocation density should reach a plateau after a significant increase. In the case of the grain size, a continuous increase is observed in the overall deformation. After the largest cold-rolling reduction, for the ε_{VM} = 0.168 sample, the grain size is determined to be ~50 nm, which is 2.5 times as large as the initial grain size. Accordingly, the dislocation density is measured to be around $2.90 \times 10^{16}\ m^{-2}$, which is three times as many as in as-received state.

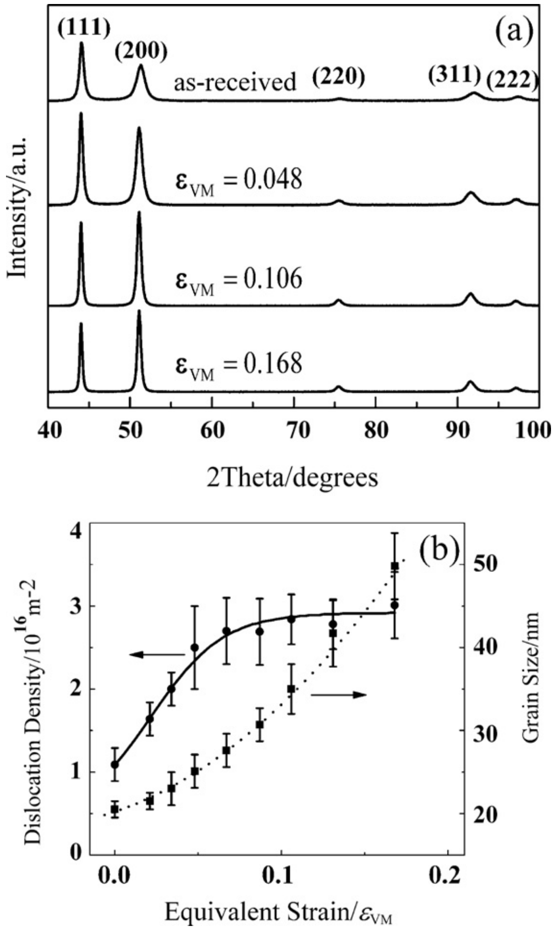

Fig. 3. (a) Typical XRD profiles of as-received and deformed samples. (b) The evolutions of dislocation density and grain size as a function of equivalent strain.

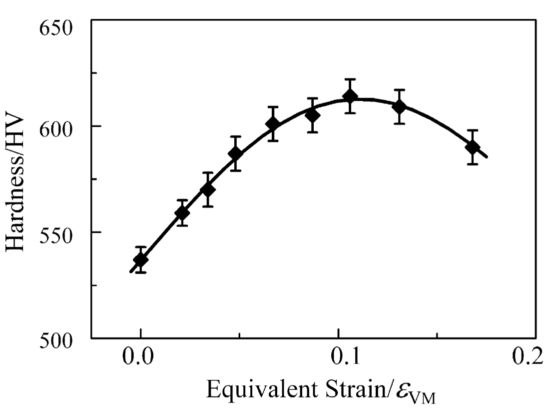

Fig. 4. Change of Vickers hardness (HV) for nc Ni–20Fe alloys as a function of equivalent strain.

Fig. 4 shows the hardness evolution of nc Ni–20Fe alloys during deformation. It can be seen that the hardness increases from 537 HV to 614 HV with increasing strain from 0 to 0.10. However, after the strain of 0.10, the hardness decreases with increasing equivalent strain. Compared with the pure Ni, the hardness of the current nc Ni–20Fe alloy is greatly improved with the Fe alloying effect, which is known as solid solution hardening [20]. In our previous investigation, the pure nc nickel enters into strain hardening slowly and reaches the peak hardness at an equivalent

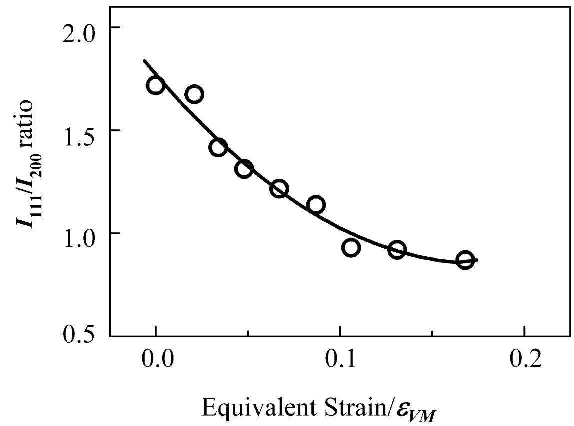

Fig. 5. The texture evolution of nc Ni–20Fe alloys as a function of equivalent strain.

strain of about 0.30 [21]. Noted that the nc Ni–20Fe alloy reaches a peak hardness just at the equivalent strain of 0.10, it can be concluded that the presence of Fe plays a significant role in the strain hardening. An alloy element Fe is known to decrease SFE [22]. The low SFE suppresses cross-slip of dislocations, leading to an increased work hardening rate [12]. According to the theoretical model proposed by Gutkin, it is energetically favorable for the Ni–20Fe alloy with low SFE successively emitting partial dislocations from triple junctions of grain boundaries (GBs) [23]. Due to dislocation accumulation during deformation, the contribution of high-density dislocations to the flow stress will obviously result in the increase of hardness. These results are consistent with the low SFE metals will strain harden more rapidly. Also, it is evident that stacking faults (or twins) is another factor influencing mechanical property of metals [24]. The stacking faults could become barriers to the dislocation motion so that they have a positive relationship to hardness. Limitation to dislocation slip systems imposed by the stacking faults could possess a certain degree of hardness enhancement.

Moreover, texture evolution could also have been responsible for the observed strain hardening during the early stage of deformation in nc Ni-Fe alloy. There is a simple evaluation for the texture evolution. Fig. 5 shows the change in the (1 1 1)/(2 0 0) diffraction intensity ratio (I_{111}/I_{200}) with increasing equivalent strain. When the samples are subjected to deformation within the strain of ~0.10, it can be seen that the (2 0 0) orientation strengthens, indicating that obvious grain rotation occurs during the deformation. This grain rotation could induce grain coarsening, as evidenced from Fig. 3b showing an increase in the average grain size. Noted that strain hardening still takes place in spite of grain coarsening. It has been suggested that plastic flow is dominated by grain rotation and dislocation activity [21,25]. The emission of dislocations from the GB occurs preferentially in the grains with a favorable orientation. However, there is a limitation on the number of dislocation accumulating at/near the GBs. In order to continue plastic deformation, new dislocation sources, e.g. GBs of other grains with an unfavorable orientation, have to be activated. Thus, this will lead to an increase in the stress required to deformation. Once ε_{VM} exceeds ~0.10, the (2 0 0) preferred orientation changes little, indicating that the grain rotation and dislocation activity become difficult. It can also be seen from Fig. 3b that dislocation density reaches a steady-state value in this stage of deformation. These observations imply that deformation-induced grain growth will play an important role in the mechanical property at large strain deformation.

Based on the above information, attention will be paid on the relationship between microstructure and mechanical properties. It is believed that the change in mechanical property of nc Ni–20Fe alloy is a combined effect of crystal defects and grain size. At early stages of deformation or at small strains ($\varepsilon_{VM} < 0.10$), the increase in hardness is a positive contribution of the high-density crystal defects, exhibiting strain hardening. For metals, it has been well known that the dislocation controls the strain hardening behavior. In general, when the initial dislocation density is smaller than the saturation value, an increase in the dislocation density as well as strain hardening will occur during further deformation, as observed in the present work and other literatures presented elsewhere [26,27]. However, the dislocation contribution is only a part of the total flow stress. While at late stages of deformation or at large strains ($\varepsilon_{VM} > 0.10$), with dislocations being saturated, the hardness starts to decrease due to the negative contribution of grain size, exhibiting strain softening. It is well known that the relationship between flow stress and grain size can adequately be expressed by a modified HP relation. And as a result, the hardness of Ni–20Fe drops due to the increase in grain size.

4. Conclusions

The microstructure evolution and mechanical property of electrodeposited nc Ni–20Fe alloy during cold rolling is investigated. Grain growth is observed after deformation. The grains remain equiaxed suggesting that grain rotation induced grain growth, which is confirmed by quantitative X-ray analysis. Massive dislocations and deformation twins are observed in the deformed samples. The dislocation density increases firstly and then tend to saturation after an equivalent strain of ∼0.10. Correspondingly, the hardness of nc Ni–20Fe alloy increases when the equivalent strain increases from 0 to ∼0.10, in spite of grain growth. However, once the ε_{VM} exceeds 0.10, the hardness starts to decrease. It is suggested that the mechanical behavior is a combined effect of crystal defects and grain size. At small strains, the hardness increases due to high density of crystal defects; while at large strains, after the crystal defects is saturated, the hardness decreases with further grain growth.

Acknowledgments

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