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Microstructural and texture evolution of copper-(chromium, molybdenum, tungsten) composites deformed by high-pressure-torsion

Julian M. Rosalie*, Pradipta Ghosh, Jinming Guo, Oliver Renk, Zaoli Zhang

Austrian Academy of Sciences, Erich Schmid Institute of Materials Science, A-8700 Leoben, Austria

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ABSTRACT

Cu-refractory metal composites containing Cr, Mo or W were subjected to severe plastic deformation using room temperature high-pressure torsion (HPT). A lamellar microstructure developed in each of the composites at equivalent strains of ~75. The refractory metals developed {*hkl*}⟨111⟩ fibre textures with a slight tilt to the tangential direction. This texture was stronger and more clearly defined in Mo and W than in Cr.

By applying additional HPT deformation to these samples, perpendicular to the original shear strain, it was found that the lamellar structure of Cu30Mo70 and Cu20W80 (wt%) composites could be retained at high equivalent strains and the refractory layer thickness could be reduced to 20–50 nm in Cu20W80 and 10–20 nm in Cu30Mo70. Although neighbouring regions of the microstructure were aligned and there was evidence of local texture in both composites, the bulk texture of Cu30Mo70 became weaker during this second step of HPT deformation. This was attributed to the refractory metal lamellae being discontinuous and imperfectly aligned.

This work shows that it is possible to form ultrafine composites of Cu-group VI refractory metals via high-pressure torsion, with namolamellar structures being possible where there is a sufficient volume fraction of Mo or W.

1. Introduction

Copper-refractory metal composites are important industrial materials consisting of varying proportions of copper and one or more of the refractory metals, namely Nb, Ta, Cr, Mo, W and Re. These composites are employed in high temperature, high current applications such as heat sinks [1], electrodes in thermoelectric devices [1-3] and in aerospace components [4] which demand high thermal and/or electrical conductivity of Cu and the capability to operate at higher temperatures than copper alone can sustain. The high melting points (from 1907 °C (Cr) to 3422 °C (W)) [5] and low thermal expansion coefficients (4.5–7.3 $\mu m\, m^{-1}\, K^{-1}$) [6] of the refractory elements provide the required high temperature strength and dimensional stability.

Nanostructured forms of these composites have been produced by severe plastic deformation (SPD). While techniques such as accumulative cold drawing and bundling (ADB) and equal channel angular extrusion (ECAE) have been used [7, 8], the best studied examples are the Cu-Nb composites produced by accumulative roll-bonding (ARB) [9-14] by which it has been possible to produce lamellar structures with widths of 10–20 nm [9, 12-14].

The lamellar microstructure endows these composites with excellent thermal stability [12, 15] by restricting diffusion through the alternating layers. Fracture toughness studies on lamellar composites have also shown a strong anisotropy due to crack deflection along the lamellae [16]. Thus, the ability to develop nanolamellar composites of refractory elements in Cu matrix will open-up the possibility for developing high strength, tough and thermally stable components for high temperature applications.

The production of such nanolaminates relies on the high ductility of the group V refractory metals, with ductile to brittle transformation temperatures (DBTTs) of ~77 K [17, 18] and ~4 K [19] for Nb and Ta, respectively. Unfortunately, the group VI refractory metals (Cr, Mo, W) are more brittle, with DBTTs in the range of 553–573 K for 99.96% purity Cr [20], about 373 K [21] for Mo and 400 K for W [22].

High-pressure torsion (HPT) is a technique for applying high strains under quasi-hydrostatic conditions, thus permitting the deformation of brittle materials. This has been applied to Cu-Cr [23-25] Cu-W [26-28] and Cu-Mo [29] composites. Extensive deformation of Cu-Cr [24] results in the formation of equiaxed, nanometer scale grains. A similar microstructure was reported after two-step deformation of Cu50Mo50 [29]. In contrast, a Cu30Mo70 (wt %) composite retained a lamellar structure with layer widths of ~5 nm for Cu and ~10–20 nm for Mo [29]. It appears that Cu-10 wt %W and Cu-25 wt %W composites also retained lamellar structures for strains up to at least 900 [28].

* Corresponding author.
E-mail address: julian.rosalie@oeaw.ac.at (J.M. Rosalie).

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However, each of these studies were conducted using different sample sizes, pressure and applied strain, making it difficult to compare various Cu-refractory metal composites.

This work sets out to provide a comparison of the microstructural evolution of copper composites containing a group VI refractory element (Cr, Mo, W) during equivalent room-temperature HPT deformation. In particular it examines the extent of microstructural refinement, the formation and decomposition of lamellar microstructures and the development of crystallographic texture during deformation.

2. Experimental details

The materials used in this work were provided by Plansee SE, Austria. Cu-Mo and Cu-W composites took the form of liquid-metal infiltrated (LMI) plates, with compositions of Cu30Mo70, Cu50Mo50 and Cu20W80 (all compositions are given in wt %). Cu-Cr was in the form of 4 mm slices sectioned from directionally-solidified Cu57Cr43 billets.

Step 1 HPT was carried out at room temperature on a 4000 kN apparatus rotating at 0.0625 revolutions per minute (rpm) under an applied pressure of 4.5 GPa. The sample thickness, t and number of revolutions, N, were adjusted to achieve a nominal von Mises' equivalent strain, ϵ_{eq} of ~75 at a given radius, r, based on the equation [30]:

$$\epsilon_{eq} \cong \frac{\gamma}{\sqrt{3}} = \frac{2\pi r N}{\sqrt{3}\,t}. \quad (1)$$

Samples were constrained within tool steel anvils of diameter 30 mm and anvil gap 3.5 mm (Cu-Mo, Cu-W) or 1.5 mm (Cu-Cr), corresponding to a nominal sample thickness of 7.5 mm and 3.5 mm, respectively. Table 1 lists the deformation conditions for each composite, and gives the sampling radius and equivalent strain used for XRD analysis.

Vickers microindentation hardness testing was conducted on cross-sectioned samples of each HPT-deformed composites, using 300 g or 500 g loads.

The as-received and post-deformation microstructures of each composite were examined using a Gemini 1525 scanning electron microscope (SEM). Sample preparation was via mechanical polishing and grinding, with final polishing using a Buehler vibromet polisher.

Composites were then subjected to higher-strain HPT deformation to further refine the microstructure. For Mo and W-based composites, the experiments used the two-step method developed by Bachmaier et. al. [31]. Cylinders were cut from the deformed 30 mm diameter Cu-Mo and Cu-W samples, and sliced into discs with a nominal height of 1 mm. (The central axis of the cylinder was at a distance of approximately 8 mm from the centre of the 30 mm diameter disc.) These were deformed at a pressure of 7.3 GPa in anvils with a cavity height of 0.20 or 0.25 mm. A total of 20–50 rotations of HPT deformation were applied, at a rotation rate of 0.4 rpm. Because the shear plane in step 2 is perpendicular to that in step 1, this method has been shown to be particularly effective for microstructural refinement [31]. Unfortunately, the 4 mm thickness of the Cu-Cr starting material did not permit this procedure. Therefore, 8 mm diameter discs of the as-received material with a nominal height of 1 mm were deformed to 100 rotations (this was considered acceptable since previous studies on this material showed minimal change in hardness and grain size between deformation of 100 and to 1000 rotations [25]). Hardness testing was conducted to determine appropriate strain levels for further analysis. The sample size and deformation conditions for step 2 HPT samples are set out in Table 2.

Table 1
Sample compositions and deformation conditions for step 1 HPT. The von Mises' equivalent strain ϵ_1 is given for r': the radial distance at which the XRD samples were extracted (see Fig. 1).

	% Cu (wt)	Thickness t (mm)	Rotations N	Radial distance r' (mm)	Strain ϵ_1
Cu-Cr	57	3.5	5	15	78
Cu-Mo	50	7.5	10	15	73
	30	7.5	10	15	73
Cu-W	20	7.5	20	8	77

Table 2
Sample compositions and deformation conditions for step 2 HPT. In the case of Cu57Cr43, a single step deformation with a higher number of rotations was used instead, as described in the text. The von Mises' equivalent strains (ϵ_1 and ϵ_2) for step 1 and step 2 deformation are listed for the conditions examined via TEM and synchrotron XRD.

	% Cu (wt)	Thickness $t_1 : t_2$ (mm)	Rotations $N_1 : N_2$	Radial distance r' (mm)	Strain $\epsilon_1 : \epsilon_2$
Cu-Cr	57	–:0.7	–:100	–:3	–:1500
Cu-Mo	50	7.5:0.7	10:20*	8:3	50:400
	50	7.5:0.7	10:50†	8:3	50:1000
	30	7.5:0.7	10:50	8:3	50:1000
Cu-W	20	7.5:0.7	20:35	8:3	100:700

* Synchrotron XRD.
† TEM.

Suitably-deformed HPT discs were mechanically ground and polished, dimpled and thinned to perforation using a Gatan 691 precision ion polisher. The electron transparent region corresponded with a radial distance of ~3 mm. The corresponding strains ($\epsilon_1 : \epsilon_2$) were ~50:1000 for Cu-Mo, ~100:700 for Cu-W and ~1500 for Cu-Cr (ϵ_2 only) composites. These foils were examined using a JEOL 2100F transmission electron microscope, operating at 200 kV using high-angle annular dark field scanning TEM (HAADF-STEM). Inner and outer collection angles of 65.51 mrad and 174.9 mrad, respectively, were used to provide atomic contrast imaging.

The bulk crystallographic texture of samples deformed till ϵ_{eq} ~ 75 (i.e, first step of HPT) were measured using a Rigaku Smartlab system with Cu Kα radiation. An approximate ~4 mm × 4 mm samples normal to the radial direction were used for these measurements. The defocusing corrections due to sample tilts were made using powders or powder compacts of the same size and composition. The texture measurements on the second step HPT deformed samples were performed with synchrotron radiation at the high energy materials science beamline P07 (operated by the Helmholtz-Zentrum Geesthacht) of the PETRA III synchrotron facility at DESY in Hamburg with photon energies of 101 keV. For this experiment, radial slices with a nominal width of 1 mm were cut from the HPT discs (see Fig. 1 (c)). The transmitted diffraction rings were measured with a beam size of 0.8 mm (high) × 1 mm (wide). The samples were rotated about their radial axis by 180° with an interval of 5° in order to obtain three-dimensional texture information. Further, the intensity of the diffracted beams were corrected for the attenuation. A LaB_6 NIST standard was used for calibrating the detector distance and orientation with respect to the sample. For both techniques, the measured pole figures were analysed with Labosoft software. For W and Mo (110), (200) and (211) pole figures were measured while for Cr (200), (211) and (022) pole figures were measured, (due to overlap of the Cu (111) and Cr (110) peaks). The measured pole figures were used to calculate the orientation distribution function (ODF), from which the (110) and (222) projections were recalculated.

3. Results

3.1. Hardness

The hardness of the composites after HPT deformation is plotted