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Nanocrystalline material with anomalous modulus of resilience and springback effect

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ABSTRACT

Stability of nanocrystalline microstructural features allows structural materials to be synthesized and tested in ways that have heretofore been pursued only on a limited basis. Here, we demonstrate using quasi-static compression and three point bend tests that, in a stabilized nanocrystalline metal with tailored solute concentrations, i.e., NC-Cu-3 at.%Ta, extraordinary properties such as ultrahigh hardness along with anomalus modulus of resilience and springback effects can be manifested. Such effects influence a wide range of materials response including elastic energy absorption, damping, fatigue and wear. The present study, therefore, represents a pathway for designing highly resilient materials for everyday applications.

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A material's ability to significantly recover large elastic strains during deformation has a profound effect on everyday applications [1]. Such a measurement is known as “resilience”, i.e., a material's ability to absorb a large amount of energy per unit volume when elastically deformed and to subsequently release it upon unloading. Dictated by linear elastic mechanics, this unit of measure is enhanced by increasing the strength of a metal which exhibits a low elastic modulus. Such metals are ideal candidates for advanced spring, contact fatigue, and wear resistant applications, and are conventionally produced by casting, with alloys primarily comprised of Fe, Ni, Ti, or Cu as the base element, followed by wrought processing, quenching, and annealing for aging/tempering [1]. Because of their conditioning, many of these polycrystalline alloys can be used at low to moderately high temperatures (297–673 K) for a variety of applications including biomedical, agriculture, construction, military, and aerospace applications [2–4].

In general, strength/hardness and modulus of resilience are important indicators of a material's ability to resist permanent deformation during low load contact events in many applications. For instance, often in bio-medical applications, the modulus of resilience is correlated with clinical wear [5,6]. Similarly, in the cases of electrical contact or actuators in Microelectromechanical systems (MEMS), a high amount of mechanical energy storage before permanent deformation is critical [7,8]. Therefore, the purpose of this study is to evaluate the resilience of nanocrystalline (NC) materials in light of these potential applications. This is, in part, due to the fact that based on the strength attributed to the Hall-Petch relation [9–12], NC metals can easily exhibit an order of magnitude increase in yield strength as compared to their coarse grain counterparts [13–15], and therefore they have a tremendous capacity to store elastic strain energy and resist permanent deformation.

Resilience is an underpinning parameter that impacts a large range of material responses including elastic energy absorption, damping, fatigue, and wear. Curiously, however, resilience is a topic not readily discussed in the NC metals community despite numerous reports on the dramatic increases in the strength attained in metals or alloys with low elastic moduli, such as Al, Ti, and Cu. This likely stems from the inability to fully consolidate bulk structures and to achieve reasonable strength while maintaining grain size well below 100 nm under external stimuli. Thus, the balance of strength and microstructural stability has not been demonstrated in a NC metal, which would allow manifestation of highly resilient behavior during repeated loading events.

To demonstrate this behavior, a series of bulk NC-alloys of Cu with various Ta concentrations were processed using high-energy cryogenic mechanical alloying followed by consolidation of the powder to a bulk material using equal-channel angular extrusion at 973 K. Further details related to the processing, impurity levels, and microstructural stability can be found in [13,16–18]. Unlike recent publications by the authors on NC Cu-Ta alloys, this work varies the composition of Ta in Cu to obtain high strength and good resilience while maintaining microstructural stability (i.e., reduced stress induced grain growth) [13,16–18] and achieve a level of robustness to be an effective structural material. To assess grain size distributions and microstructural characteristics, Transmission Electron Microscopy (TEM) was employed. TEM characterization was carried out in the as-received and post-deformed conditions using an aberration corrected ARM-200F at 200 keV. Multiple bright field and dark field images were captured in both the high

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resolution TEM and STEM modes to assess the microstructure and quantify statistics such as grain size distribution, etc. For TEM characterization, samples were prepared using conventional procedures by thinning a 3 mm disk from the bulk specimen to approximately 70 μm. Subsequently, the specimens were dimpled to about a 5 μm thickness. Ion milling was performed using a Gatan Precision Ion Polishing System (PIPS) at liquid nitrogen temperatures to obtain electron-transparent regions in the specimens. The samples were also plasma cleaned in Ar prior to TEM observations to reduce contamination.

Primary microstructural characterization using TEM revealed a microstructure consisting of an average grain size of 50 nm and 80 nm for Cu-10 at.%Ta and Cu-3 at.%Ta alloys, respectively, in Figs. 1a–b. These images show that the matrix grains are equiaxed in nature and relatively free of lattice dislocation networks, with some twins present. Tantalum particles exhibit a range of sizes from atomic sized clusters (d < 14 nm) to larger particles (d > 14 nm). In higher magnification images shown in Fig. 1c, there is an extremely dense network of Ta based nanoclusters evident in the microstructure of the Cu-3 at.%Ta alloy. Similar images for 10%Ta can be found in the reference [13]. Statistical analysis reveals the mean nanocluster diameter to be 2 nm. The nanoclusters exist within the matrix and along grain boundaries, as shown in Fig. 1c. Tantalum based nanoclusters existing along or near grain boundaries were observed to be slightly larger in diameter as compared to the intergranular regions. This preferential coarsening is related to increased Ta diffusion kinetics along fast diffusion pathways such as grain boundaries and triple junctions [19]. These nanoclusters

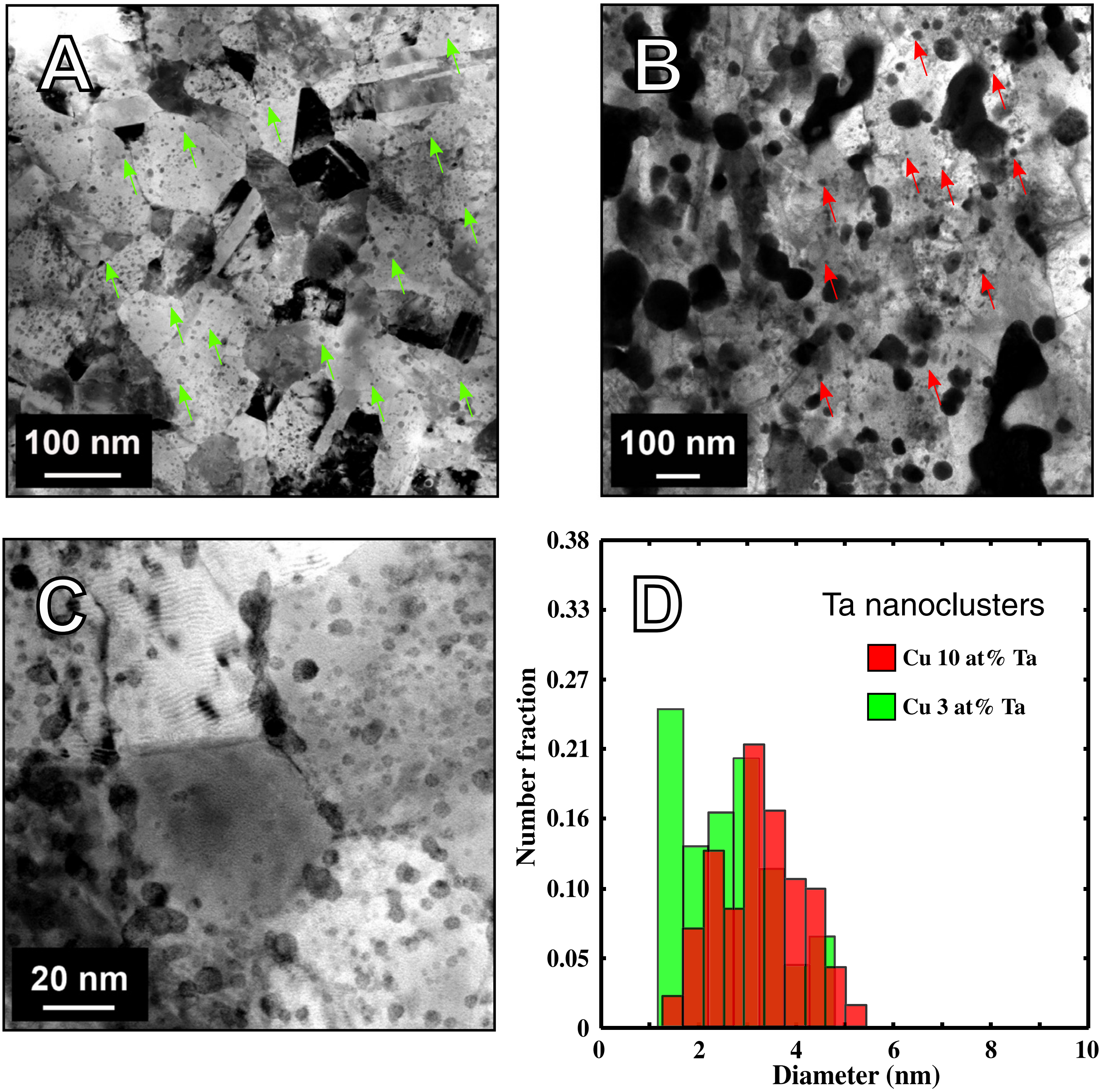

Fig. 1. Bright field STEM image indicating the features of as-received (a) Cu-3 at.%Ta alloy and (b) Cu-10 at.%Ta alloy. (c) Bright field STEM image of as-received Cu-3 at.%Ta alloy showing distribution of Ta nanoclusters (d < 14 nm). (d) size distribution of Ta nanoclusters in the as-received Cu-3 at.%Ta and Cu-10 at.%Ta alloys.