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Effect of Grain Orientation on Ductility in a Nanocrystalline Ni-Fe Alloy

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Abstract: The influence of columnar grain geometry on mechanical property was studied in an electrodeposited nanocrystalline Ni-Fe alloy. The compressive results show that the strength is independent of grain orientation. However, the plastic strain increased remarkably when the loading axis is parallel to the direction of grain columns, which is due to the enhanced grain boundary and dislocation activities. The significance of current study is that a new strategy was developed to improve the ductility of nanocrystalline materials.

Attributed to the smallness of grains, nanocrystalline materials possess superior strength, which offers this class of materials a promising application perspective. Therefore, studies on their mechanical properties have attracted extensive research interest over the past two decades. Early literature suggested that nanostructured metals might be intrinsically brittle because these materials exhibited little or no ductility.^{1,2} However, it was discovered later that the pre-mature failure is associated with the existence of processing defects,³ and the nano-grained metals actually behave in a ductile behavior,^{3,4} which was confirmed later by other works.^{5,6} For instance, nanostructured Ni-15%Fe alloy (~10nm) and pure Ni (~45nm) demonstrated a tensile plastic strain of 6.3% and 8.8%, respectively.^{3,4} Traditionally, a material's success in structural applications depends on the achievement of not only high strength but also good ductility. As a result, there have been considerable research efforts focusing on how to improve the ductility of nanomaterials further. Currently, various strategies have been reported. One is to introduce twins in grains. In a 400-500nm Cu, both strength and plastic strain increased significantly with an increase in the twin density.⁷ The function of twins is to provide the dislocation nucleation sources at twin boundaries. Another strategy is to take advantage of phase transformation. At the grain sizes of 100-350nm, the transformation-induced plasticity (TRIP) steels can exhibit a plastic strain over 20%.⁸⁻¹⁰ In addition, an improved ductility can also be realized by introducing a bi/multi-modal microstructure. The uniform elongation enhanced in this way could be as high as 30% in Cu (200nm-3 μ m) and Ni (100nm-10 μ m).^{11,12} Recently, another strategy developed in a 100nm 7075 Al alloy is

to create second-phase precipitates in grains, and, consequently, the uniform plastic strain increased from 3% to 7%.¹³ In addition to microstructural modifications, mechanical performance of nanocrystalline metals could also be dramatically improved at cryogenic temperatures.^{14,15} It is obvious that although these strategies can improve the ductility efficiently, they were usually accomplished in materials with grain sizes much larger than 100nm, i.e. out of nanocrystalline region (conventionally <100nm). In the present study, the dependence of ductility on grain orientation was addressed in a Ni-Fe alloy with an average grain size of approximately 25nm.

A 3.3mm thick electrodeposited Ni-20%Fe (wt.%) alloy plate was obtained from the Integran company. The rectangular specimens (side×side×length = $1.5 \times 1.5 \times 3 \text{ mm}^3$) were prepared: the sample length is either parallel or perpendicular to the growth direction. In addition, the $3.3 \times 3.3 \times 7 \text{ mm}^3$ samples were also made in that the sample length is perpendicular to the growth direction. Before testing, the specimen ends were polished to ensure that they were flat and normal to the loading axis. The compression tests were conducted at a strain rate of $5 \times 10^{-3} \text{ s}^{-1}$ at room temperature. At least two samples were tested per condition. For the purpose of dependable comparison, all the specimens were machined from the same plate. The microstructure was characterized using transmission electron microscopy (TEM) and x-ray diffractometer.

Figure 1 shows the x-ray diffraction patterns in the plan and cross-sectional views.

The peak indices were labeled for the cross-sectional view pattern, and the plan view pattern has the same peak indices at the corresponding 2θ positions. The normalized peak intensities are summarized in Table 1. Clearly, this alloy is single phase with face-centered cubic (fcc) structure. The inset schematically defines that the directions parallel and perpendicular to the growth direction (thickness) are called the plan and cross-sectional views, respectively. In the cross-sectional view, the grains exhibited almost random orientation. Nevertheless, the plan view pattern shows that there was a moderate (100) texture. The detailed microstructure was characterized by TEM, as shown in Figure 2. The plan view image, Fig. 2(a), demonstrates that this alloy has a relatively narrow grain size distribution and grains exhibited an equiaxed shape. The average grain size is about 22nm. However, in the case of cross-sectional view, Figure 2(b) displays that grains have a high aspect ratio, which indicates that the columnar grains developed along the growth direction during electrodeposition. Some grains' aspect ratios are over three. The grain size along the growth direction is approximately 60nm, while it is about 27nm perpendicular to the growth direction. Such columnar microstructure is associated with the (100) out-of-plane texture observed in Fig. 1, and similar columnar grain structure was also found in the electrodeposited Ni.¹⁶

Figure 3 presents the stress-strain curves of two types of specimens, and the results are summarized in Table 2. When the loading axis is perpendicular to the growth direction, like the literature results, nanocrystalline Ni-Fe alloy exhibited a high yield

strength (1720MPa) and appropriate ductility (9.1%). In addition, when the sample geometry was increased from $1.5 \times 1.5 \times 3 \text{ mm}^3$ to $3.3 \times 3.3 \times 7 \text{ mm}^3$, as shown in Table 2, both strength and plastic strain did not have noticeable changes, suggesting that the specimen size does not affect mechanical property as it is much larger than the grain size. Among the prevailing methods of fabricating bulk nanostructures are powder metallurgy, high-pressure torsion, sputter deposition, and electrodeposition. The first two generally do not produce columnar grains. Although the last two can make columnar structures, the samples made by these techniques usually have a thickness less than 0.2mm, which is not sufficient to perform macroscopic mechanical testing along the growth direction. As a result, the dependence of mechanical property on grain orientation has not been well studied so far. A thickness of over 3mm of the Ni-Fe plate allows us to do such investigation. Interestingly, Figure 3 and Table 2 show that when the load was applied along the growth direction (longitudinal direction of grain columns), plastic strain increased dramatically from 9% to 20% without a sacrifice of strength. That is, mechanical performance of nanocrystalline metals could be significantly improved by the use of special grain geometry.

Although several strategies have been established to improve ductility, they do not apply to the current case because those materials usually have sub-micrometer grain sizes.⁷⁻¹³ In principle, the ductility of one material is closely related to its deformation mechanism. For nanocrystalline metals, the deformation mechanism undergoes a transition from dislocation-mediated plasticity to grain boundary sliding when the

grain size is below a critical value.¹⁷⁻¹⁹ For the fcc metals, the critical value is about 15-20nm. Note that such transition is not abrupt, and both dislocation and grain boundary activities are prominent simultaneously when the grain size is near the critical value. But their contribution to the ductility may depend on grain size and applied strain.^{20,21} At a grain size of about 25nm, the ductility is believed to originate from a combination of grain boundary sliding and dislocation activities.²¹ The grain boundary sliding is a shear process parallel to grain boundary planes and is associated with the applied stress's component resolved parallel to grain boundary planes. In general, slipping along the longitudinal direction is apt to occur when grain boundary plane is close to the loading axis because the smaller angle leads to the larger resolved stress component parallel to grain boundary plane at a given applied stress. Compared to the situation that the loading axis is perpendicular to the growth direction, when the loading axis is parallel to the growth direction, due to the unique columnar grain structure, the grain boundary area with high stress component parallel to the grain boundary plane is larger. That is, the grain boundary activities may be enhanced when the applied stress is along the columnar direction. As for the dislocation activities in nanocrystalline metals, owing to the fine grain sizes, the grain interiors are usually devoid of dislocation sources, and the plasticity is controlled by the dislocation nucleation at grain boundaries. Assume the dislocation source per unit of grain boundary area is a constant and randomly distributed at grain boundaries, when the compression loading axis is parallel to the growth direction, a large part of grain boundaries on columnar surfaces, i.e. more dislocation sources, bear a tensile stress.

Also, in this regard, the (100) grain orientation provides more equally operational slip systems. This scenario may intensify the dislocation and grain boundary activities. Thus, when the loading axis is parallel to the growth direction, both the grain boundary and dislocation activities might be enhanced, accordingly, resulting in an improvement in the ductility.

In summary, a thick electrodeposited nanocrystalline Ni-Fe alloy plate exhibited a moderate (100) out-of-plane texture and a nearly random in-plane grain orientation. This unique crystallographic orientation is attributed to the development of columnar grains along the growth direction. The compression test results demonstrate that the ductility is dependent on the grain orientation. When the loading axis is along the columnar direction, a pronounced improvement in ductility was achieved without a loss in the strength, which is related to the enhanced grain boundary and dislocation activities. This approach could be extended to other nanocrystalline materials.

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References

- ¹ J. R. Weertman, D. Farkas, K. Hemker, H. Kung, M. Mayo, R. Mitra, and H. Van Swygenhoven, *Mater. Res. Soc. Bull.* **24**, 44 (1999).
- ² X. Y. Qin, X. G. Zhu, S. Gao, L. F. Chi, and J. S. Lee, *J. Phys.: Condens. Matter.* **14**, 2605 (2002).
- ³ H. Q. Li and F. Ebrahimi, *Acta Mater.* **54**, 2877 (2006).
- ⁴ H. Q. Li and F. Ebrahimi, *Appl. Phys. Lett.* **84**, 4307 (2004).
- ⁵ K. M. Youssef, R. O. Scattergood, K. L. Murty, and C. C. Koch, *Appl. Phys. Letter.* **85**, 929 (2004).
- ⁶ C. D. Gu, J. S. Lian, Z. H. Jiang, and Q. Jiang, *Scripta Mater.* **54**, 579 (2006).
- ⁷ L. Lu, R. Schwaiger, Z. W. Shan, M. Dao, K. Lu, and S. Suresh, *Acta Mater.* **53**, 2169 (2005).
- ⁸ K. X. Tao, H. Choo, H. Q. Li, B. Clausen, J. E. Jin, and Y. K. Lee, *Appl. Phys. Lett.* **90**, 101911 (2007).
- ⁹ Y. Q. Ma, J. E. Jin, and Y. K. Lee, *Scripta Mater.* **52**, 1311 (2005).
- ¹⁰ S. Cheng, H. Choo, Y. H. Zhao, X. L. Wang, Y. T. Zhu, Y. D. Wang, J. Almer, P. K. Liaw, J. E. Jin, Y. K. Lee, *J. Mater. Res.* **23**, 1578 (2008).
- ¹¹ Y. Wang, M. Chen, F. Zhou, and E. Ma, *Nature* **419**, 912 (2002).
- ¹² Y. Zhao, T. Topping, J. F. Bingert, J. J. Thornton, A. M. Dangelewicz, Y. Li, W. Liu, Y. T. Zhu, Y. Zhou, and E. J. Lavertie, *Adv. Mater.* (in press).
- ¹³ Y. H. Zhao, X. Z. Liao, S. Cheng, E. Ma, and Y. T. Zhu, *Adv. Mater.* **18**, 2280 (2006).

- ¹⁴ H. Q. Li, H. Choo, and P. K. Liaw, *J. Appl. Phys.* **101**, 063536 (2007).
- ¹⁵ Y. M. Wang, E. Ma, R. Z. Valiev, and Y. T. Zhu, *Adv. Mater.* **16**, 328 (2004).
- ¹⁶ K. S. Kumar, S. Suresh, M. F. Chisholm, J. A. Horton, and P. Wang, *Acta Mater.* **51**, 387 (2003).
- ¹⁷ J. Schiøtz and K. W. Jacobsen, *Science* **301**, 1357 (2003).
- ¹⁸ V. Yamakov, D. Wolf, S. R. Phillpot, A. K. Mukherjee, and H. Gleiter, *Nature Mater.* **3**, 43 (2004).
- ¹⁹ H. Van Swygenhoven, P. M. Derlet, and A. Hasnaoui, *Phys. Rev. B* **66**, 024101 (2002).
- ²⁰ H. Q. Li, H. Choo, Y. Ren, T. A. Saleh, U. Lienert, P. K. Liaw, and F. Ebrahimi, *Phys. Rev. Lett.* **101**, 015502 (2008).
- ²¹ N. Q. Vo, R. S. Averback, P. Bellon, S. Odunuga, and A. Caro, *Phys. Rev. B* **77**, 134108 (2008).

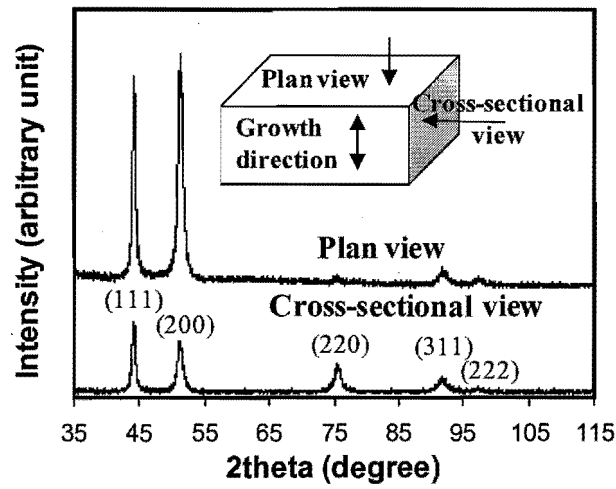


FIG. 1. X-ray diffraction patterns of nanocrystalline Ni-Fe alloy in the plan and cross-sectional views.

Table 1. The texture results of the as-deposited Ni-Fe alloy.

| FCC peaks | 111 | 200 | 220 | 311 | 222 |
|--------------------------------|-----|-----|-----|-----|-----|
| Std. int. (Ni ₃ Fe) | 100 | 60 | 30 | 40 | 10 |
| Cross-sectional | 100 | 75 | 44 | 27 | 14 |
| Plan view | 90 | 100 | 8 | 11 | 7 |

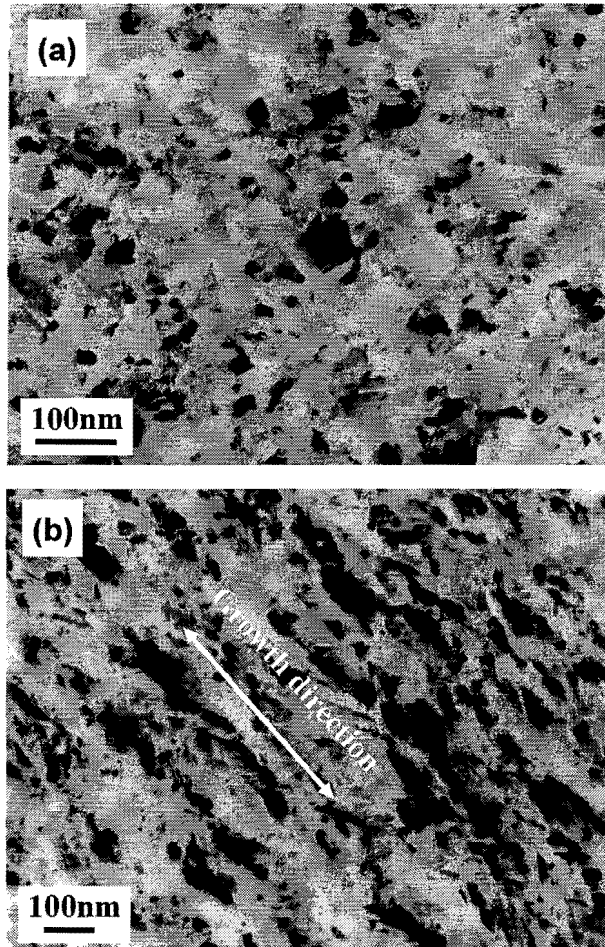


FIG. 2. Dark-field TEM images showing the grain geometries. (a) plan view and (b) cross-sectional view.

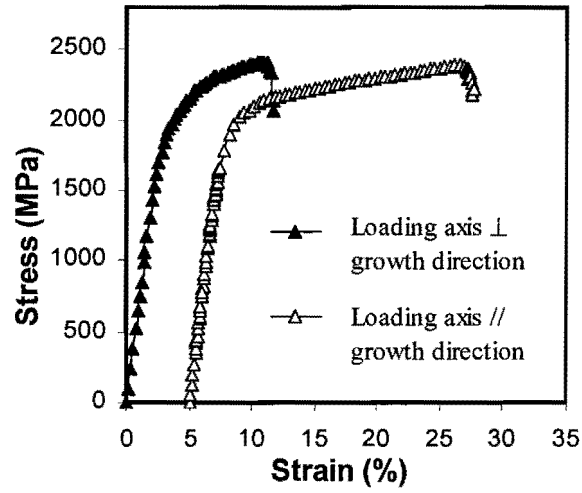


FIG. 3. Compressive stress-strain curves of nanocrystalline Ni-Fe alloy.

Table 2. A summary of compression results. L = sample length;

LA = loading axis; GD = growth direction.

| Samples | Yield stress (MPa) | Ultimate stress (MPa) | Plastic strain (%) |
|------------------------|-----------------------|--------------------------|-----------------------|
| L = 3mm, LA \perp GD | 1720 ± 6 | 2448 ± 38 | 9.1 ± 0.6 |
| L = 7mm, LA \perp GD | 1705 | 2315 | 9.0 |
| L = 3mm, LA // GD | 1705 ± 5 | 2410 ± 10 | 20.6 ± 1.4 |