Manufacturing of high-strength multilayered composite by accumulative roll bonding

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Abstract
In this study, high strength Al/Mg composite was produced using Al 5052 and Mg AZ31B by applying three passes of the accumulative roll bonding (ARB) process at room temperature. For this purpose, microstructure, plastic instability, and mechanical properties were assessed by optical microscopy, x-ray diffraction (XRD), uniaxial tensile test, and Vickers microhardness. The microstructure showed that by rising the exerted strain, grains were drawn in the rolling direction and grain refinement in Mg layers occurred. The crystallite size for Al after first and second ARB passes reached 192 nm and 173 nm, respectively. The lamellar structure was also maintained after the second pass; however, necking, fracture, and waveform formation in Mg layers were quite visible. The results of mechanical properties showed that by increasing the number of ARB passes, the values of UTS and microhardness for both Al and Mg layers increased sharply at the zero pass and gradually at first and second ARB passes, respectively. Also, the maximum UTS reached 475 MPa, which was at least a 100% improvement compared to previous researches. Variation of microstructure and mechanical properties during ARB process are related to many factors such as severe plastic deformation, locking of dislocations, continuous recrystallization, lamellar structure and two mechanisms of work hardening and grain refinement at initial and last ARB cycles play a central role.

1. Introduction
The accumulative roll bonding (ARB) process is one of the severe plastic deformation (SPD) methods that can produce ultrafine-grained multilayered composites with desirable mechanical properties [1]. The ARB process, like other forms of SPD, enables it to apply immense strain without altering the sample geometry [2]. The advantage of the ARB method over other forms of SPD is that it is inexpensive, and does not require expensive and cumbersome molds and equipment. It is also capable of industrialization due to its continuous use [3, 4]. The successful fabrication and production of numerous ultrafine-grained and Nano-structure of various metals, alloys, and composites by ARB have so far been reported. According to previous researches, the mechanical and microstructural properties of the specimens and composite produced by ARB method have been improved that the most important of them are Al/Mg [5, 6], Al/Cu [7–9], Al/Ni [10, 11], Al/Ti [12, 13] and Al/Ti/Mg [14] and Al/Cu/Mg [1, 15, 16] composites. So far little research has been done on the fabrication of Mg composite samples, and most of them have been at high temperatures. Magnesium and its alloys are widely used today in the automotive, aerospace, and industry structures [17, 18]. The reason for the attention of Mg alloys is its excellent strength, low density, good mechanical properties, high energy absorption, excellent machining, and good thermal conductivity [17–19]. Magnesium and aluminum are the lightest functional metals. Magnesium has a density of about 1700 kg m⁻³, and aluminum has a density of about 2700 kg m⁻³. Aluminum also has more strength and formability than magnesium and can enhance magnesium ductility properties at ambient temperature. In recent years, the use of lightweight aluminum and magnesium in the transportation of vehicles...
such as aircraft, ships, and cars due to low density has increased sharply, and for this reason, their use together creates the need for dissimilar connection.

On the other hand, because magnesium has an HCP structure, and its slides are confined to room temperature, its formability is limited. Creating a uniform structure with small grain size has been used by researchers to overcome this problem [17, 18, 20, 21]. To this end, processes such as ARB (e.g., severe deformation) have been considered. Following is a brief explanation of previous research on Al/Mg composites using the ARB process. Chen et al for the first time used warm ARB process to produce Al/Mg and characterize the microstructure and mechanical properties [22]. They used Al 1100 and Mg AZ31 as raw materials and applied the ARB process at 300 °C for three cycles. They reported grain size for Al and Mg layers 875 and 656 nm, respectively [22]. Also, they concluded by rising the applied strain, bonding between layers and microhardness for both layers improved. In another similar study, in 2010 by Wu et al, Al 5052 and pure Mg were used to fabricate Al/Mg composite via ARB method at 400 °C in three passes [6]. They reported that due to the high process temperature, grain refinement was not observed and also because of a crack in the intermetallic compound, the tensile strength in the third pass decreased [6]. In 2012, Chang et al developed Al/Mg composite using Al 1050 and pure Mg at ambient temperature in three ARB cycles [23]. Their results indicated that the grains refined in Mg layers at the end of the zero pass. Then, by raising the number of ARB passes, they uniformed and homogenized [23]. Also, experimental results of the tensile test in both rolling and transverse directions showed that due to the significant anisotropy at ambient temperature, the tensile strength at RD was lower than TD and strength changing in terms of ARB passes were also minor [23]. In summary, it can be concluded that ARB process at ambient temperature can achieve better mechanical properties and microstructure with smaller and more homogeneous grain size and on the other hand, the high-temperature process results in the formation of intermetallic, reducing the strength and not improving the structure. Also, in both situations, the mechanical properties of the Al/Mg composite produced are noticeable in the first cycle and are negligible in subsequent passes.

In this study, in order to produce the Al/Mg composite with high mechanical properties, Al 5052 and Mg AZ31B were used as raw materials. Then, three passes of the ARB process at ambient temperature were applied. Mechanical properties and microstructure of produced composite during the ARB process were analyzed by uniaxial tensile test, microhardness measurements, XRD, and optical microscopy. Finally, the mechanical properties were compared with similar values in previous studies.

2. Experimental procedure

2.1. Materials

Materials employed in this study include 5052 Al alloy and AZ31B Mg alloy. Table 1 demonstrates chemical composition, mechanical properties, and the primary dimension of these materials.

2.2. ARB process

Multilayered Al/Mg composite was prepared at two steps, as shown in figure 1. At first, a zero pass was fabricated, and then the accumulative roll bonding process was carried out. To fabricate the zero pass, at first, two 5052 Al alloy sheets, and Mg AZ31B sheet were prepared at same dimensions. Then, these three sheets were degreased in acetone, dried in the air, and were scratch brushed and stacked on each other. Then, the sheets were assembled as a sandwich stack included 5052 Al alloy layers as the outer surfaces and one Mg AZ31B layers as the inner one. The stack was clamped through steel wires at four edges to prevent slippage. Finally, to fabricate zero pass, a 60% reduction in thickness was applied so that the thickness of the sandwich stack reaches 1.5 mm from 3.5 mm by using rolling at room temperature. After fabrication of the zero pass, the samples were cut into half and then surface treatment, including degreasing with acetone, drying in the air, and roughing with a steel brush was applied. Finally, the roll-bonded sheets with a 50% reduction in thickness value were created after clamping. The ARB process repeated up to two cycles. The process was performed using a laboratory rolling mill with 107 mm in roller diameters at room temperature in which no lubricant was used.

2.3. Microstructure and mechanical properties

The microstructures of processed specimens were evaluated by optical microscopy (OM). Specimens after each pass were cut using wire electro-discharge machining in the parallel to the rolling direction. Then, they were grinding using sandpaper numbers 180 to 4000 and polishing by use of alumina powder with a size of 1 μm and 3 μm. Then, to examine the microstructure of the magnesium layer, the samples were etched in HCL solution for 60 s. The phases formed in the produced samples were additionally calculated by crystallite size and lattice strain by Williamson-Hall method using 2-theta diffraction patterns in the range of 20°—90°. The phases mentioned in this study were determined by Xpert high score software.
Table 1. Specifications of primary materials.

| Material       | Chemical composition (%)                        | Ultimate tensile strength (MPa) | Microhardness (HV) | Primary dimension (mm) |
|----------------|-----------------------------------------------|--------------------------------|--------------------|------------------------|
| 5052 aluminum alloy | Al bal., Mg 2.2, Fe 0.4, Cr 0.2, Si 0.2, Mn 0.1, Zn 0.1, Cu 0.1 | 60.4                           |                    | 120 × 50 × 1.5         |
| MgAZ31B        | Mg 95.8, Al 3, Zn 1, Mn 0.2                    | 170.35                         | 55.2               | 120 × 50 × 1.5         |
Mechanical properties of multilayered Al/Mg composite fabricated by the ARB process were studied through uniaxial tensile tests and microhardness measurements. The uniaxial tensile test samples were prepared for the unprocessed and ARB processed sheets oriented along the rolling direction according to the ASTM E8/E8M-9 standard. The gauge length and width of the tensile test specimens were 6.92 and 2.5 mm, respectively. The uniaxial tensile tests were performed at a nominal initial strain rate of $1 \times 10^{-4}$ s$^{-1}$ at room temperature using SANTAM tensile testing machine. The Vickers microhardness tests were done for initial, and ARBed samples using JENUS apparatus under a load of 200 gr applied for 10 s. Microhardness tests had been implemented to both aluminum and magnesium layers at ten different points randomly on the cross-sections perpendicular to the rolling direction. Then, for each magnesium and aluminum layers, the minimum and maximum hardness were disregarded.

3. Results and discussion

3.1. Plastic instability and microstructure

The structure and plastic instability changes of the multilayered Al/Mg composites processed by ARB are shown in figure 2. As can be seen, the continuity of the layers during ARB cycles is preserved due to the high thickness of Mg over Al layers. It is also observed that with increasing applied strain on the Mg layer, plastic instabilities (fractures and necking) have occurred which are shown by oval and square intersections in figure 2(c). The deformation of the constituent layers is uniform in the lower cycles, while the deformation of the layers converted to the waveform with increasing the number of ARB cycles. In general, during the plastic deformation of the dissimilar metals simultaneously, plastic instabilities are caused by differences in the mechanical properties of the matrix and reinforcement layers (Mg and Al layers) [24, 25]. Plastic instability is influenced by the ratio of initial thickness, strength coefficients, and strain hardening constants of the layers [26, 27]. Layer breaks occur due to differences in mechanical properties with the mechanism of the joining the micro-cracks due to the crack and activation energy of the very limited dislocations. Based on previous research reported that necking for hard phase has occurred in the Al/Cu/Ni/ SiC in which cold rolling causes necking of both Ni and Cu layers and in Cu/Zn/Al causing necking of Cu [27, 28]. These studies show that the development of hard phase necking coincides with the shear failure of the soft layer by shear bands created at the Al/Cu interfaces at
an angle of 45° in the rolling direction and this shear failure, in turn, results in the failure of the hard phase layers [14, 29, 30]. It should be noted that by rising the volume fraction of the hard phase, the necking and fracture rate decreases. This result is in line with the findings of Lee et al., who showed that by increasing the hard-to-soft phase ratio, the higher the critical strain for necking in multi-layered composites are needed [29]. It is worth noting that due to the differences in the properties of the layers, we need more passes for uniform distribution of Mg in the Al matrix. However, it was limited to two passes of the ARB process to obtain the optimum mechanical properties and save time and money.

Figure 3 shows the microstructure of the Mg layer in the processed composites during the various passes of the ARB process. It is observed that due to the cumulative strain during the ARB process and the high density of dislocations, shear bands have emerged. In the zero pass, grains drawn in the rolling direction are also seen. As the applied strain (number of ARB cycles) increases and the density of the dislocations rises, the complexity of dislocations causes dislocation cells to form and eventually decreases grain size in the second pass compared to zero pass. However, more strain is needed to homogenize magnesium grain size.

The x-ray diffraction pattern of the samples processed at different passes of the ARB process is shown in figure 4. Al and Mg peaks were observed at both passes, and no new peak was generated due to sample processing at room temperature. The flattening of the main Al peak in the second pass relative to the first pass indicates the formation of fine crystallites and a high density of defects by the accumulative strain of the ARB process. The crystallite size of the samples processed at different passes was calculated by the Williamson Hall method for at least three peaks using equation (1).

\[ \beta = \frac{1}{d} \sqrt{\varepsilon} \]

Where \( \beta \) is the width at half maximum peak in radians, \( \theta \) the diffraction angle, \( \lambda \) the x-ray wavelength (equivalent to 1.5406 angstroms), \( d \) the crystallite size, and \( \varepsilon \) the strain of the network. \( \beta \) can be calculated...
according to equation (2). $\beta_{abs}$ is Peak width at half maximum intensity and $\beta_{inst}$ is Peak width caused by device error.

$$\beta_x \cos \theta = \frac{0.9 \lambda}{d} + 2\varepsilon \sin \theta$$  \hspace{1cm} (1)$$

$$\beta_i^2 = \beta_{abs}^2 - \beta_{inst}^2$$  \hspace{1cm} (2)$$

The crystallite size of the aluminum substrate was obtained using the Williamson Hall method in the first and second passes, respectively, 192 and 173 nm, respectively. According to research, several mechanisms of fine-graining in Al/Mg composites processed by ARB process have been reported.

The mechanism of formation of the microstructure during the ARB process is an important issue. 4 Possible mechanisms are suggested as follows:

(1) Continuous recrystallization: recent experiments have suggested that the mechanism of formation of Nano and ultrafine grains in heavily deformed metals is continuous recrystallization, which occurs by splitting the ultrafine grains, creating boundaries with short-grain boundary retrieval and migration [31, 32].

(2) Severe shear deformation: this is the result of friction between the sheet and the rollers under oil-free conditions. This shear deformation dramatically increases the equivalent strain and promotes grain refinement [27, 33].

(3) Locking of dislocations by oxide particles created in joints or particles added as reinforcements: by performing the ARB process, creating an appropriate distribution of oxide particles that can be a factor in preventing dislocation motion and increasing the density of dislocations [28].

(4) Formation of lamellar Structure: after several passes of the ARB process, the thickness of the Mg and Al layer decreases. According to previous researches, the interlayer of these layers acts as a substantial barrier to dislocation movement and results in dislocation accumulation [25, 28, 34].

3.2. Mechanical properties

The engineering stress-strain curves of the initial sheets and multilayered Al/Mg composite in different passes of the ARB process with are shown in figure 5. As can be seen, the tensile strength of processed composite higher than initial Al and Mg sheets. Ultimate tensile strength (UTS) and elongation values were also extracted from the stress-strain diagrams and are presented in figure 6. According to figure 6, the tensile strength and elongation of the processed composites increased steadily and reached their highest value in the second pass. The tensile strength in the second pass is 475 MPa, which is 1.8 and 3 times higher than the aluminum and primary
magnesium sheets, respectively. The reason for the increase in strength can be attributed to the decrease in grain size (in accordance with the obtained crystallite size), sample processing at room temperature, the continuity of Mg reinforcement layers in Al matrix (figure 2) and the strain hardening due to the nature of the ARB process [24, 35–38]. In other words, in the ARB process, the increase in the strength of the multi-layered composites can be attributed to the strain hardening or dislocation strength, grain size improvement and grain boundary strength [24, 28, 39]. The strain hardening is due to the increased dislocation density caused by cold work. This factor plays an essential role in the early stages of the ARB process, which by increasing the ARB passes and creating fine grain structure and improving the grain size gradually its effect decreases in subsequent passes and finalization of the fine grains in the final stages of the process [3, 40, 41]. In the ultrafine structure, the creation of new boundaries concerning the Hall Patch relationship will strengthen. Also, the shear strain effect created during the ARB process due to friction between the layers themselves increases the equivalent strain and increases the strength [24, 28]. Other factors can be related to residual stresses by affecting the dislocation motion and the number of layers and their thickness. In the final stages, the number of layers’ increases, which can be a factor in increasing strength.

The Vickers microhardness changes for Al and Mg layers according to different cycles of the ARB process are presented in figure 7. It is observed that by increasing the ARB process counts, the microhardness is increased for both Al and Mg layers continuously so that in the first cycle, this happens at an increasing rate, and as the passes pass, the rate decreases. At the end of the primary sandwich (zero pass), the microhardness values for Al and Mg reached to 91.6 and 76.1 VHN, respectively, which improved by more than 1.55 and 1.37 compared to the prototypes. Also, the maximum values at the last ARB passes are achieved. The leading cause of the increase in
The microhardness of layers during the ARB process is the cold work, and the decrease in the increasing rate is also related to the reduction of its effect at higher applied strain [2, 9, 40, 41].

Table 2 shows a comparison between the mechanical properties of the present study with that of other composite studies involving Mg and Al layers processed using the ARB process. As can be seen, due to the weak bonding strength in the presence of Mg or Al layers, all the studies have been carried out in the initial preheating state to better bonding. However, the present study was carried out at room temperature, and this has led to a higher dislocation density and improved mechanical properties. It should be noted that in studies involving Al and Mg layers the maximum strength obtained in the second ARB pass is obtained and the continuation of the ARB process has led to the separation of Mg layers and a decrease in tensile strength. Therefore, the purpose of the present work was to obtain maximum strength and not extend until the second pass. Also, the comparison of the length of the present study with other studies showed that the bonding strength between Al and Mg layers in the present study is comparable to other studies. The maximum strength in the same applied strain obtained in the present study was 1.82, 2.11, and 2.45 times higher than [6], [23] and [42], respectively. This represents a highly robust composite process due to room temperature processing and the use of Al 5052.

4. Conclusion

This paper focuses on achieving the highest tensile strength for multilayered Al/Mg composites. For this purpose, Al 5052 and Mg AZ31B alloys and two passes of ARB process at room temperature were used to produce Al/Mg composite, and the microstructure and mechanical properties were studied experimentally, and the following results were obtained:

![Figure 7. The variation of microhardness values for Al and Mg layers in multilayered Al/Mg composite in different passes of the ARB process.](image-url)
(1) By applying high strain during rolling, proper bonding between the Al/Mg layers was achieved, grains were drawn in the rolling direction at ambient temperature, and the lamellar structure was maintained, however necking may occur. The fracture was shown at the end of the second ARB process. Also, as the applied strain increases and the density of the dislocations rises, the complexity of dislocations causes dislocation cells to form and eventually decreases grain size in the second pass compared to the zero pass. Also, XRD analysis showed that the crystallite size for Al was 192 and 173 nm in the first and second ARB passes, respectively.

(2) The results of the tensile test showed a significant improvement in the zero pass and a gradual improvement in the subsequent passes. At the end of the second cycle, the tensile strength reached 475 MPa, which is 1.8 and 3 times higher than the Al and Mg sheets, respectively.

(3) The reason for the increase in strength can be attributed to the decrease in grain size, sample processing at room temperature, the continuity of Mg reinforcement layers in Al matrix and the strain hardening due to the nature of the ARB process.

(4) The maximum strength in the same applied strain obtained in the present study was 1.82, 2.11, and 2.45 times higher than similar researches, respectively. This represents a highly robust composite process due to room temperature processing and the use of Al 5052.

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