

Effect of grain size on the competition between twinning and detwinning in nanocrystalline metalsS. Ni,¹ Y. B. Wang,¹ X. Z. Liao,^{1,*} H. Q. Li,² R. B. Figueiredo,³ S. P. Ringer,¹ T. G. Langdon,^{4,5} and Y. T. Zhu^{6,†}¹The University of Sydney, Sydney, New South Wales 2006, Australia²Los Alamos National Laboratory, Los Alamos, New Mexico 87545, USA³Federal University of Minas Gerais, Belo Horizonte, Minas Gerais 31270-901, Brazil⁴University of Southampton, Southampton SO17 1BJ, United Kingdom⁵University of Southern California, Los Angeles, California 90089-1453, USA⁶North Carolina State University, Raleigh, North Carolina 27695-7907, USA

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Both twinning and detwinning have been reported to occur during the deformation of nanocrystalline (nc) face-centered-cubic metals. This raises the issue of how these two processes compete with each other. Here, we report that the twinning process dominates in a certain range of grain sizes, whereas, the detwinning process dominates outside of this range to annihilate all twins. These experimental observations establish a full spectrum of grain-size effects on deformation twinning and detwinning and are explained by the deformation physics. They also provide a fundamental basis for understanding and designing the mechanical behavior of nc metals and alloys.

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I. INTRODUCTION

To twin or to detwin? This is an important question for many nanocrystalline (nc) face-centered-cubic (fcc) metals and alloys. Deformation twinning has been reported extensively,^{1–7} and detwinning has been observed both experimentally^{8–10} and in molecular-dynamics simulations.¹¹ However, there is no clear information on the conditions required for these individual processes to be activated and to predominate. Specifically, nc fcc metals have been reported to deform by twinning when the grains are smaller than a certain size, and an *inverse grain-size effect*, where twinning becomes more difficult with decreasing grain size, was observed when the grain sizes were very small.^{1,12–14} However, detwinning of growth twins was observed recently during the deformation of nc fcc metals.^{8,9,15} These observations raise a critical issue: What is the effect of grain size on the competition of deformation twinning and detwinning?

This issue has significant implications for both the science and the applications of nc materials. Growth twins and deformation twins have been reported to provide a unique mechanism to simultaneously increase the strength and ductility of nc materials.^{16–19} Understanding the grain-size effect on deformation twinning and detwinning would enable us to predict the stability and evolution of microstructures and mechanical properties and to intelligently design nc fcc materials for practical applications.

II. EXPERIMENTAL PROCEDURE

In this paper, an electrodeposited nc Ni-20Fe (wt %) alloy with preexisting growth twins and an average grain size of 20 nm was used to study the effect of grain size on the deformation twinning and detwinning. It has been reported that plastic deformation induces grain growth in nc materials.^{20–27} This provides us with a unique opportunity to systematically vary the grain size by controlling the amount of deformation imposed on the nc alloy. Disks with diameters slightly less than 10 mm and thicknesses of ~ 0.8 mm were subjected to high-

pressure torsion (HPT) to different turns under a pressure of 6 GPa at room temperature, using a quasiconstrained HPT facility. The average grain size increased to 115 nm after 30 HPT turns, enabling us to study statistical changes in twin density during deformation over a wide nano-grain-size range from 10 to more than 100 nm. Comprehensive transmission electron microscopy (TEM) observations were carried out at the edge part of each HPT disk. The size distributions of all grains and twin-containing grains at each deformation stage were estimated statistically by measuring at least 400 grains with clear grain boundaries, e.g., grains that were usually on strong diffraction conditions and, therefore, appeared dark, from multiple bright-field TEM images. Low-angle grain boundaries can clearly be identified from a bright-field image when the grains are on strong diffraction conditions (close to a zone axis), although they may not be seen in normal TEM.²⁷ Here, a twinned grain is defined as a grain containing at least one twin.

III. RESULTS AND DISCUSSION

Figure 1 shows the evolution of the size distribution for both of all grains and the subset of grains that contain twins with increasing numbers of HPT turns. The initial as-deposited sample has a narrow grain-size distribution in the range of ~ 10 – 35 nm, and about 30% of these grains contain growth twins that were formed during the sample synthesis [Fig. 1(a)]. After deformation via five HPT turns, the average grain size increased to about 40 nm, and the grain size distribution was broader [Fig. 1(b)]. Significantly, only 7% of these grains contain twins, which is a dramatic drop from the initial state. Figure 1(b) also demonstrates that grains of diameters larger than ~ 35 nm, which were formed by deformation-induced grain growth, did not contain twins. These observations indicate that extensive detwinning occurred during the deformation in grains smaller than 35 nm and that new grains formed during grain growth did not contain twins.

Deforming the sample to ten HPT turns increased the average grain size to ~ 50 nm and caused the disappearance of twins in grains of diameters < 35 nm [Fig. 1(c)], suggesting

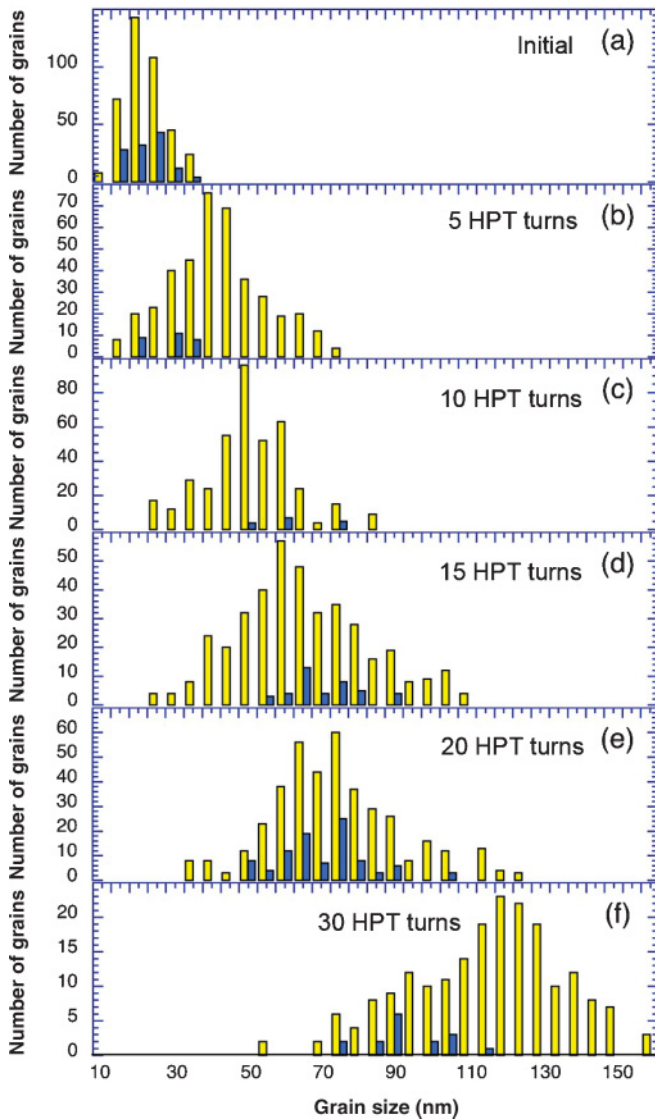


FIG. 1. (Color online) The size distributions of all grains (light yellow bars) and grains that contain twins (dark blue bars) with increasing HPT turns in a nc Ni-20 wt % Fe alloy. The statistical data are measured using high-resolution TEM (HRTEM) with the sample in a location close to the edge of each HPT disk. At least 400 grains with clear grain boundaries were counted for each deformation stage.

that all growth twins have been removed by detwinning. Interestingly, twins appeared in 6% of grains that were larger than 45 nm (or 4% of all grains). It can reasonably be concluded that these twins were formed by deformation because these large grains initially did not contain twins [Fig. 1(b)]. Thus, deformation twinning is activated in grains larger than 45 nm.

Increasing the deformation strain to 15 and 20 HPT turns increased the fraction of twinned grains to 10% and 24%, respectively, indicating a significant increase in twin density. Careful examination of Figs. 1(c)–1(e) reveals that, although the overall grain-size distribution shifts to larger size values, the grain size where the highest twinning fraction appears remains almost unchanged. Furthermore, Fig. 1(e) shows that, at 20 HPT turns, the size distribution of the twinned grains matches well with that of all grains, and their mean grain sizes

were almost the same, ~ 72 nm for all grains and ~ 68 nm for twinned grains. Note that this was also the grain-size distribution that yielded the highest fraction of twinned grains. These observations indicate that there is an optimum grain size for twinning, which is not significantly affected by the overall grain-size distribution. This is consistent with the inverse grain-size effect on the formation of deformation twins reported earlier.^{1,12} It should be noted that this optimum grain-size range for twinning is affected by intrinsic materials properties, such as stacking fault energy as well as by externally applied deformation conditions, such as stress and strain rate.¹

Further deforming the sample to 30 HPT turns increased the average grain size to 115 nm [Fig. 1(f)]. However, the deformation twins exist only in smaller grains, and the fraction of the twinned grains is reduced to 8%. When a grain that contains a deformation twin grows larger, the twin can either be annihilated by the detwinning process, which leads to a reduction in twin density or can remain in the grain, which leads to a larger grain that contains the twin. Examining and comparing Figs. 1(e) and 1(f) reveals that significant detwinning occurred during the additional deformation from 20 to 30 HPT turns. Concurrently, some deformation twins remained in the growing grain, which resulted in a distribution of twinned grains to larger sizes. However, all twins are predicted to disappear with larger deformation to further increase the grain size.

The experimental finding shown in Fig. 1 can be summarized as follows. There exists an optimum grain-size range for the formation of deformation twins. Outside of this grain-size range, the detwinning process dominates to annihilate existing twins. This observation raises two scientific issues. First, what is the reason for the observed grain-size effect on twinning and detwinning? Second, what is the mechanism for the detwinning process?

The first issue can be understood with the following analysis. Experimental observations and analytical modeling have revealed that, in a nc fcc metal, there exists a grain-size range within which deformation twins would form.^{1,12–14} From Fig. 1, it appears that the grain-size range for the formation of deformation twins in the current Ni-Fe alloy under HPT deformation conditions is ~ 45 – 100 nm. However, as discussed later, the detwinning process can be caused by the interaction between the dislocation and the twin boundary, which occurs in grains of all sizes. In the initial sample, all grains are smaller than 45 nm. Therefore, the detwinning process was active while the twinning process was inactive in the sample under HPT deformation, which eventually led to the annihilation of all initial growth twins. In the grain-size range of 45– 100 nm, the twinning process prevailed over the detwinning process, which led to the formation of deformation twins in a large fraction of grains. In grains larger than 100 nm, the twinning process was no longer active while the detwinning process remained operational, leading to the observed reduction in deformation twins.

Comparing the reduction in twinned grains after five HPT turns with that from 20 to 30 HPT turns in Fig. 1, it appears that the detwinning tendency is stronger at small grain sizes (below 35 nm) than at large grain sizes (> 100 nm). This is reasonable because the detwinning process involves

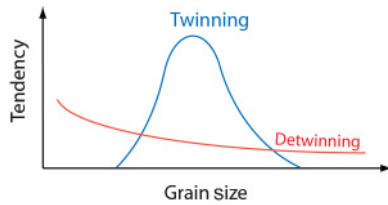


FIG. 2. (Color online) Schematic of the grain-size effect on the twinning and detwinning tendencies.

dislocation interactions with twin boundaries, which need to overcome relatively high-energy barriers as compared with other deformation mechanisms.²⁸ Materials with smaller grains deform plastically under a higher applied stress, which makes it easier to overcome the energy barrier for detwinning. Therefore, detwinning statistically should be easier in smaller grains. From the above discussions, the tendency for twinning and detwinning during plastic deformation can be illustrated schematically as in Fig. 2. It should be noted that the observed grain-size effect on twinning and detwinning is not caused by texture evolution during the HPT. Our x-ray analysis indicates that the intensity ratio of the $\{111\}$ reflection to the $\{200\}$ reflection does not change significantly from 5 to 30 HPT turns, suggesting that the texture did not change significantly after 5 HPT turns. The severe plastic deformation resulted in a change in grain size and other structural features including dislocation density and dislocation configuration. However, these other features appeared to have no significant effect on the twin density evolution.

The second issue is that the detwinning process in grains of all sizes is associated with detwinning mechanisms. Figure 3 shows three HRTEM images that reveal three scenarios for detwinning. Figure 3(a) shows two one-atomic-layer single steps on the twin boundary, while Fig. 3(b) shows a pair of opposing one-layer steps. These two types of steps on the twin boundary are formed by the interactions of a 30° Shockley partial with the twin boundary.^{28,29} The 30° Shockley partial could be emitted from the grain boundary or could be produced by the dissociation of a lattice dislocation. Repetition of these dislocation reactions at a twin boundary will result in the disappearance of the twin.

Figure 3(c) shows a three-layer step and a six-layer step on a twin boundary. This is formed by the stop-start three partial detwinning mechanisms reported earlier.^{8,9} Specifically, three Shockley partials with the sum of their Burgers vectors equal to 0 glide in a coordinated way to remove three twin layers. One partial glides forward first under the applied stress and stops due to a stress drop, leaving behind a stacking fault. Then, the other two partials are driven forward by the stacking fault and their interaction force with the first partial.

The grain-size effect on twinning and detwinning observed here has a profound impact on applications for nc materials. To enhance their strength and ductility, one may want to adjust the stacking fault energy by means of alloying to produce a nanostructure with a high density of deformation twins or growth twins. In addition, to maintain a relatively stable microstructure, grain sizes need to be in the range that is optimal for the formation of the deformation twins. This is critical under some service conditions, such as fatigue

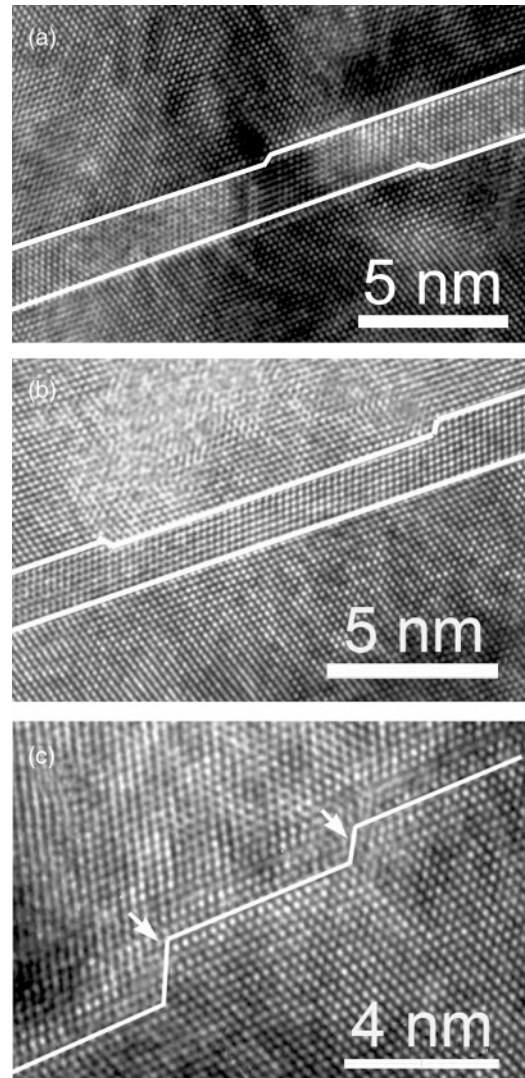


FIG. 3. HRTEM images revealing steps on the twin boundary that were produced by the detwinning process: (a) one-layer single steps, (b) a pair of opposing one-layer steps, and (c) a three-layer step and a six-layer step.

where the cyclic stress could induce extensive dislocation interactions with Twins. Otherwise, the detwinning process will significantly soften the material, which may lead to accelerated failure.

IV. SUMMARY

To summarize, our statistical analysis of TEM data reveals a competition between deformation twinning and detwinning in nc fcc Ni-Fe alloys across a wide range of grain sizes from 10 to >100 nm. The deformation twinning process prevails over the detwinning process over the grain-size range of 45–100 nm during HPT deformation, with the optimum grain size of ~ 70 nm for twinning. Outside of this grain-size range, the detwinning process prevails over the twinning process, which leads to the annihilation of existing growth twins and deformation twins. The detwinning process is caused primarily by the dislocation interactions with twins and is more readily activated in smaller grains due to their higher flow stresses.

These observations provide some guidance for designing nc materials with twin structures that can enhance both strength and ductility.

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