



# High-strength and highly-uniform composite produced by anodizing and accumulative roll bonding processes

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## ABSTRACT

The anodizing and accumulative roll bonding (ARB) processes are used in this paper as a new, effective alternative for manufacturing high-strength and highly-uniform aluminum/alumina composites. Four different thicknesses of alumina layers are grown on the substrate using an anodizing process and the microstructural evolution and mechanical properties of the resulting aluminum/alumina composite are investigated. Microscopic investigations of the composite show a uniform distribution of alumina particles in the matrix. It is found that alumina layers produced by the anodizing process neck, fracture, and depart as the number of accumulative roll bonding passes increases. During ARB, it is observed that as strain increases with the number of passes, the strength and elongation of the produced composites correspondingly increase. Also, by increasing alumina quantity, tensile strength improves so that the tensile strength of the Al/3.55 vol.% Al<sub>2</sub>O<sub>3</sub> composite becomes ~3.5 times greater than that of the annealed aluminum used as raw material.

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## 1. Introduction

Ultra-fine grained (UFG) metallic materials whose mean grain size is smaller than 1 μm are expected to exhibit excellent mechanical properties. Severe plastic deformation (SPD) can be explained as deformation to large strains below recrystallization temperature without intermediate thermal treatments that can result in UFG structures [1]. Several noble techniques have been developed to create high strain in metals with minimal changes in the initial sample dimensions, such as equal channel angular pressing (ECAP) [2,3], high pressure torsion (HPT) [3,4], multi-axial forging (MAF) [5], constrained groove pressing (CGP) [6], and accumulative roll bonding (ARB) [7–9]. Among these processes, the ARB process developed by Saito et al. [7] has several advantages over other SPD processes that include: (1) high load capacity 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. Due to its feasibility as a continuous process, ARB is the only appropriate process for manufacturing nanocrystalline and ultra-fine grained sheets and plates, which are the most widely used in commercial and industrial applications [7]. The evolution of microstructures and the related mechanical properties during ARB cycles at room temperature were studied for several metal strips such as commercial pure Al [7,10], Cu [11,12], Brass [13], Zr [14] and IF steels [10,14].

There has been a wide interest in developing metal matrix composites (MMCs) due to their unique mechanical properties such as lightweight and high elastic modulus. Aluminum is a lightweight and relatively weak metal. Its applications are limited when high-modulus and strength are required. Although high-strength aluminum alloys have been developed, addition of alloying elements and microstructural control can play but only a small role in enhancing their stiffness. The demands for lightweight, high-modulus, and high-strength materials have led to the development of MMCs [15].

Many manufacturing processes have been used for producing such composites. In general, most metal matrix composites are produced by squeeze or stir casting, spray forming, or powder metallurgy techniques. In these methods, reinforcements are incorporated or added into the matrix by ex situ methods. Fine grain sizes, homogeneous reinforcement, and strong bonding of reinforcements with the matrix will certainly improve mechanical properties. The reinforcement particulates used in these methods are usually coarse and rarely below 5 μm. They tend to agglomerate together leading to a non-homogeneous distribution and poor wettability of reinforcement oxides, which badly influences the mechanical and electrical properties of the composites obtained [16]. A number of efforts such as mechanical alloying or rapid solidification have been made to overcome the agglomeration of reinforcements and to obtain dispersed nanoparticles, but they have only led in most cases to contamination, porosity, and poor economical efficiency [17–19].

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In order to overcome the above problems, the present authors have used a combination of anodizing and ARB processes. The technique involves the formation of a thin  $\text{Al}_2\text{O}_3$  layer on commercially pure aluminum strips by anodizing. The bonding between the strip and the  $\text{Al}_2\text{O}_3$  layer is achieved by the ARB process to further break down the thin alumina film and to disperse the alumina particles (platelets) into the aluminum matrix.

A number of studies have investigated the microstructures and mechanical properties of multilayered composite strips or foils produced by the ARB process including Al–Cu [20], Al–Ni [21], Al–Mg [22,23], Al–Mn [24], Al–Ti [25], and Al–Ti–Nb [26]. However, the above studies of ARB produced composites have principally focused on the mechanisms of grain refinement and the effect of strain on microstructural evolution. A few studies [27–29] have also been dedicated to the microstructure and mechanical properties of Al/ $\text{Al}_2\text{O}_3$  composite strips produced by anodizing and only one-pass of roll bonding. The main problem with these investigations is the nonuniformity of the composite due to the application of only one-pass roll bonding.

The present work is, to the best of our knowledge, the first of its kind that focuses on the aluminum/alumina composites produced by anodizing and ARB processes. The aim of the study is to produce high strength MMCs with different volume fractions of  $\text{Al}_2\text{O}_3$  finely and uniformly dispersed in the aluminum matrix by the above processes. The alumina contents used were 0.48, 1.13, 2.40, and 3.55 vol.%.

## 2. Experimental procedure

### 2.1. Materials

As-received commercial purity aluminum sheets were cut into 200 mm × 50 mm × 0.4 mm strips parallel to the sheet rolling direction. Then, the strips were annealed at 643 K for 2 h (specifications given in Table 1).

### 2.2. Anodizing process

Some of the strips were anodized in 15 wt.% sulphuric acid under an applied voltage of 16 V for four different times (1, 5, 30, and 60 min) after being subjected to the annealing treatment in order to produce four oxide film thicknesses. Prior to anodizing, the samples were cleaned in NaOH and then in a  $\text{HNO}_3$  bath. Chemical compositions of the baths are given in Table 2. To ensure a constant and homogeneous temperature throughout the solution, forced convection was provided by electrolyte stirring. The oxide layers were formed at a low electrolyte temperature (16 °C) favoring rapid growth and reduced dissolution of the oxide layer. Then, strips were neutralized in ammonium acetate (Table 2) under an applied voltage of 16 V for 15 min to enhance bonding in the ARB process. The thickness of the alumina layers obtained by the anodizing process was determined by scanning electron microscopy analysis of the oxide cross-sections. Average and standard deviations of about 20 measurements were calculated. The alumina coating thicknesses on anodized Al 1100 for 1, 5, 30 and 60 min were  $2.2 \pm 0.1$ ,  $5.1 \pm 0.2$ ,  $10.8 \pm 0.4$ , and  $16 \pm 0.5$   $\mu\text{m}$ , respectively.

**Table 1**  
Specifications of commercial purity aluminum.

Material	Chemical composition (wt.%)	Condition	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 2**  
Specifications of the baths used.

Bath	Chemical composition
NaOH	60 g/l NaOH + 120 g/l $\text{Al}^{3+}$ + 10 g/l additives
$\text{HNO}_3$	50 wt.% $\text{HNO}_3$ + $\text{H}_2\text{O}$
Ammonium acetate	2 g/l $\text{NH}_4\text{CH}_3\text{COO}$ + $\text{H}_2\text{O}$

### 2.3. Surface preparation

To produce a satisfactory metallurgical bond by the ARB process, it is essential to remove any contaminations that may be present on the surfaces of the metals to be joined [30,31]. These surfaces are composed of oxides, adsorbed ions, greases, moisture, and dust particles. A number of authors have claimed degreasing followed by scratch brushing with a rotating steel brush to be the best method for surface preparation [30,32]. Therefore, the preparation processes for some of the strips (non-anodized) used in this study included degreasing in an acetone bath followed by scratch brushing the surfaces using a stainless steel brush with wires 0.26 mm in diameter. The initial surface roughness of the specimens was 0.5  $\mu\text{m}$ , which, after scratch brushing, rose to about 4.2  $\mu\text{m}$  in the longitudinal and transverse rolling directions. It is important not to touch the cleaned surfaces, because grease or oil on the faying surfaces impairs the formation of a strong joint. To avoid any oxide formation or interference with bonding, the rolling process must be carried out immediately after degreasing and scratch brushing [30,31].

### 2.4. Accumulative roll bonding (ARB) process

The schematic illustration of the ARB process is shown in Fig. 1. This technique included two steps. In the first step, the two annealed strips were surface prepared, and then the anodized strip was laid between the prepared surfaces of strips. The strips were stacked over each other, were fastened at both ends and roll-bonded to 60% reduction. The roll-bonded strips were then cut in half. The second step was designed to yield a uniform distribution of reinforcement particles in the matrix and also to remove porosities from the interfaces between the layers. In this step, the procedure in the first step was repeated up to eight cycles with a specific percentage of reduction equal to 50% in each cycle. The amount of particles and the number of cycles can be manipulated to obtain an ideal strength and elongation. Generally, the time between surface preparation and rolling was kept to less than 120 s. Care was taken to properly align the strip surfaces prior to rolling. The ARB experiments were carried out with no lubrication, using a laboratory rolling mill, with a loading capacity of 20 tons.

### 2.5. Microstructure evaluation

The microstructures of the ARB processed composite strips with various numbers of cycles were evaluated by optical microscopy (OM) and scanning electron microscopy (SEM) (PHILIPS XL30). To evaluate alumina distribution in the matrix and the bonding condition of the samples, optical examination of the strips was con-

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