

## Predictions for partial-dislocation-mediated processes in nanocrystalline Ni by generalized planar fault energy curves: An experimental evaluation

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Generalized planar fault energy (GPFE) curves have been used to predict partial-dislocation-mediated processes in nanocrystalline materials, but their validity has not been evaluated experimentally. We report experimental observations of a large quantity of both stacking faults and twins in nc Ni deformed at relatively low stresses in a tensile test. The experimental findings indicate that the GPFE curves can reasonably explain the formation of stacking faults, but they alone were not able to adequately predict the propensity of deformation twinning. © 2006 American Institute of Physics. [DOI: 10.1063/1.2186968]

Nanocrystalline (nc) materials possess unusual mechanical properties, which are attributed to their unique deformation mechanisms.<sup>1–8</sup> Deformation mechanisms identified in nc materials include partial dislocation emission from grain boundaries,<sup>9–17</sup> deformation twinning,<sup>9–16</sup> full dislocations,<sup>10,12,14</sup> and grain boundary sliding.<sup>10,18–20</sup> Recent molecular dynamics (MD) simulations suggest that generalized planar fault energy (GPFE) curves significantly affect the partial-dislocation-mediated deformation processes in nc materials.<sup>12</sup> Specifically, the GPFE curves were predicted to control the nucleation of partials and twins from grain boundaries.<sup>12</sup> This concept has been accepted by many in the community and has inspired more theoretical studies. For example, a recent analytical model,<sup>21</sup> using an approach similar to the analysis of dislocation emission from a crack tip,<sup>22,23</sup> concluded that the GPFE curves indeed affect the nucleation of stacking faults and deformation twins in nc materials. However, these predictions have yet to be verified experimentally.

Ni is a perfect candidate for studying the effects of GPFE curves on the partial dislocation mediated deformation processes in nc materials. As shown in Fig. 1,<sup>12</sup> Ni has a very high stable stacking fault energy (SFE),  $\gamma_{sf}$ , which makes it energetically unfavorable to form a stacking fault by emitting only a leading partial from a grain boundary. On the other hand, Ni also has a high unstable SFE,  $\gamma_{usf}[(\gamma_{usf} - \gamma_{sf})/\gamma_{sf} \approx 0.4]$ , and an even higher unstable twin fault energy,  $\gamma_{utf}[(\gamma_{utf} - \gamma_{sf})/\gamma_{sf} \approx 0.9]$ , which hinder the nucleation of trailing partials and twinning partials. These GPFE curve features predict several deformation scenarios in nc Ni depending on the applied stress, including the following:<sup>12</sup> (I) If the stress is too low to overcome  $\gamma_{usf}$ , no partials can be nucleated, and no stacking faults or twins are formed. (II) If the stresses can overcome  $\gamma_{usf}$  but are not much higher, we

should observe stacking faults despite of the high  $\gamma_{sf}$ , but no twins or trailing partials. (III) If the local stress is sufficiently high to overcome  $\gamma_{utf}$ , both stacking faults and twins should be observed. The MD simulations observed only stacking faults, or scenario II. No trailing partials or twin nucleation were found to operate. It was pointed out from a comparison with the behavior of nc Al and Cu that it is the GPFE features, not the stable SFE alone, that lead to the observation of stacking faults in nc Ni.<sup>12</sup> However, Ref. 12 also cautions that the stacking faults could be an artifact of the short time scale inherently associated with the MD simulations. It is therefore important to test, on laboratory time scales, if any subsequent processes involving trailing or twinning partials occur and whether they follow the GPFE predictions.

It is the objective of this study to experimentally evaluate the effects and predictions of the GPFE curves for nc Ni. An electrodeposited nc Ni film with an average grain size of 25 nm and a thickness of 100  $\mu\text{m}$  was purchased from

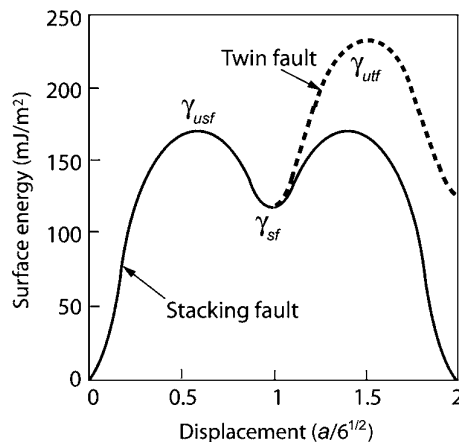


FIG. 1. Schematic of the generalized planar fault energy curves for Ni (Ref. 12).  $\gamma_{sf}$  is the stable stacking fault energy;  $\gamma_{usf}$  is the unstable stacking fault energy;  $\gamma_{utf}$  is the unstable twin fault energy;  $a$  is the lattice constant.

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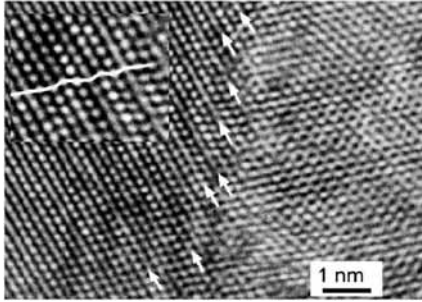


FIG. 2. An HRTEM image for nc Ni deformed at LNT showing an equilibrium grain boundary with a misorientation of  $16^\circ$ . A high density of stacking faults (marked by arrows) indicates that many partials were emitted from the grain boundary. The inset shows the details of stacking faults.

Goodfellow Inc. Growth twins were observed in the as-deposited nc Ni but were rare. X-ray analysis measured a residual strain of 0.33%, which corresponds to a residual stress of 310 MPa. The electrodeposited nc Ni has grain boundaries not very far from equilibrium<sup>21</sup> and those used in the MD simulations. The nc Ni foils were deformed under tension at a strain rate of  $3 \times 10^{-3} \text{ s}^{-1}$  at room temperature (RT) and liquid nitrogen temperature (LNT, 77 K). Samples tested at both temperatures have an elongation to failure of  $\sim 4\%$ , and the maximum tensile flow stress was  $\sim 1200$  MPa at RT and 1500 MPa at LNT. The maximum shear flow stress is half of the tensile stress on slip planes inclined  $45^\circ$  to the tensile direction, i.e., 600 MPa at RT and 750 MPa at LNT.

High resolution electron microscopy (HREM) revealed a few occasional deformation twins and full dislocations, but no stacking fault, in the nc Ni deformed at RT, indicating that partial-dislocation-mediated processes did not play a significant role in the deformation. Therefore, at RT the applied stress is too low to overcome  $\gamma_{\text{usf}}$  (scenario I). This is also supported by the observation that at RT no deformation debris can be found upon unloading, via *in situ* x-ray peak broadening measurements.<sup>24</sup>

In contrast, after deformation at LNT, the nc Ni showed extensive stacking faults and frequent deformation twins. As shown in Fig. 2, stacking faults extend from a high-angle ( $16^\circ$ ) equilibrium grain boundary into the grain interior, verifying that partials indeed were emitted from the grain boundary, consistent with the predictions of GPFE curves.<sup>12</sup> Figure 3 shows a deformation twin in a 25 nm grain. These observations indicate that partial-dislocation-mediated processes are prominent at the higher deformation stresses experienced by the nc Ni at LNT. This warrants a comparison with the MD partial dislocation processes, simulated at RT but high strain rates.

The observation of extensive stacking faults in nc Ni (Fig. 2) despite its high stable SFE indicates that the GPFE curves did play a significant role in the deformation of nc Ni at LNT. Specifically, the high unstable SFE and unstable twin fault energy in the GPFE curves hindered the nucleation of the trailing partials and twins, thus allowing a large number of stable stacking faults to exist in the nc grains. This is consistent with the observations in MD simulations.<sup>12</sup>

However, deformation twins in fairly large numbers were also observed in the nc Ni deformed at LNT (Fig. 3). This is an unlikely scenario considering GPFE curves alone, where  $(\gamma_{\text{utf}} - \gamma_{\text{sf}}) / \gamma_{\text{sf}}$  is as high as 0.9 for Ni. This raises the

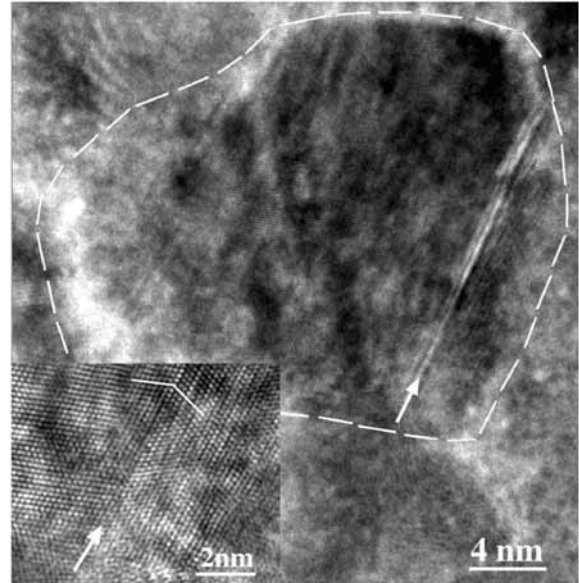


FIG. 3. An HRTEM image of nc Ni deformed at LNT showing a deformation twin. The twin boundary is marked by a white arrow. The inset shows a section of the twin at a higher magnification.

question if the applied stress is so high that during the tensile deformation we have spanned a wide stress range to enter scenario III. As discussed below, this is not the case.

We believe that the applied stress at LNT was in the stress range of scenario II, not scenario III, for the following two reasons. First, at LNT the maximum shear stress is 750 MPa, which is only 150 MPa (25%) higher than 600 MPa, the shear stress at RT. Because 600 MPa is not high enough to overcome  $\gamma_{\text{usf}}$  to produce stacking fault, it is unlikely that the 750 MPa is high enough to overcome  $\gamma_{\text{utf}}$  to produce twins, given the significant difference between  $\gamma_{\text{usf}}$  and  $\gamma_{\text{utf}}$  on the GPFE curves (see Fig. 1).

Second, a more quantitative analysis based on a recent analytical model also suggests that the applied stress was much lower than the critical stress required for overcoming  $\gamma_{\text{utf}}$ . The model predicted that an optimum diameter range exists in which nc face-centered-cubic metals are easiest to twin.<sup>25,26</sup> For Ni, this range is from 19 to 27 nm. Coincidentally, the average grain size of the nc Ni used here is 25 nm, which falls within this grain size range. The critical shear stress for twinning is calculated to be 1050–1070 MPa. Because the model does not consider unstable twin fault energy, the calculated stress is much lower than the stress for overcoming  $\gamma_{\text{utf}}$ .<sup>26,27</sup> In other words, the real critical stress for twin nucleation should be much higher than 1050 MPa. At LNT the experimental maximum shear stress is only 750 MPa. Assuming that at some grain boundary locations the 310 MPa residual stress is in such a character and orientation that it can be added numerically to the applied maximum shear stress, the total shear stress would be 1060 MPa, barely reaching 1050 MPa. This stress is still much lower than the critical stress required for overcoming  $\gamma_{\text{utf}}$ .

Apparently twinning was enabled despite of the relatively moderate applied shear stresses in a tensile test. In the following we discuss several factors that favor the twin nucleation, overcoming the high difficulty predicted by the GPFE curves. First, a stacking fault produces extremely high stress concentrations near itself.<sup>28</sup> For example, the shear

stress near a stacking fault in nc Cu may reach as high as 3.0–3.5 GPa. Such a high stress concentration could easily overcome the energy barrier for twin nucleation posed by the high  $\gamma_{\text{utf}}$ . Second, the twinning partial has the same Burgers vector as the leading partial that generated the stacking fault, while the orientation of the trailing partial is very different. Therefore, the applied stress that caused the emission of the leading partial also promotes the emission of the twinning partial instead of the trailing partial.<sup>28</sup> Third, stress concentrations exist at local grain boundaries. Although the emission of a leading partial relaxes the stress concentration, it could build up again near the same location to help with the nucleation of either the trailing partial or a twin.<sup>12</sup> Therefore, the high  $\gamma_{\text{utf}}$  has only limited effect on hindering the nucleation of twins in experimental samples. The GPFE curves alone are not sufficient to predict the twinning propensity.

Another factor that may affect the partial dislocation processes at LNT is the sluggish atomic reshuffling at the grain boundaries. MD simulation<sup>12</sup> has suggested that the emission of the leading partial may lower the local stress concentration, which delays the nucleation of the trailing partial. Lower deformation temperature could help concentrate stresses due to slower atomic mobility. The release of a partial without the need to change the Burgers vector is likely to be favored. However, it is not clear if this factor was important in our experiment, because the strain rate used was rather low. Further investigation is needed to clarify this issue.

In summary, our experimental observations indicate that the GPFE curves can predict stacking faults reasonably well, but do not adequately explain the twinning propensity in nc Ni. Despite a high stable SFE, the formation of stacking faults is facilitated by the much higher  $\gamma_{\text{utf}}$  and  $\gamma_{\text{usf}}$  when nc Ni is deformed in tension at LNT. However, the predictive power of the GPFE curves on the nucleation of twins is rather limited due to local stress concentrations associated with stacking faults and other structural inhomogeneities, as well as due to the favorable orientation of twinning partials. Twins appear to have a fairly good chance to nucleate in nc Ni once stacking faults are formed, despite of the high  $\gamma_{\text{utf}}$ . Finally, we note that this study is on nc Ni with near-equilibrium grain boundaries, and the conclusions here may not apply to nc materials with highly nonequilibrium grain boundaries, such as those produced by severe plastic deformation.

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