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Microstructure and mechanical properties of ultrafine grained Cu-0.8 wt%C alloy with a bimodal microstructure produced by powder metallurgy techniques

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ABSTRACT

Bulk ultrafine grained Cu-0.8 wt%C alloy samples were fabricated by spark plasma sintering (SPS) of a nanocrystalline Cu-0.8 wt%C alloy powder prepared by high energy mechanical milling. The SPS temperature was 800 °C. The SPSed samples exhibited a bimodal microstructure consisting of ultrafine and coarse Cu grains (average grain sizes: ~95 nm and ~1 μm respectively) and a yield strength of 483 MPa, but underwent a premature fracture at a stress of 511 MPa with a low elongation to fracture of only 0.3%. It is established that the premature fracture and low tensile ductility are caused by the high flow stress associated with grain boundary strengthening and reaching the strength of IPBs before reaching its maximum. Hot extrusion of the SPSed sample at 800 °C caused substantial growth of the ultrafine Cu grains and enhancement of the strength of IPBs, leading to a clearly lower tensile yield strength of 353 MPa, but mature fracture with an ultimate tensile strength of 428 MPa and a significantly improved elongation to fracture of 6.5%.

1. Introduction

Bulk ultrafine grained (UFG) metallic materials have attracted close attention of many researchers due to their potential to have excellent mechanical properties for engineering applications, including high strength which can be two or more times of that of their coarse grained counterparts [1]. However, bulk UFG metallic materials generally exhibit a low tensile ductility at room temperature because of their low strain hardening capacity during plastic deformation [2], and this may limit their practical applications. It was found that UFG metallic materials with a bimodal microstructure can possess a combination of high strength and good tensile ductility [3–8]. The ultrafine grains in the bimodal microstructure render the material with a high strength due to grain boundary strengthening, while the coarse grains (larger than 1 μm) provide the strain hardening capacity to maintain the tensile deformation stability needed for good tensile ductility.

Bulk UFG metallic materials can usually be synthesized by either severe plastic deformation (SPD) of bulk coarse grained metallic materials followed by thermal annealing under controlled conditions [3], or thermomechanical consolidation of nanocrystalline powders [8–12]. The SPD processes include equal channel angular pressing, high pressure torsion and rolling. The nanocrystalline metallic powders can be typically produced by high energy mechanical milling (HEMM) of a metallic powder or powder mixture.

Wang et al. [3] fabricated an UFG Cu sample with a bimodal microstructure consisting of micrometer-sized grains embedded in a matrix of Cu nanograins (< 100 nm) and ultrafine Cu grains (< 300 nm) by rolling a Cu sample in liquid nitrogen to achieve 93% deformation followed by annealing the cold worked sample at 200 °C for 3 min. It was found that the Cu nanograins and ultrafine grains imparted a high yield strength of 340 MPa and the bimodal microstructure led to a high elongation to fracture of 65% including an uniform elongation of 30%. Guo et al. [5] fabricated a trimodal-structured Zr sample composed of ~24% coarse grains (> 1 μm), ~56% ultrafine grains (100–1000 nm), and ~20% nanoscale grains and subgrains (≤100 nm) by cryorolling combined with low-temperature annealing. The sample had a much enhanced ultimate tensile strength (UTS) of 658 MPa and a uniform elongation of 8.5% which is high for nanocrystalline metallic materials.

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Similar results were reported for an Al-Mg alloy [4]. In this work, three powder blends with the ratios of 100:0, 85:15 and 70:30, respectively, between cryomilled nanocrystalline and unmilled coarse grained Al-7.5at%Mg alloy powders were compacted by uniaxial pressing, and then the powder compacts were hot extruded to produce rods. The extruded rods fabricated with an addition of unmilled powder exhibited a bimodal microstructure consisting of nanocrystalline matrix and coarse-grained bands with an average grain size of ~ 1 μm. The tensile yield strength of the extruded rods decreased, and their elongation to fracture increased with increasing the volume fraction of the coarse grained powder. These studies demonstrated that a bimodal microstructure consisting of a matrix of nanograins or ultrafine grains and a volume fraction of coarse grains is highly desirable for achieving a combination of high strength and good ductility needed by engineering applications.

In this study, we demonstrate that spark plasma sintering (SPS) of a nanocrystalline Cu-0.8 wt%C alloy powder can lead to formation of a bimodal microstructure consisting of ultrafine and coarse Cu grains and amorphous C nanoparticles distributed at grain boundaries in the ultrafine grained regions. We also show that hot extrusion of the SPSed sample enhances the strength of the interparticle boundaries and leads to much improved tensile ductility of the material, while still maintaining a fairly high yield strength.

2. Experimental procedures

A nanocrystalline Cu-0.8 wt%C alloy powder was prepared by HEMM of 100 g Cu powder (99.99 wt% purity and particle sizes < 45 μm) and 1 g stearic acid ($CH_3(CH_2)_{16}COOH$) as a process control agent (PCA). The HEMM experiments were performed under argon and with a QM-3SP4 planetary ball mill (Nanjing Nanda Instrument Ltd.) and using 6 stainless steel balls of 25 mm in diameter and 13 stainless steel balls of 12.5 mm in diameter. The ball to powder weight ratio was 5:1. The powder was mixed for 3 h with a speed of 200 rpm prior to HEMM, and then the mixed powder was milled for 48 h with a speed of 500 rpm with 30 min break after every 3 h of milling. Subsequently, the milled powder was transferred to a cylindrical graphite die with a diameter of 28 mm and sintered at 800 ℃ using a SPS furnace (KCE-FCT-HP D 25/4-SD) under a pressure of 50 MPa and with a holding time of 5 min. The heating rate used for SPS was 100 ℃/min. One of the SPSed samples was heated to 800 ℃ by an induction coil, held at the temperature for 2 min, and then extruded into a cylindrical rod with an extrusion ratio of 9:1. The chemical compositions of the as-SPSed sample and as-extruded rod were determined by using an infrared absorption carbon–sulfur analyzer (Eltra CS800, for carbon contents), an inductive coupling plasma emission spectrometer (ICP, ICAP 7600, for detectable Fe, Cr, Mn, Ni contents), and gas chromatograph (SQM-2L, for oxygen contents), respectively. The results of the chemical analyses are shown in Table 1.

The density of the consolidated samples was determined using an automatic density meter DH-120M and based on the Archimedes' principle. The relative density of the samples was calculated using 8.66 g/cm^3 for the value of the theoretical density of Cu-0.8 wt%C alloy determined based on the theoretical density of pure copper (8.96 g/cm^3) and that of carbon (0.76 g/cm^3).

Table 1
The chemical compositions of the as-SPSed and as-extruded Cu-0.8 wt%C alloy samples respectively.

sample	Fe/wt%	Cr/wt%	Mn/wt%	Ni/wt%	O/wt%	C/wt%
as-SPSed Cu-0.8 wt %C	0.15	0.07	0.005	0.04	0.24	0.762
as-extruded Cu-0.8 wt%C	0.15	0.07	0.006	0.06	0.28	0.779

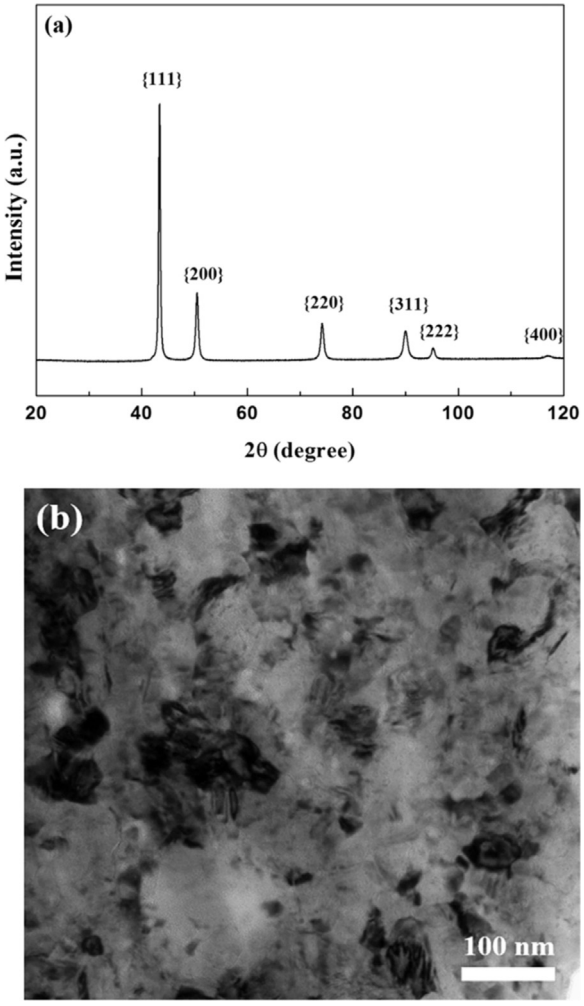

Fig. 1. (a) XRD pattern and (b) TEM bright field micrograph of the as-milled nanocrystalline Cu-0.8 wt%C alloy powder particles.

The as-milled powder and consolidated samples were examined by using an X-ray diffractometer (XRD) (Rigaku Ultima IV) with a step size of 0.02° and a scanning speed of 10°/min. For the as-SPSed sample, the XRD analysis was done on its transverse section perpendicular to the pessing direction. For the as-extruded rod, the XRD analysis was done the longitudinal section parallel to the extrusion direction. For comparison, XRD analysis was also performed on as-received Cu powder using the same procedure. The average grain sizes were calculated using the full width at half maximum (FWHM) of the peaks of the XRD patterns and the Williamson-Hall equation [13].

The microstructures of the as-SPSed and as-extruded samples were characterized with an optical microscopy (OLMPUS BX51M), scanning electron microscopy (SEM) (FEI Nova Nano SEM 230) together with energy-dispersive spectrometry (EDS) and transmission electron microscopy (TEM) (JEM-2100F) together with EDS. The size distributions of the ultrafine grains in the as-SPSed and as-extruded samples were determined by analyzing TEM digital micrographs. The size distributions of the coarse grains in the samples were obtained by analyzing the images of coarse grains shown in the optical microscopy micrographs. For each size distribution and associated average grain size determination, at least 100 Cu grains were analyzed. Vickers microhardness measurements were performed on polished samples with a load of 500 g and a dwelling time of 10 s. At least ten measurements were made for each value of the microhardness.

Dog-bone shaped tensile test specimens with a rectangular cross-section of 2 × 3 mm^2 and a gauge length of 15 mm cut from the as-