*Abnormal Grain Growth in Nanocrystalline Nickel**

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Motivation—Metals with nanometer-sized grains exhibit improved mechanical properties such as higher strength and hardness. Methods to improve their ductility and toughness are needed, however, and their stability at elevated temperatures needs to be examined. Grain growth in nanomaterials is expected to occur readily due to their high content of grain boundaries, but the mechanism needs to be determined and understood.

Accomplishment—We have imaged nanocrystalline Ni formed by pulsed laser deposition during in situ annealing in the transmission electron microscope (TEM). We found that this material evolves by "abnormal grain growth," in which isolated grains grow into the surrounding nanocrystalline matrix that remains unchanged. This growth was seen at temperatures as low as 225°C and up to 400°C; it proceeded erratically, with radial growth rates of $6x10^{-3}$ to 6 nm/s. Figure 1 shows partially and fully regrown films. The abnormal growth appears to occur by dissolution of adjacent grains and regrowth, instead of their rotating to align with the atomic lattice of the growing grain as might have occurred. No preferred orientation was found for the abnormal grains. We also found that the 200 kV electron beam of the TEM enhanced the growth rate by imaging either before or during annealing.

A key discovery is that numerous defects were incorporated into the growing grains, including dislocations, stacking faults, twins, and stacking fault tetrahedra (Fig. 2), in contrast with the widely-held expectation of defect-free growth. Stacking fault tetrahedra, which form from vacancies, are especially notable since they have been found only in fcc metals that were quenched from high temperature, irradiated, or perhaps heavily strained. Since we observed them while at modest temperatures, imaged them with electrons too low in energy to displace atoms, and produced only small thermal strains, their presence requires a new formation mechanism.

To understand abnormal grain growth more fundamentally, mesoscale (continuum) simulations of the thermal evolution of nanocrystalline Ni grains that used orientation-dependent grain-boundary energies and mobilities were performed. Simulations using such properties exhibited abnormal grain growth of grains with no preferred orientation, just as observed.

Significance—Our Ni has relatively uniform grain size and few impurities, and is ideal for understanding growth in nanocrystalline metals. In addition to determining the mechanism, our modeling indicates that grain-boundary properties play a key role in producing this abnormal growth. Growth enhancement by the electron beam may result from disrupting oxides on the film surface that might pin grain boundaries. The stacking fault tetrahedra appear specific to grain growth in a nanocrystalline matrix; we hypothesize that the atomic density deficit of the numerous high-angle grain boundaries is a source of vacancies that become trapped within growing grains, leading to the tetrahedra. Atomic-scale modeling of grain growth in nanocrystalline Ni is planned, which may shed light on the detailed processes producing the tetrahedra. Finally, our work indicates that nanocrystalline Ni is unsuited for uses at temperatures of 225°C or more, which would alter its internal structure and properties. However, abnormal growth can be used to tailor structures with large grains in a nanocrystalline matrix, which could exhibit increased ductility.

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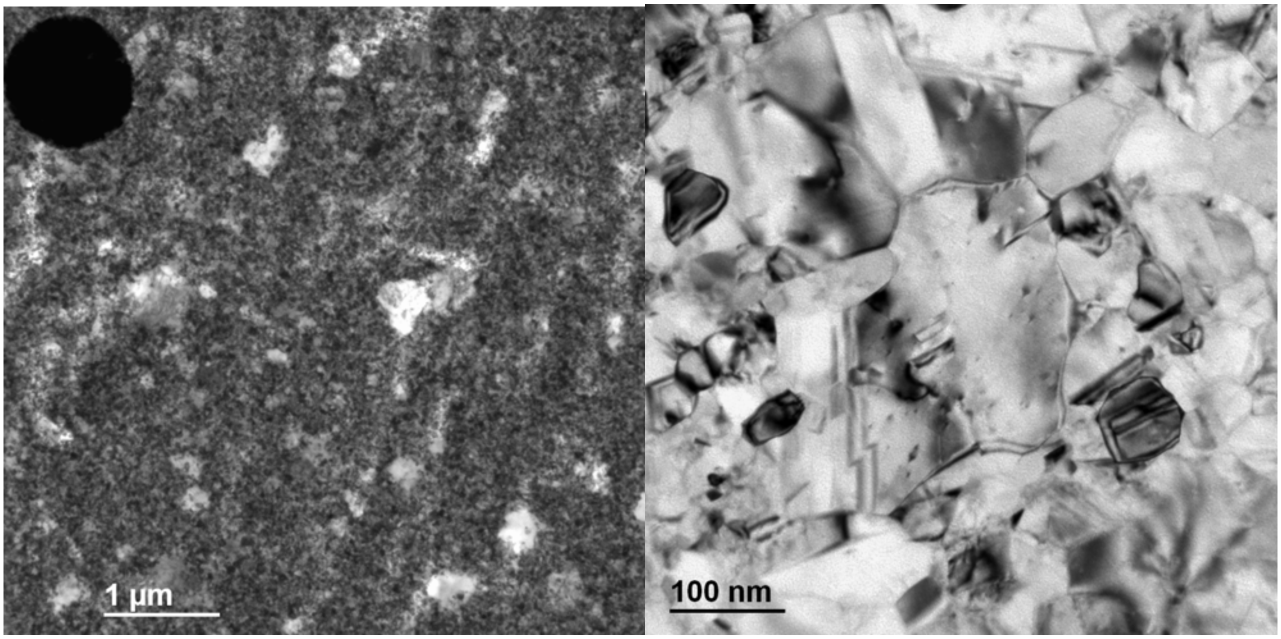

Figure 1. Left: TEM image of abnormal grains (light gray) in the nanocrystalline matrix (speckled); dark spot was used as a marker. The 150 nm-thick film was annealed 20 min. at 275ºC to partially react the film. Right: Image of near-fully reacted 80 nm-thick film, annealed 10 min. at 400ºC.

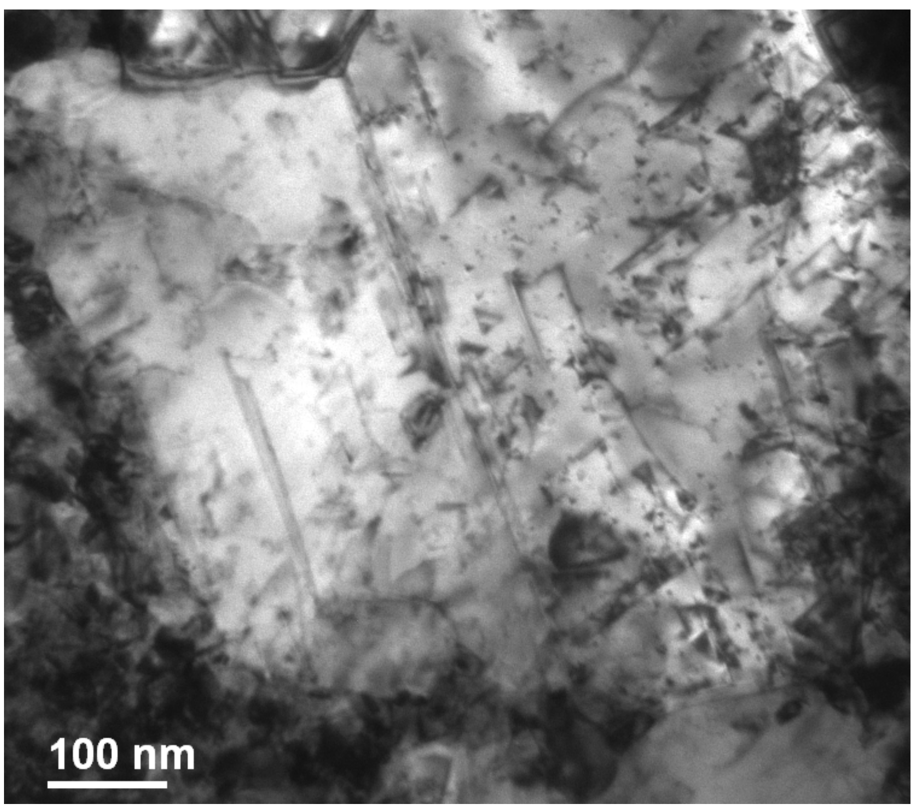

Figure 2. Higher magnification image of a grain from the film of Fig.1-left, showing many defects, including stacking fault tetrahedra (triangles). A large one (with stripes) is at the center.