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Strengthening and weakening by repeated dynamic impact in microcrystals and nanocrystals

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ABSTRACT

Experiments on micrograined (mg) and nanocrystalline (nc) Ni revealed strengthening and weakening following repeated dynamic impact. The strengthening in mg-Ni arises from intragranular dislocations without a significant change in grain size, whereas the weakening in nc-Ni is due to concurrent grain growth. The strength of mg and nc-Ni samples after deformation settles at ~900 MPa, with differing contributions from intragranular dislocations and grain sizes.

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1. Introduction

Can high-strength nanocrystalline metals be strengthened further by repeated dynamic impact: that is the question addressed in this report.

Strain hardening and grain refinement reduce dislocation mobility and they are potent strengthening mechanisms in conventional pure metals. The overall strength of pure metals is dictated by a combination of the intragranular dislocation content and the grain size, and this can be expressed as

$$\sigma = \sigma_0 + \sigma(\rho) + k_d\, d^{-1/2} \quad (1)$$

where σ is the strength at a grain size d, σ_0 is a friction stress, $\sigma(\rho)$ is the strengthening related to an intragranular dislocation content, and k_d is the Hall–Petch constant. In nanometals, there is a possibility of a negative or zero value for k_d [1,2], so that Hall–Petch strengthening for conventional metals may not apply when the grain size is typically less than ~10 nm. Since it is difficult to accumulate intragranular dislocations in nanocrystals with $d < 10$ nm, the intragranular dislocation strengthening component may not be important in such metals.

In many materials prepared by severe plastic deformation (SPD), involving the development of a cellular or subgrain structure that is converted to high angle boundaries, the grain size is refined and then stabilized at a minimum value depending on the testing temperature, stacking fault energy and composition [3–5]. In contrast, there is no external mechanical working in nanometals prepared by electrodeposition. Bulk nanocrystalline metals made by electrodeposition have high strengths, with a typical value of ~1 GPa for nano-Ni having a grain size of ~15–20 nm. The stability of such microstructures has been an important area of research; alloying and impurities can play a significant role in grain size stabilization [6,7].

Fig. 1 illustrates schematically the changes anticipated in the grain size and strength during large strain quasi-static deformation of metals. For conventional coarse-grained metals, the grain size gets refined with deformation (full line) leading to a concomitant increase in strength (dashed line). In contrast, for nanocrystalline metals, it is possible that concurrent grain growth during deformation (dashed line) will lead to weakening (full line). Thus, large strain deformation in coarse-grained metals and nanocrystals may have opposite trends in changes of grain size and strength.

Following an early study by Muller [8] in 1972 on dynamic deformation in Ni using a split-Hopkinson bar, there have been a few other studies on the material. Dynamic deformation in Ni with a grain size of ~4 μm by Dirras et al. [9] revealed an increase in dislocation density; at very high strain rates of $\sim 10^4\ s^{-1}$, dynamic recovery and recrystallization occurred due to the high temperatures from large-strain adiabatic deformation. A recently developed

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technique termed dynamic channel angular pressing, involving strain rates of $\sim 10^4\ s^{-1}$ and room temperature, has been shown to refine the grain size of Ni from an initial value of $\sim 200\ \mu m$ to $< 1\ \mu m$ after 3 passes [10].

In contrast to microcrystals, there is very limited information available on dynamic deformation in nanocrystals. Thus, for example, Liaw et al. [11] conducted split-Hopkinson bar experiments on nanocrystalline Ni–Fe and they reported a compression strength of ~ 2.2 GPa at a strain rate of $10^3\ s^{-1}$ compared to a value of ~ 2 GPa for quasi-static testing at $10^{-3}\ s^{-1}$. Slight flow softening was reported from ~ 2.2 to 2 GPa at a strain of ~ 0.15–0.2, and grain growth was observed from an initial value of ~ 19 nm to a final value of ~ 35 nm after dynamic deformation. Quasi-static testing at $10^{-3}\ s^{-1}$ led to an increase in grain size to ~ 23 nm at a lower strain of $\sim 7\%$.

Lu and colleagues [12–14] have developed surface treatment techniques and dynamic plastic deformation to refine the microstructure in a large range of materials such as steel, Cu and Ni. Comparison on Ni with a grain size of $\sim 10\ \mu m$ revealed that a dynamic plastic deformation approach (strain rate of $\sim 10^2\ s^{-1}$) refined the microstructure more than quasi-static deformation to the same total strain, by producing a higher frequency of low angle boundaries [14].

Repeated loading and unloading did not influence the compression strength of nano-Ni micropillars [15], suggesting that the microstructure was not modified significantly. However, sliding contact fatigue of nano-Ni led to significant grain growth in the vicinity of the scratches [16]. Similarly, while a nano-Ni with a grain size of ~ 20 nm is relatively stable during uniaxial loading [17], experiments with indentation suggest that mechanical loading substantially enhances grain growth [18].

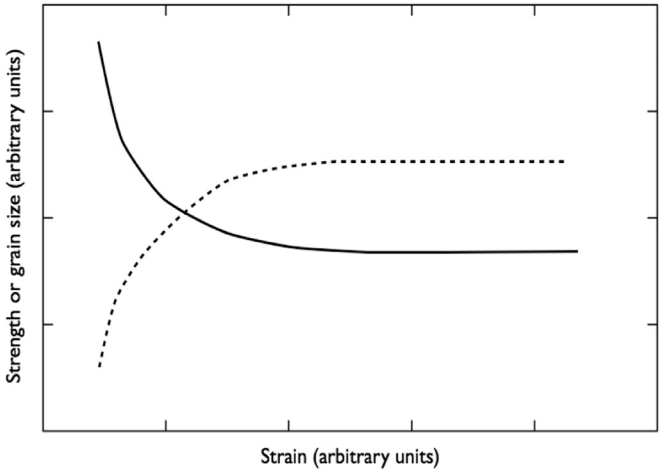

Fig. 1. Schematic illustration of the variation with strain in the strength and grain size. For conventional micrograined samples, the dashed and full lines correspond to strength and grain size, respectively. For nanocrystalline samples, the dashed and full lines correspond to grain size and strength, respectively.

Deformation in conventional metals leads to the development of an internal dislocation structure that causes both an increase in strength and a possibility of grain refinement. In contrast, nanocrystals are frequently prone to grain growth upon deformation. Thus, deformation-induced microstructural changes may have different contributions to strength from dislocations and grain size in conventional and nanometals. We examine in this report the influence of repeated dynamic impact on strength and microstructure evolution in microcrystalline and nanocrystalline Ni.

2. Experimental materials and procedure

This study uses a conventional micrograined (mg) rolled and recrystallized Ni (linear intercept $d = 4.2\ \mu m$, including twins) and an electrodeposited nanocrystalline (nc) Ni ($d = 20$ nm) produced by a standard pulsed electroplating technique [19]. The initial thicknesses of the mg-Ni and nc-Ni sheets were 200 and 170 μm, respectively. Polished and cleaned samples were attached with a tape to a 50 ml hardened steel jar of a Retsch cryo-mill and then milled using 10 hardened 10 mm steel balls at room temperature and 20 Hz frequency. Individual samples were milled for up to 25 min, with interruptions every minute to ensure that the samples remained stuck. The average hardness from 15 to 20 measurements was obtained using a Vickers microhardness tester at a load of 15 g. Microstructural analysis was performed on the electropolished mg-Ni samples using a high resolution scanning electron microscope (Zeiss Supra 40) equipped with an Bruker electron backscattered diffraction (EBSD) system.

The EBSD data were recorded at 0.15 μm step size and analyzed using HKL Channel 5 software. The misorientation angle (θ) profile (in 1° bin) was measured using 1.5° as a lower cut off. Boundaries with angles $1.5° < \theta < 15°$ were considered as low angle grain boundaries (LAGBs) whereas others were considered as high angle grain boundaries (HAGBs). The kernel average misorientation (KAM) was analyzed from the EBSD data, with 3° as the upper cut off angle. This component shows the average local misorientation around a given pixel (8 neighboring pixels used in this study) and can be used to determine the geometrically necessary dislocation (GND) densities [20]. The GND densities were also analyzed using the Atom software [21]. The average subgrain size λ (1.5–15°) and grain size d (15–65°) were obtained by a line intercept method.

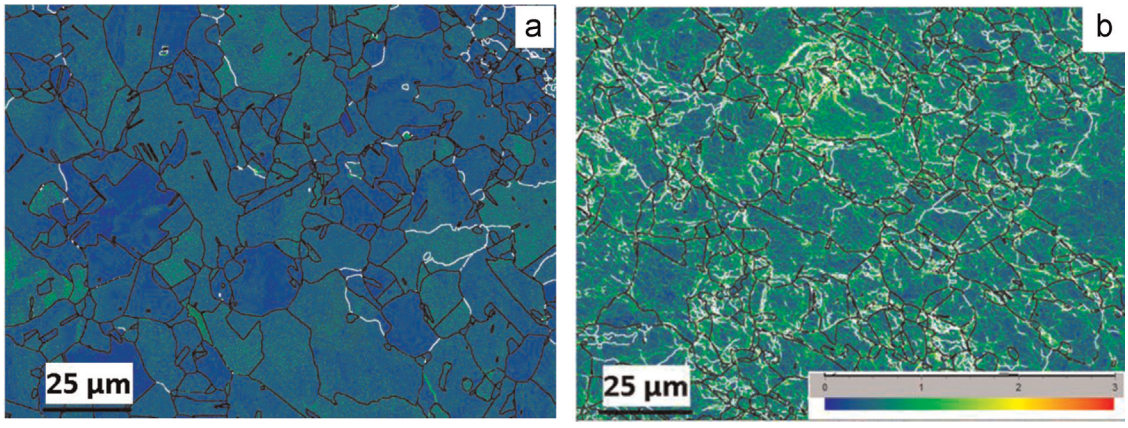

Fig. 2. EBSD micrographs of the mg-Ni (a) before and (b) after milling for 25 min. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article).

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