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Fabrication of nanostructured Al/Cu/Mn metallic multilayer composites by accumulative roll bonding process and investigation of their mechanical properties

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ABSTRACT

Multilayered Al/Cu/Mn composites were produced from aluminum 1100 strips, commercial copper foils and manganese powders, through accumulative roll bonding (ARB). The structural and microstructural evolution of the produced composites was studied by X-ray diffraction and scanning electron microscopy. Also, their mechanical properties at various ARB cycles were studied by microhardness and tensile tests. In this process after nine ARB cycles, a multilayered Al/Cu/Mn composite with homogeneously distributed, fragmented copper layers and Mn powders in the aluminum matrix was produced. Also, it was observed that with increasing strain by progression of the ARB process, the strength and microhardness of the produced composites increased.

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1. Introduction

Metal matrix composites are known as key engineering materials in industry. Some of them, including aluminum-based metal matrix composites are commonly used in aerospace, automotive and structural industries because of their outstanding properties such as appropriate electrical and high thermal conductivity, low density, high wear resistance and corrosion resistance [1–4]. One type of these materials called metallic multilayer composites (MMCs) consisting of alternative metal, reinforced metal layers and metal powder, dramatically improve a range of mechanical properties [5–8].

Recently, various methods including electro deposition, ion sputtering, evaporation, and magnetron sputtering deposition have been used to fabricate thin metallic film multilayer or multilayered composites [9,10]. The aforementioned processes require expensive tools and complex steps, which have limited their use at industrial scales [11]. Accumulative roll bonding (ARB) has been used as a novel and applicable method of multilayer composite production due to its relatively simple processing and low cost [12]. The ARB process has been successfully applied for production of Ti–Al–Nb [13], Ti–Al [14], Al–Ni [12], and Al–Mg [15] multilayer composites. The evolution of microstructures and related mechanical properties as a function of the ARB cycles at room temperature has been studied for the multilayer strips [16–19]. For instance, Danesh Manesh et al. [20,21] produced Al matrix metallic multilayer composites using the ARB process, and reported that increasing the number of ARB cycles, reinforcement layers start to neck and fracture, leading to the separation and fragmentation of this phase. The ARB process is a severe plastic deformation method to produce nanostructured materials by introducing structural defects such as dislocations inside the material [22–24]. It is the only severe plastic deformation method applicable to produce continuous bulk materials and metallic multi-layer composites [22]. ARB has the following advantages for producing MMCs: The potential to produce composites in the form of sheets [25] and the ability to produce composites with nanocrystalline and ultrafine grained (UFG) structure [23]. The ARB process consists of roll bonding of cleaned and stacked sheets by 50% thickness reduction, cutting them into stacks and again rolls bonding. By repeating this procedure, very high strains have been successfully introduced into the materials, and significant structural refinement has been achieved [22,26]. Tsuji et al. [27] have shown that the repetition of the ARB process, typically above five cycles, can develop pancake shaped or elongated lamellar UFG structures in various metallic materials.

In this study, an innovative manufacturing process for Al/Cu/Mn composite was developed by using aluminum strips and copper foils and micron sized manganese powder as the starting materials. The present work is the first focus on the manufacture of Al–Cu–Mn metallic multi-layer composite by the accumulative roll bonding process. Afterwards, the structural and mechanical properties of the produced multilayer composites at the different passes of the ARB process were investigated.

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2. Experimental procedure

2.1. Materials

1100-Aluminum alloy strips with the thickness of 0.8 mm, commercial copper foils (99.6%) with the thickness of 150 μm, and manganese powders with the average grain size of 60 μm were used as the primary materials. The chemical composition and some mechanical properties of the Al and Cu used in this study have been mentioned in Tables 1 and 2.

2.2. Production of the Al/Cu/Mn multilayer composite

Al strips were first cut with the length of 150 mm and width of 50 mm and annealed at 623 K in ambient atmosphere. Also, the Cu foils were cut with the same dimensions and annealed at 773 K in ambient atmosphere. Al strips and Cu foils were degreased in acetone and scratch-brushed with a 90 mm diameter stainless steel circumferential brush with 0.35 mm wire diameter. To fabricate the Al/Cu/Mn multilayer composites by the ARB process, three Al strips and two Cu foils were stacked over each other, while the Mn powders were dispersed between every two layers. The sequence of the stacked layers was one foil of Cu between the two strips of Al alternatively to produce a multilayer sample with a thickness of 2.7 mm and a composition of 70 wt.% Al–25 wt.% Cu–5 wt.% Mn. The stacked strips were fastened at both ends by steel wires to make it ready for the rolling process. The strip was roll-bonded with a draft percentage of 66% reduction at room temperature. The reduction of 66% was used for the creation of an appropriate bonding between the aluminum strips [28]. At this cycle that was the end of the first step, the number of the layers was 5.

The well roll-bonded strip was cut into two strips by a shearing machine. The strips were degreased in acetone, scratch-brushed and after stacking over each other, without Mn particles between them, roll-bonded with a draft percentage of 50% reduction (Von Mises equivalent strain of 0.8). The last step of the process was repeated up to nine cycles without annealing between each cycle. After nine accumulative roll bonding cycles in total, the Al/Cu/Mn multilayer composites including well dispersed Mn powders were produced.

2.3. Structural evaluation

Microstructural observations were performed using a scanning electron microscope (Philips-FEG). SEM samples after the ARB process were prepared using a shearing machine in parallel to the rolling plane (rolling direction–transverse direction or RD–ND plane).

XRD measurements were carried out on the RD–TD plane of the ARB processed sheets. The XRD experiments were performed by an X-ray diffractometer (XRD, Bruker Advance 2) using Cu Kα_1 radiation (λ = 0.15406 nm). The data was collected at room temperature with a 2θ range between 10° and 90° with a step size and scan rate of 0.03° and 6 s, respectively. The X-ray tube was operated at 40 kV and 40 mA. The Williamson–Hall formula was used to analyze the XRD data. To confirm the Williamson–Hall result, the Rietveld refinement of the XRD patterns was done using MAUD software (by Luca Lutterotti, 1997-2011, University of Trento-Italy) and the required structural information of the individual phases was then extracted.

Table 1
Dimensions and chemical composition of the 1100 Al and Cu alloy.

Materials	Chemical composition (wt%)	Sheet dimensions, (*L*, *W*, *t*) (mm × mm × mm)
Aluminum 1100	99.16 Al, 0.11 Si, 0.55 Fe, 0.11 Cu	150 × 30 × 0.8
Commercial pure copper	99.9 Cu, 0.005 Fe, 0.00 P, 0.002 Zn	150 × 30 × 0.15

2.4. Mechanical properties

Mechanical properties of the composites were studied by micro-hardness and uniaxial tensile tests. Tensile test specimens were machined by a wire cut machine from the rolled sheets according to the 1/5 scale of the JIS-No. 5 specimen, oriented along the rolling direction. The gauge length and width of the tensile test specimens were 10 and 5 mm, respectively and the length of them was 50 mm. The tensile test at ambient temperature was carried out at a nominal strain rate of 8.3×10^{-4} s^{-1} by using an Instron tensile testing machine. To have accurate results, at least five tensile experiments were conducted on each sample and subsequently averaged. The uniform elongation of the specimens was determined as the difference between the gauge lengths before and after testing.

Vickers microhardness (HV) tests, using a load of 15 g for 15 s (according to ASTM: E 384 standard), were performed on the cross-section (TD plane) of the ARB processed samples. The length, width, and thickness of the hardness test samples were 10, 5 and 1 mm, respectively. The mean value of ten separate measurements taken at randomly selected points on the composite was reported.

3. Results and discussion

3.1. Structural and microstructure evaluation

Fig. 1 shows the typical XRD patterns of the pure Al before ARB and the Al/Cu/Mn composite after nine ARB cycles. The crystallite size, as one of the important structural parameters of nano-materials, can be obtained by X-ray diffraction (XRD) technique, as the crystallite size is related to the diffraction peak broadening. Significant peak broadening is observed especially for the smallest crystallite size values [29–31]. Obviously, the (1 1 1) and (2 0 0) peaks are the most suitable for crystallite size determination, because they do not overlap with neighbors. The mentioned peaks are used for the calculation of crystallite size. As it can be seen from Fig. 1, the (1 1 1) peak in the Al/Cu/Mn composite sample is more broad than the same peak in the pure Al. This confirms that the crystallite size of the Al/Cu/Mn sample is finer in comparison with the pure Al sample. The Williamson–Hall method was used to calculate the crystallite size from the XRD patterns of the ARBed Al/Cu/Mn composite after 9 cycles and the pure Al before the ARB process. It should be noted that the XRD measurements were performed on the RD–TD plane. The calculated mean crystallite size of the ARBed Al/Cu/Mn composite was determined 65 nm. In addition to the Williamson–Hall method, the Rietveld's bulk structure refinement analysis of the X-ray diffraction step scan data of the ARBed Al/Cu/Mn composite samples was done to obtain the crystallite size. The Rietveld software MAUD 1.85 is specially designed to refine simultaneously both the structural and microstructural parameters through a least-square method. The mean crystallite size for the Al/Cu/Mn composite and pure Al was 60 nm.

The formation of an ultrafine microstructure during the ARB process can be explained in terms of dislocation generation and rearrangement to form a cell- or subgrain structure followed by grain boundary sliding during further deformation, which is the most probable deformation mechanism in extremely fine nano-crystalline materials [32]. In fact, the formation process of ultrafine grains in highly strained materials can be characterized by both the grain subdivision in submicrometer scale during intense straining

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