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Investigation of nanostructured Al/Al_2O_3 composite produced by accumulative roll bonding process

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ABSTRACT

In this study, the accumulative roll bonding (ARB) process was used for manufacturing nanostructured aluminum/15 vol.% alumina composites. Microstructural characterization by transmission electron microscopy (TEM) identified the severe shear deformation, however, the grain growth was restrained by particles of oxide film and recrystallization produced the nanograins with an average size <100 nm after the 13th cycle of composite strip. The findings also indicated that the presence of large particles and deformation structure in the vicinity of the particles made the particle stimulated nucleation (PSN) of recrystallization possible. The Williamson–Hall method was used to calculate the grain size from the X-ray diffraction (XRD) patterns, which were 150 nm for pure aluminum and 63 nm for aluminum/alumina composite after 13 cycles of the ARB process. The findings also revealed that after the first cycle, hardness rapidly increased, then dwindled, and finally reached saturation as the number of ARB cycles increased.

1. Introduction

A growing interest has been shown in developing metal matrix composites (MMCs) due to their unique mechanical properties such as lightweight and high elastic modulus. Aluminum is a light and relatively weak metal. Its applications are limited when a high-modulus and enhanced strengths are both required. Although high-strength aluminum alloys have been developed, the addition of alloying elements and microstructural control has had a very small effect on enhancing the alloy stiffness. The demands for lightweight, high-modulus, and high-strength materials have led to the development of MMCs [1–3].

Ultra fine grained (UFG) metallic materials where the mean grain size is smaller than 1 μm are expected to exhibit excellent mechanical properties. Accumulative roll bonding (ARB) process, consisting of multiple cycles of rolling, cutting, stacking and solid-state deformation bonding, is one of the effective severe plastic deformation (SPD) processes which can produce bulk UFGed metallic material. The ARB process developed by Saito et al. [4,5] has several advantages over other SPD processes as follows: (1) high load forming facilities and expensive dies are not needed, (2) productivity rate is high, and (3) the amount of material to be produced is not limited. Because the process is continuous, ARB is appropriate for manufacturing nanostructured and ultra fine grained strips, which can be used in commercial and industrial applications [5,6]. The evolution of microstructures and the related mechanical properties during ARB cycles at room temperature were studied for several metal strips such as commercially pure aluminum [5,7,8], Cu [9], Brass [10], and IF steels [4]. However, the researches focusing on the ARBed (ARB-processed) MMCs are very limited [6,11–13]. There are also reports on ECAP deformation of Al/SiC [14] and $Al6061/Al_2O_3$ [15] composites, where hardness and yield stress increase were observed accompanied by considerable grain refinement, however, no information on defect formation or role of particles was given. In addition, structural analysis of the ARBed aluminum/alumina composites like transmission electron microscopy (TEM) investigation and X-ray diffraction (XRD) has not been reported yet.

The objective of this work was to develop the ARB process for manufacturing the nanostructured aluminum/15 vol.% alumina composite strip and investigate the role of processing in microstructural evolution by TEM and XRD. This work also investigates the influence of strain hardening and grain refinement on the hardness of the pure aluminum and composite strips produced by ARB process.

2. Experimental procedure

The materials used in this study were fully annealed strips of commercial purity aluminum alloy (specifications are given in

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Table 1
Specifications of the commercial purity aluminum [11].

Material	Chemical composition (wt.%)	Temperature	Tensile strength (MPa)	Yield strength (MPa)	Elongation (%)	Hardness (HV)
Al 1100	99.11Al, 0.17Si, 0.49Fe, 0.12Cu, 0.02Mn, 0.09 others	As-received	157.4	142.3	7.2	48
		Annealed	84.5	39.3	37.8	19

Table 1) and Al_2O_3 particles (<50 μm). Two strips of 200 mm × 50 mm × 1.5 mm were degreased in acetone bath and scratch brushed with a stainless steel wire brush 0.26 mm in diameter. After surface preparation, Al_2O_3 particles were uniformly dispersed between the two strips which were then stacked over each other and fastened at both ends by copper wires. Attention was also paid to proper alignment of the two strip surfaces prior to rolling. The roll bonding process was carried out with no lubrication, using a laboratory rolling mill with a loading capacity of 20 tons. The roll bonding process was carried out with an amount of reduction equal to 50% (corresponding to a von Mises equivalent strain ε_{vM} of 0.8 per cycle). Then, the roll bonded strips were cut in half. The same procedure was repeated up to 5 cycles at room temperature (the first step). Then, to achieve a uniform distribution of reinforcement particles in the matrix and also to remove porosities in the interfaces of aluminum–aluminum and aluminum–alumina, the above procedure was repeated again up to 13 cycles without adding reinforcement particles (the second step). The schematic illustration of the ARB process for manufacturing of composite is shown in Fig. 1.

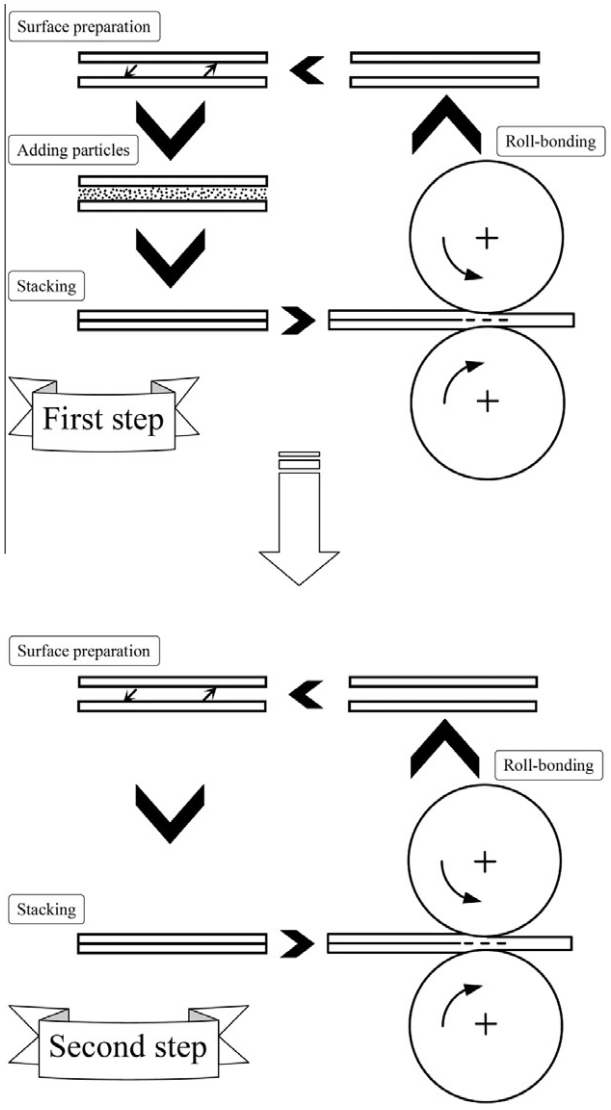

Fig. 1. Schematic illustration showing the principles of the ARB process for manufacturing of composite [11].

The microstructural observations were performed using PHILIPS CM20 transmission electron microscopy and the samples after ARB process were prepared using electrolytical thinning in electrolyte consisting of 1/3 HNO_3 2/3 CH_3OH at subzero temperatures. Thin foils parallel to the rolling plane (RD–TD (rolling direction–transverse direction) plane) were prepared so that the observed area was at about 500 μm below the surface.

The XRD pattern of the ARBed pure aluminum and aluminum/alumina composite was recorded with a diffractometer operating at 40 kV and 30 mA using Cu Kα radiation. The XRD data were recorded in step-scan mode with a step size of 0.050 and a step time of 1 s. The average grain size was calculated from the Williamson–Hall plot. Williamson and Hall [16] expressed integral breadth (β) for all the reflections of a sample in terms of reciprocal unit β^* ($\beta^* = \beta\cos(\theta)/\lambda$), as a function of d^* ($d^* = 2\sin(\theta)/\lambda$) as follows:

$$\frac{\beta\cos\theta}{\lambda} = \frac{1}{D} + 2\varepsilon\left(\frac{2\sin\theta}{\lambda}\right) \quad (1)$$

where β is the difference in the integral breadth between a standard specimen (β_{std}) and the measured sample (β_{obs}), D is the average grain size, λ is the wavelength of X-rays (1.54056 Å for Cu Kα radiation), and ε is the average microstrain. The plot of $\beta\cos(\theta)/\lambda$ vs. $2\sin(\theta)/\lambda$ gives the value of microstrain from the slope and the grain size from the ordinate intersection.

Vickers hardness of the samples was measured in RD–TD plane under a load of 10 kg. Hardness was measured randomly at 10 different points on the strips for each sample, the maximum and minimum results were disregarded, and the mean hardness value was calculated using the remaining eight values.

3. Results and discussion

Fig. 2 demonstrates TEM microstructure and the corresponding selected area diffraction patterns (SAD) of aluminum matrix observed in RD–TD plane of the aluminum/15 vol.% alumina MMC produced by ARB process after the 5th, 7th, 10th, and 13th cycles. According to Fig. 2a, after 5 ARB cycles, bright and dark contrast changes suggest formation of low angle grain boundaries consisting of dislocations visible at subgrain boundaries. It was confirmed in the SAD pattern which shows 111 and 200 diffraction spots elongated in the direction of Debye–Scherrer rings indicating a low misorientation. For this reason, it can be concluded that the grain structure at this stage mainly consisted of subgrains with dislocation cell structures. Fig. 2a, indicates that the distribution is inhomogenous across subgrains. As mentioned earlier in our previous paper [11], during the ARB process, alumina particles present in the aluminum matrix are responsible for an increase of the dislocation density. These dislocations are probably generated at the reinforcement/matrix interface to accommodate strain incompatibility between the two phases. Regarding Fig. 2a, the average size of grains after 5 cycles is ~600 nm. After the 7th cycle (Fig. 2b), in comparison with the previous sample (Fig. 2a), the dislocation