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# Nano-sized twins induce high rate sensitivity of flow stress in pure copper

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## Abstract

We have investigated the rate sensitivity of flow stress and the extent of strengthening in polycrystalline copper containing different volume fractions of nano-sized twins, but having the same average grain size. The specimens were produced by pulsed electrodeposition, wherein the concentration of twins was varied systematically by varying the processing parameters. Depth-sensing instrumented indentation experiments performed at loading rates spanning three orders of magnitude on specimens with the higher density of twins (twin lamellae width ~20 nm) revealed an up to sevenfold increase in rate-sensitivity of hardness compared to an essentially twin-free pure Cu of the same grain size. A reduction in twin density for the same grain size (with twin lamellae width ~90 nm) also resulted in a noticeable reduction in rate-sensitivity and hardness. The presence of a high density of nano-scale twins is also seen to impart significant hardness, which is comparable to that achieved in nano-grained Cu. Post-indentation analyses of indented Cu with nano-scale twins in the transmission electron microscope reveal deformation-induced displacement of coherent twin boundaries (CTBs), formation of steps and jogs along CTBs, and blockage of dislocations at CTBs. These processes appear to significantly influence the evolution of thermal activation volume for plastic flow which is some three orders of magnitude smaller than that known for microcrystalline Cu. Transmission electron microscopy also reveals CTBs with a high density of dislocation debris and points to the possibility that displaced CTBs may serve as barriers to dislocation motion and that they may also provide sources for dislocation nucleation, especially near stress concentrations, very much like grain boundaries. Possible consequences of these trends for deformation are explored.

*Keywords:* Nanostructured metals; Twins; Copper; Nanoindentation; Rate sensitivity; Activation volume

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## 1. Introduction

The strengthening of engineering metals and alloys through grain size reduction has long been a strategy for microstructure design [1,2]. Studies of nanostructured materials in the past two decades have shown that grain refinement in the nanometer range leads to substantial strengthening where abundant grain boundaries (GBs) create obstacles to dislocation motion [3–7].

Recent experimental observations have led to the discovery that nanostructured metals, with average grain size and range of grain size typically smaller than 100 nm, exhibit deformation characteristics that are highly sensitive to the rate of loading [8–11]. Uniaxial tensile experiments in electrodeposited nanocrystalline Cu [8] and tensile [9,10] and depth-sensing indentation [11] experiments in electrodeposited nanocrystalline Ni have established that an increase in loading/strain rate

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results in a pronounced increase in resistance to plastic flow. A systematic comparison by Schwaiger et al. [11] of loading rate and strain rate under controlled indentation of nominally defect-free, pure, electrodeposited Ni showed that nanocrystalline specimens (average grain size ~40 nm) were highly sensitive to the rate of deformation. However, microcrystalline (average grain size >1 μm) and ultra-fine crystalline (average grain size ~200 nm) specimens exhibited flow behavior that was relatively insensitive to the rate of loading. Strain-rate jump tests [12,13] performed on ultra-fine crystalline copper (average grain size ~200 nm) produced by equal-channel angular pressing (ECAP) with very high initial defect density, on the other hand, showed a strong rate-sensitivity of plastic flow.

The strain rate sensitivity of a ductile metal in uniaxial deformation is commonly written (e.g. see Refs. [12,14]) as

$$m = \frac{\sqrt{3}kT}{v^{*}\sigma} = \frac{3\sqrt{3}kT}{v^{*}H}, \tag{1}$$

where $m$ is a non-dimensional rate-sensitivity index, $k$ is the Boltzmann constant, $T$ is the absolute temperature, $\sigma$ is the flow stress, $H$ is the hardness (which is usually assumed to be three times the flow stress) and $v^*$ is the activation volume which is the rate of decrease of the activation enthalpy with respect to flow stress at a fixed temperature:

$$v^{*} = \sqrt{3}kT\left(\frac{\partial \ln \dot{\varepsilon}}{\partial \sigma}\right), \tag{2}$$

where $\dot{\varepsilon}$ is the strain rate. The rate-sensitivity parameter $m$ and the activation volume $v^*$ provide quantitative measures of the sensitivity of flow stress to loading rate and also give insights into the deformation mechanisms. Experimental studies suggest that the strain rate sensitivity $m$ of initially (nominally) defect-free nanocrystalline (NC) Cu and Ni is several times higher than that for the microcrystalline (MC) counterpart [8–11]. Furthermore, while the activation volume of conventional microcrystalline face-centered cubic (fcc) metals is on the order of $\sim 1000b^3$, where $b$ is the magnitude of the Burgers vector, the corresponding value for nanocrystalline fcc metals exhibiting enhanced rate sensitivity of plastic flow was found to be reduced by nearly two orders of magnitude (e.g. see Ref. [12]).

Like conventional grain boundaries, coherent twin boundaries (CTBs) can also obstruct the motion of dislocations [15]. This is the case for deformation, annealing, and growth twins, as long as they have interfaces with identical atomic structures [15]. Early studies of α-brass with twin spacing in the micrometer regime showed that twin boundaries (TBs) are equivalent to conventional GBs with respect to such strengthening phenomena as those arising from the Hall–Petch mechanism [16]. (A detailed review of strengthening due to CTBs in microcrystalline materials along with the associated references can be found in Ref. [15].) Exploiting the possibility that TBs could serve as effective barriers to dislocations, recent work has demonstrated that when nano-scale twins are introduced by the pulsed electrodeposition process in polycrystalline Cu and by magnetron sputter deposition in AISI 330 stainless steel [17–19] and in Ni–Co alloys [20], significant strengthening results.

The foregoing considerations naturally lead to two questions which, to our knowledge, have hitherto not been systematically examined:

1. Does the controlled introduction of twins in a polycrystalline fcc metal lead to markedly enhanced rate-sensitivity of plastic flow?
2. If so, what are the mechanistic contributions to such rate-sensitivity of deformation in twinned metals?

Developing an understanding of these issues would provide valuable insights into the mechanical deformation characteristics of a variety of engineering metals and alloys, in which twinning is a prominent mode of deformation. Such an understanding would also offer helpful information for microstructure design of engineering alloys wherein twinning is purposely orchestrated at the processing stage. Furthermore, elevated rate sensitivity may delay necking during tensile deformation and can possibly contribute to ductility. Therefore, fabrication steps that employ such methods as superplastic forming would also benefit from an improved understanding of the connections between twinning and rate-sensitivity of deformation.

In this work, we report a series of systematic experiments that demonstrate that controlled introduction of nano-sized twins in pure copper leads to a significant increase in the rate-sensitivity of plastic flow. For this purpose, we have produced specimens with different volume fractions of nano-sized twins in ultrafine-crystalline (UFC) Cu by recourse to pulsed electrodeposition, while keeping the grain size of the different specimens roughly the same. The rate sensitivity of plastic deformation was studied using depth-sensing instrumented indentation for which the loading rate was varied by three orders of magnitude. Significantly higher loading rate sensitivity is observed for the higher density of twin boundaries. Possible mechanisms contributing to this effect are explored.

## 2. Materials and experimental methods

### 2.1. Materials

High purity Cu sheets (20 mm × 10 mm × 0.1 mm in size) with nano-sized growth twin lamellae were synthe-