Investigation of microstructure and mechanical properties of Cu/ZnO nano composite produced by ARB process

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Abstract. In this study, ARB process was used to produce Cu/Nano ZnO composite and samples were subjected up to six ARB cycles. Microstructural and mechanical properties of the composite within different ARB cycles were investigated by scanning electron microscopy (SEM) and tensile and micro hardness tests. The results showed that increasing the number of cycles, not only helped the distribution of reinforcing Nano-reinforcement in the matrix, but also improved the initial bonding strength, so that at final cycles, structural integration was achieved. Mechanical experiments also showed that increasing the number of ARB cycles, increased yield and ultimate strengths as well as micro hardness. However, elongation decreased up to second cycle and then increased by later final cycles. SEM studies of the fracture surfaces after the tensile test showed that the fracture mechanism of the composite was shear ductile rupture.

Keywords: ARB process, Nano ZnO particles, Mechanical properties, Microstructure.

1. Introduction

The attractive properties that can be obtained with metal matrix composites (MMCs) such as, increase in yield and tensile strengths at room and high temperatures, increase in young’s modulus and reduction of thermal elongation as compared to that of conventional metals and alloys. A main due for production of MMCs based on copper is their electrical and thermal conductivities, that largely depends on getting good characteristic values and increasing wear resistance considerably at the same time [1]. Several methods have been used for producing MMCs such as squeeze or stir casting, powder metallurgy (PM) and roll bonding processes. In this work the rolling bonding process (as a new method) such as accumulative roll bonding (ARB) is used. This process is a kind of severe plastic deformation technique, which can manufacture sheet metals to ultrafine – grained microstructure [2], that developed by Saito et.al [3]. The aim of ARB is produce ultrafine grained and high strength sheet metals without changing specimen dimensions, by impose a severe plastic deformation (SPD) on the material. S.A.Hosseini et.al [4] reported that by ARB process and via producing nano structure pure copper, the copper strips can be strengthened without a dramatic change in their electrical conductivity. So high- conductivity and high- strength pure copper can be obtained. The
structural and mechanical properties of several MMCs have been studied during ARB process including Cu/Al₂O₃[5], Al/SiC[6], Al/SiO₂[7], Al/Al₂O₃[8] and Al/B₄C[9].

In the present paper, Cu/ZnO composite was manufactured via ARB process, afterwards the microstructure evolution and mechanical properties of manufactured composite was investigated at different cycles of ARB for the first time.

2- Experimental procedure

2-1- Materials

The commercial pure copper strips with thickness of 0.8 mm, was used for this research. The copper strips were cut into samples with dimensions of 120mm × 60mm × 0.8mm and then were annealed at 773 K for 1 h. Also 0.5 wt. % ZnO particles (10-30nm) were used as reinforcement.

2-2- Surface preparation

At the first step, the annealed strips were degreased by acetone and then scratch brushed with a rotating stainless steel brush with 0.3 mm wire diameter. Brushing the surface not only causes removal of the oxide layer but also creates a coarse surface, which helps to sinking down into the layers of sheets and make the satisfactory bond between the layers during the ARB process.

2-3- ARB process

ARB process consists of two stages. In the first stage, after surface preparation, distribution of the reinforcement particles were performed between two pieces of copper sheets. Then in order to avoid sliding strips on each other during rolling and achieve a safe product two strips were carefully stacked over each other and finally fastened at both ends by copper wires. The ARB process was done without any lubricant, using a laboratory mill and before each rolling pass. The roll was properly cleaned by acetone. This process was done using a rolling machine with rolling diameter of 120mm. The rolling reduction and rolling speed were 50% and 29 rpm respectively. The rolled strip was cut into two halves and the same procedure repeated once again, thus Cu/0.5wt.%ZnO was produced. In the second stage, in order to achieve a relative uniform distribution of ZnO particles in the copper matrix, the rolled sample obtained from the previous stage was cut in half and the above procedure was repeated up to four passes without dispersing the ZnO particles.

2-4- Investigation of structure and mechanical properties

The microstructure observations were carried out via scanning electron microscopy (SEM) (Philips x130) to evaluate effect of ARB cycles on how well the reinforcement particles were distributed in the manufactured composite. Also, in order to determine the rupture mode, the surfaces after tensile test were observed by SEM. To evaluate changes in strength of the ARB processed sheets were used from uniaxial tensile test and specimen dimensions were chosen according to the ASTM B 557M standard to get oriented along the rolling direction. The tensile tests were performed at room temperature by a KSLAB03 testing machine with a nominal initial strain rate of 8.3 x 10⁻⁴ s⁻¹. The elongation of the samples was determined as the difference between gauge lengths before and after testing. Mechanical properties of the annealed copper strips are shown in table 1.
Table 1: Mechanical properties of annealed copper strips

| Hardness (HV) | Elongation (%) | Tensile strength (Mpa) | Yield strength (Mpa) |
|---------------|----------------|------------------------|----------------------|
| 68.3          | 27.2           | 191                    | 99                   |

3- Results and discussion

Fig. 1 shows the SEM micrographs of the Cu/0.5wt.%ZnO composite manufactured by the ARB process after the 2th and 6th cycles. Fig. 1(a) shows that after second cycles there are some particle agglomerated, clustered and particle free zone in copper matrix. There are several mechanisms of effective to change the lamination uniform distribution of the particles in the matrix. At the initial cycles, during the rolling reinforcement particle layers are separate to small pieces and virgin metal is extruded through the pieces [10]. By increasing the number of cycles, agglomerated ZnO particles began to uniformly dispersed and particle free zones has all but disappeared. Fig. 1(b) indicates that after the sixth cycles a relative homogenous distribution of the reinforcement particles into the copper matrix is obtained for the composite.

![SEM micrographs of Cu/0.5wt.%ZnO composite microstructure at RD-ND plane for (a) 2 and (b) six cycles.](image)

The yield strength, tensile strength and elongation obtained from the engineering stress – strain curves of the Cu/0.5wt.%ZnO composite manufactured by the ARB process (after the six cycles) versus number of cycles are shown in fig. 2. It shows that the yield and tensile strengths of composite increases with increasing strain. Maximum Yield and tensile strengths reach 368 MPa and 390 MPa at the sixth cycle, respectively. It has been reported that in ARB processed materials, strengthening mechanisms during ARB process are governed by strain hardening by dislocations and grain refinement. The main mechanism in the first and second cycles is strain hardening and in the final stages grain refinement plays the main role in strengthening. Furthermore in the MMCs also the reinforcing role of the ZnO particles in the copper matrix is
considered [5,6,8,11]. In fact, the particles are as an obstacle to dislocations movement by preventing its movement, which increases the strength of composite [11]. M.Shaarbaf et al.[12], showed that for pure copper, tensile strength increased from ~ 210 to ~ 430 MPa, after eight cycles, the tensile strength of the ARB processed copper is about two times higher that initial value. Comparison of this conclusion with conclusions that derived from this work demonstrates the reinforcing roll of the ZnO particles in the Cu/ ZnO composite. The elongation decreased up to second cycle and the improved by increasing strain (and then again increased in the next cycles). The reduce in the ductility of the ARB processed Cu/ZnO composite sheets in the initial cycles, can be cause of the extremely strain hardening, insufficient bonding between the copper layers [13]. And also decrease of dislocation mobility and the small numbers of existing shear bands [14]. Increasing the elongation after two cycles is because the, the bonding between the ZnO particles and the Copper matrix becomes stronger because of the greater rolling pressure applied [6]. And the fine dispersion of ZnO particles in Cu matrix [14].

Fig.2. The variations of strength and elongation of Cu/0.5wt.%ZnO composite in different ARB cycles.

Fig.3. shows micro hardness variations of Cu/0.5wt.%ZnO composite as a function of the rolling cycles. Indicated that with the increasing number of ARB process cycles micro hardness was increased. But a drastic increase in the hardness value can be seen at the initial cycles. So that micro hardness value after one cycle was about 1.7 times more than that of the annealed copper. The prompt increase of hardness at the initial cycles is attributed to strain hardening [13]. The micro hardness- increasing rate at the final ARB process cycles is low. It has been reported that this behavior which is caused by increasing number of cycles. Dislocations can be eliminated by annihilation process; so that the density of dislocations decreases at higher cycles in addition dislocation mobility at final cycles is low. Which could also affect the final hardness [15, 16].
Fig. 3. Variation of hardness for Cu/0.5wt.%ZnO composite after different ARB cycles.

Fig. 4. Shows the SEM micrographs of fracture surfaces of the Cu/0.5wt.%ZnO composite after tensile tests. Fig. 4 (a) shows that Cu/ZnO composite illustrated a ductile fracture, having big and deep dimples and shear zones. This kind of fracture occurs by formation and coalescence of micro voids a head of the crack and very limited dislocation activity [17]. Fig. 4 (b) reveals that by increasing strain the number and depth of dimples sharply decrease and the fracture mode of composite changed to cleavage or transgranular brittle fracture. Rezayat et.al [10]. Showed that presences of reinforcement particles have a main role on the fracture surface. Advent of particles in the core of dimples and this simply confirms the beginning of the fracture from interface of particle-matrix.

Fig. 4. The SEM micrographs of fracture surfaces of Cu/0.5wt.%ZnO composite at (a) two cycles and (b) six cycles.
5- Conclusions

1- The SEM microstructures showed that by increasing the number of ARB cycles, the uniformity of ZnO particles in the copper matrix improved.

2- The microhardness and Tensile strength of the composite increased with the increasing number of ARB cycles and reached up to 152 HV and 390 Mpa respectively at the end of sixth cycle.

3- SEM observation of fracture surfaces indicated that failure mode in Cu/ZnO composite manufactured by ARB process in initial cycles is ductile rupture with deep dimples and changed to brittle fracture in the final cycles.

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