I. INTRODUCTION

The mechanical behavior of bulk nanostructured metals is not well understood or documented. A large volume fraction of boundary material might cause anomalous behavior, and for this reason, nanostructured solids have been regarded as composite materials made of a crystalline “phase” with long-range order and a more disordered grain boundary phase. Further, reports on the deformation behavior of nanostructured thin films of gold and silver have indicated that mechanisms other than dislocation motion, for example, grain boundary sliding and grain rotation, may become dominant in thin films, even at room temperature, for grain sizes of 25 nm and lower. However, studies of bulk nanostructured metals have been limited by poor densification, grain growth during consolidation, and contamination of materials.

The purpose of this study was to investigate the mechanical behavior of a fully dense, “bulk” nanostructured metal. The iron-copper system was selected for study in this research for several reasons. First, materials of immiscible systems such as Cu-Nb, Cu-Ta, and Cu-Fe have been shown to have desirable combinations of physical and mechanical properties. Johnson and co-workers reported on the mechanical alloying behavior of several binary compositions of the iron-copper system, which responded favorably to mechanical alloying. Although the equilibrium solubility of copper in iron is very small (<0.05 pct) near room temperature, ball milling of these powders led to the formation of supersaturated solid solutions with nanoscaled grain sizes (5 to 20 nm). The copper atoms were incorporated into the bcc iron lattice, forming a homogeneous, single-phase nanostructured powder alloy. Another reason for choosing the iron-copper system was that contamination by the steel balls and ball mill walls during processing would have a minimal effect on material composition and behavior. A composition of Fe with 10 at. pct Cu was selected so that the precipitation of copper particles might inhibit grain growth during consolidation.

In our previous studies of this alloy, its consolidation behavior and high-temperature mechanical behavior have been documented, and preliminary reports concerning deformation instabilities and associated mechanics modeling have been presented. A modified Hall–Petch equation was derived based on the proposed composite nature of the nanostructured metal. The present article gives details on materials processing and mechanical behavior and relates the respective observations to the behavior of other classes of materials. Further details are available in the thesis of Carsley, and deformation studies in thin nanostructured films can be found in References 4 and 5.

II. EXPERIMENTAL PROCEDURES

A. Materials Processing

The Fe-10 at. pct Cu alloys were prepared by ball milling 88.8 g of iron powder (99.9 pct purity) and 11.2 g of copper powder (99.5 pct purity) in a Szegvari 01HD attritor sys-
tem. The initial powders were ≤44 μm in diameter (−325 mesh). The milling media were 440C hardened stainless steel ball bearings (6.75-mm diameter). The media-to-powder weight ratio was 10:1. To limit contamination, the system was fully contained in an argon glove box, in which low levels of moisture (0.03 ppm) and oxygen (≤2.5 ppm) were maintained. Milling was conducted at 550 rpm for 24 hours, with the coolant temperature maintained below 0 °C.

The mechanically alloyed powder was “canned” in copper, in the same argon glove box, without any exposure to the atmosphere. Consolidation was performed by hot isostatic pressing (Hipping) at pressures of 170 or 410 MPa and at temperatures of 600 °C or 700 °C, for a hold time of 30 minutes. Finer grain sizes were obtained by powder forging, as described in Reference 16.

Consolidated samples were sectioned and machined into rectangular-shaped compression test samples. Most of these were individually encapsulated in quartz in order to limit oxidation during the annealing process. These were annealed at 700 °C for various times to coarsen the as-consolidated structure and to determine the corresponding changes in mechanical behavior. One hot isostatically pressed (HIP) sample was machined into rectangular, sheet-type tensile test samples which were also encapsulated and annealed.

B. Materials Characterization

The densities of the consolidated samples were determined using Archimedes’ principle, according to ASTM specifications for density measurements of porous materials. Relative densities were based on the measured values and the theoretical density, which was estimated by the rule of mixtures to be 7.98 g/cm³.

The X-ray diffraction analyses were performed on both powder and consolidated samples to investigate grain sizes, lattice parameters, degree of extended solubility, and composition. Patterns were obtained with a Scintag, Inc. (Cupertino, CA) XDS2000 diffractometer with nickel-filtered Cu Kα radiation. A graphite crystal monochromator was used to focus the diffracted beam and to reduce background radiation. However, the background remained appreciable in these studies due to the fluorescence of copper in the alloys. The Scintag DMS2000 Diffraction Management System, version 2.64, was used for instrument operation, data collection, and data analysis. The samples were scanned between 38 and 104 deg 2θ, to capture four iron peaks between {110} and {220}, as well as five copper peaks between {111} and {222}. All diffraction peaks were analyzed with Scintag’s profile-fitting software to subtract background radiation and to determine the integral peak intensity, peak breadth, and position of the peak centroid.

The Scherrer equation was used to calculate grain sizes for the powders and for the HIP compacts using peak breadth values from the Scintag software. Grain sizes based on the Scherrer equation were reported for the lowest-angle peaks (i.e., {110} for iron and {111} for copper). Fourier analysis was performed on the {110} and {220} iron peaks for several powder patterns. The coefficients were analyzed with the Warren–Averbach method to determine grain size and microstrain.

Optical microscopy and scanning electron microscopy (SEM) were used to estimate grain size and particle size of the annealed samples. Backscattered electron imaging with a JEOL* JSM-35C scanning microscope provided phase contrast in order to measure the copper grain size. Optical microscopy was used to measure the grain size of the iron matrix (etched with 2 pct nital) of the coarsest structures.

Deformation patterns on the surfaces of the tension and compression test samples were investigated optically using a stereo-optical microscope. The stereomicroscope was fitted with a video camera and recorder during several compression tests, to capture the evolution of the deformation patterns with increasing strain. Real-time recordings of the video camera, linked through the stereoscope, were calibrated with crosshead speed to correspond with load-deformation data. The initiation and propagation of the deformation patterns were related to the yield point and plastic response of the alloys.

Finally, transmission electron microscopy (TEM) was used to investigate the grain structure in the solid compacts and in several postdeformation samples. The TEM foils were prepared by slicing wafers with a diamond blade on a low-speed saw. Standard 3-mm disks were punched from the wafers after grinding to a thickness of 50 to 80 μm. The foils were further thinned by dimple grinding to a thickness of ~10 μm. Final thinning was done with an ion mill at room temperature at 5 kV. Milling times varied from 4 to 30 hours depending on foil thickness, milling angle (11 to 15 deg), and beam current (0.5 to 1.0 mA). The TEM studies were performed on a JEOL JEM-100CX electron microscope at 120 kV. The structures were investigated in both bright-field and dark-field imaging modes. Selected area diffraction patterns were indexed to identify phases present. The Cu {220} reflection was isolated in order to identify copper grains in the dark-field image. Bright-field imaging was used to quantify the grain size of the alloy by measuring several hundred grain diameters for each sample. Mean grain sizes were determined for consolidated samples and for samples annealed 8, 24, and 100 hours. The reported average grain sizes include both copper and iron phases, since they could not be distinguished in bright-field mode.

The deformation microstructures were also investigated by TEM. Foils were prepared as described previously, except that care was taken to dimple grind within the shear banded areas. Dislocation studies on the deformed grains were hindered by intense mottled contrast, which was due to both radiation damage during ion milling and magnetic interaction between the beam and ferromagnetic domains.

C. Mechanical Testing

Hardness tests were performed on polished samples with a Vickers indenter using a load of 10 kg and a duration of 15 seconds. The area of each indentation encompassed many thousands of grains for each test. At least ten measurements were averaged for each sample.

Compression samples were cut from the HIP compacts using a low-speed saw with a diamond blade. Various samples were then annealed as described earlier. The samples were ultimately polished with 0.05 μm colloidal silica, to