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Enhanced microstructural homogeneity in metal-matrix composites developed under high-pressure-double-torsion

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ABSTRACT

In this work, metal-matrix composites of commercially pure copper (Cu) and silicon carbide (SiC) are synthesized by standard high-pressure torsion (HPT) and novel high-pressure-double-torsion (HPDT) processes of severe plastic deformation (SPD). Based on detailed microstructural examinations, it is found that significant homogeneity of fine particle distribution as well as weakness of crystallographic texture can be obtained in the composites fabricated using these processes. The most homogeneous particle distribution is obtained in the material processed under HPDT, which imposes the highest strain levels. Crystallographic texture in the composite is found to be weaker than in monolithic Cu processed under the same processing conditions. The randomization of texture in the composites is linked to the homogenization of the particle distribution.

1. Introduction

Metal matrix composites (MMCs) are a class of material systems consisting of at least two constituent materials, one of which is a metal. These material systems have proven effective in enhancing a range of material properties and continue to attract major attention within the material community. Examples of enhanced material properties include high strength, high strength-to-weight ratio, high corrosion resistance, and low thermal conductivity [1,2]. MMCs with fine particles ($<5\ \mu m$) are found to be particularly effective in enhancing strength [3], corrosion resistance [4], wear resistance [5], thermal [6], and electrical conductivities [7]. However, MMCs with fine particles can exhibit large microstructural inhomogeneity in particle distribution [8]. Such inhomogeneity creates large internal gradients in properties and stress–strain fields, which limit their ductility and make them prone to premature failure. Conventional deformation processes such as rolling and extrusion have been used extensively to improve the homogeneity of particle distribution in these composites [9–11]. It was found that improving the homogeneity requires very high strains (>4), especially for composites containing fine particles [9]. The processes of severe plastic deformation (SPD) have attracted much attention due to their ability to improve the homogeneity of particle distribution. The efficiency at which these techniques can improve the homogeneity depends on the level of strain possible to impose on a material system by a particular technique. While SPD processes such as equal channel angular pressing (ECAP) [12] and accumulative roll bonding (ARB) [13] have been found effective for improving the distributions, the best particle distribution thus far was obtained using the high-pressure torsion (HPT) processing [14]. In HPT, the sample is deformed by torsional turns under pressure. Similarly, the HPT technique was the most successful over other SPD techniques in refining the grain structure. In particular, HPT is more efficient compared to ECAE and ARB [15]. For example, while HPT can easily impose a strain level of 10 in only a few turns, the equivalent strain level achieved by ECAE would require at least 10 passes, which makes the later process impractical. In addition, it was found that grain sizes in Cu can refine to 150 nm by HPT [16], whereas it saturates to 270 nm after 6 ECAE passes [17]. To enhance refinement with fewer processing steps, researchers have combined more than one SPD technique, such as ECAE followed by HPT [18] or ARB followed by HPT [19]. Recently, an extension of the HPT process called high pressure double torsion (HPDT) was introduced, as a way of increasing the amount of strain imposed per turn and hence the grain size refinement was more efficient compared to standard HPT [20,21]. The main difference is that in HPDT both sides of the disk are being rotated, but in opposing directions. In this way, HPDT applies theoretically twice as much plastic strain per revolution than HPT. The amount of strain ($\gg 4$) imposed on a sample increases with the number

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of turns. HPT has been utilized to successfully fabricate Al–10 wt.% SiC [14], Al–10 wt.% Fe [22], and W–25 wt.% Cu [23] composites. In comparison with other fabrication methodologies, significant improvements were made in the homogeneity of particle size and distribution [24].

In this paper, Cu–20 vol.% SiC composites were fabricated using standard high-pressure torsion (HPT) process and the recently developed HPDT process [20]. Previously, HPDT has been used to fabricate samples of pure Cu [20]. For pure Cu, the study reported significant enhancements in grain size refinement and reduction in microstructural gradients. Here, homogeneity in the microstructure of MMC samples processed using HPDT is studied. In particular, the focus is on the homogeneity of particle distribution and crystallographic texture.

Crystallographic texture is an important feature of the microstructure in polycrystalline materials known to have a strong influence on the anisotropy of elastic [25,26], plastic [27,28], and physical [29] material properties. Therefore, a way of designing materials for anisotropy is to manipulate the distribution of the crystal orientation. Crystal structure and orientations are directly linked to the activation of microscale deformation mechanisms [30–42]. A possible method of altering deformation textures in polycrystals is to change the dislocation glide patterns in the constituent grains undergoing plastic deformation by either (a) imposed macroscopic strains or (b) local strains during a synthesis. While the first approach is more expensive and requires changes in processing equipment, the second involves changes in the local microstructures. Microstructural changes can be achieved by the addition of either a solute that can form precipitates and dispersoids [43,44] or a small volume fraction of non-deformable second phase particles; the latter yields a MMC [45,46]. Non-deforming particles larger than about 0.1 μm are known to alter textures through either the introduction of deformation zones which “disperse” sharp texture components because of large local rotations [45], or by particle stimulated nucleation through the formation of localized recrystallized grains at particle–matrix interfaces [45]. As a consequence, the metallic matrix in these materials should exhibit less preferred orientation distributions than the materials without any reinforcements, which deform by crystallographic slip not influenced by interactions with hard particles [47].

In this work, homogeneity of fine particle distribution is investigated in the MMCs synthesized using HPDT and HPT. Additionally, development of crystallographic texture in the composites is also examined. These textures are compared with texture in the monolithic Cu developed under the same processing conditions. Quantitatively it is demonstrated that significant improvements in the homogeneity of fine particle distribution and crystallographic texture distributions can be obtained using HPDT. Homogeneity of particle distribution in these composites is attributed to fast shrinkage in the particle-free zones because of large strains imposed by the process. The weak texture was found to be a concomitant process to the particle distribution homogenization.

2. Experimental and numerical procedures

Fig. 1a and b schematically shows the manufacturing procedures highlighting the differences between the HPDT and HPT processes. In HPT, one anvil rotates while in HPDT both anvils rotate in opposite directions imposing theoretically twice as much strain. In our earlier work [20], faster grain refinement was observed in the material processed under HPDT than under HPT. To make a fair comparison between HPT and HPDT processes, the number of turns in HPT should be twice as much as that in HPDT when the anvils rotate with the same speed. The HPDT process imposes larger strain rates than HPT because both ends rotate with the same speed. Larger strain rates generate higher temperatures within the tested sample because there is less time to evacuate the heat generated by plastic deformation. The increase in temperature increases the friction coefficient and therefore imposes more strain on the sample than expected by the theoretical factor of two.

Commercially pure Cu and β-SiC particulate powders with average particle sizes of 30 and 5 μm, respectively were used as starting materials, while stearic acid powders in the amount of 1 wt.% of the total weight were used as the process control agent (PCA). The mixing of the copper and the 20 vol.% of SiC powders was carried out by jar milling for 30 min. Milling medium was not used during the mixing. Since the milling process was short, any introduced plastic deformation was minimal. The mixed powders were then poured in a hollow structural steel shell with two end caps. Finally, the powders were consolidated under HPT and HPDT with an initial pressure of 3 GPa and an anvil rotation rate (ω) of 0.2 rpm at room temperature. With the increase in the cross-sectional area of the sample, the initial pressure decreased due to outflow of the material between the anvils. The value settled to about 1.8 GPa. After reaching the constant pressure, the samples were processed under 2, 4, and 6 turns. It should be noted that, unlike the conventional consolidation methods such as rolling and extrusion, which consists of a pre-compact step prior to the consolidation process, our consolidation processes start from loose powders to fabricate the material. Additionally, samples of Cu ingot were fabricated using HPT and HPDT to 2 turns under the same pressure condition. The ingot samples were of Cu of the same purity as the Cu powders used in making the composites (both are commercially 99.9% pure Cu). The copper ingot samples were taken from an extruded rod and then annealed at 650 °C for 2 h. The samples had a weak initial texture (Fig. 2). Moreover, considering very large strains imposed on the material during HPT and HPDT, any large variation in the initial texture would have minimal influence on texture after HPT or HPDT. Fig. 1c shows samples of MMCs (Cu–20 vol.% SiC) and pure Cu manufactured by HPT and HPDT with various numbers of turns as indicated in the figure. Effect of the material strength MMC vs pure Cu is evident from the shape of the deformed samples. The dimensions of the samples are provided in Table 1.

To investigate the particle distribution in the composite samples, scanning electron microscopy (SEM) was utilized. The characterization was performed in an Amray 3300FE field emission SEM operated at 7 kV in secondary electron (SE) mode imaging. From a detailed examination of the microstructure images, three characteristics of undesirable particle states were identified and classified as follows (1) soft particle clusters (SPCs), (2) rigid particle clusters (RPCs), and (3) free particle zones (FPZs) (see Fig. 3). The FPZs are empty regions without SiC particles which plastic flow can easily happen without any external obstacle. In the SPCs (see Fig. 4), the SiC particles are disconnected from the matrix and since accumulated together appear bigger than the original particle size. The adhesive strength between the SiC particles is weak; therefore, the SPCs can be considered defects that increase the porosity. Fragmentation of these particles is also possible resulting in clusters also bigger than the original particle size. In the RPCs (Fig. 5), the SiC particles are bonded with the matrix from one side and from the other side bonded with adjacent SiC particle and consequently they act as a single large particle which de-clustering of this kind needs to overcome the friction between SiC particle–particle interface. In general, it is assumed that de-clustering in the SPC areas is easier than RPC ones.

The uniformity of the particle distribution in the MMCs was quantified by the quadrat method [24,48], which has been used and proven to be effective in characterizing the homogeneity of particle distributions in MMCs [24]. The method works as follows: an image is divided into a grid of square cells (quadrats) and the number of particles, N_q, in each cell is simply counted. It consists of finding centroids of particles from binary images created based on original SE images. Each particle was carefully isolated from neighboring particles by adjusting appropriate color thresholds to determine its center. The coordinates of the centroids were stored in a matrix. These coordinate were then used to determine the number of centroids per cell. Six SE images per specimen were used at magnifications of 500× from each geometrical position (center, middle, and edge). Each image was divided into 104 quadrats.