Advances in Materials Research, *Vol. 9, No. 2 (2020) 99-107*
DOI: https://doi.org/10.12989/amr.2020.9.2.099

Effect of Tio_2 particles on the mechanical, bonding properties and microstructural evolution of AA1060/TiO_2 composites fabricated by WARB

Mohamad Heydari Vini*[1] and Saeed Daneshmand [2a]

[1] *Department of Mechanical Engineering, Mobarakeh Branch, Islamic Azad University, Mobarakeh, Isfahan, Iran*
[2] *Department of Mechanical Engineering, Majlesi Branch, Islamic Azad University, Isfahan, Iran*

(*Received September 17, 2019, Revised January 17, 2020, Accepted March 26, 2020*)

Abstract. Reinforced aluminum alloy base composites have become increasingly popular for engineering applications, since they usually possess several desirable properties. Recently, Warm Accumulative Roll Bonding (WARB) process has been used as a new novel process to fabricate particle reinforced metal matrix composites. In the present study, TiO_2 particles are used as reinforcement in aluminum metal matrix composites fabricated through warm accumulative roll bonding process. Firstly, the raw aluminum alloy 1060 strips with TiO_2 as reinforcement particle were roll bonded to four accumulative rolling cycles by preheating for 5 min at 300°C before each cycle. The mechanical and bonding properties of composites have been studied versus different volume contents of TiO_2 particles by tensile test, peeling test and vickers micro-hardness test. Moreover, the fracture surface and peeling surface of samples after the tensile test and peeling test have been studied versus different amount of TiO_2 volume contents by scanning electron microscopy. The results indicated that the strength and the average vickers micro-hardness of composites improved by increasing the volume content of TiO_2 particles and the amount of their elongation and bonding strength decreased significantly.

Keywords: metal-matrix composites; mechanical properties; TiO_2 particles; bonding strength

1. Introduction

The particle reinforced aluminum matrix composites (AMMCs) have excellent properties such as increased strength, light weight, high corrosion, wear resistant, higher service temperature, high specific modulus and thermal stability (Kaczmar *et al*. 2000, Chin 1999, Baazamat *et al*. 2015) as compared to the conventional pure metals and alloys. Theses composites have the ductility and toughness as metallic properties of metals and strength and modulus of ceramics. So, because of their mentioned specific properties, AMMCs are commonly used in automotive, aerospace and structural industries (Kaczmar *et al*. 2000, Chin 1999). TiO_2 particles are one of the most commonly used reinforcements in matrix composites and addition of this reinforcement to aluminum alloys has been the subject of several researches (Chin 1999). During the recent years, SPD techniques have

*Corresponding author, Ph.D., Assistant Professor, E-mail: m.heydari@mau.ac.ir
[a] Ph.D., Associate Professor, E-mail: S.daneshmand@iaumajlesi.ac.ir

http://www.techno-press.org/?journal=amr&subpage=5 ISSN: 2234-0912 (Print), 2234-179X (Online)

been proposed for deforming metals to very high plastic strains, with the aim of producing bulk submicron-grained materials such as equal channel angular extrusion (ECAE) (Kaczmar *et al*. 2000), cyclic extrusion compression (CEC) (Jamaati *et al*. 2011), severe plastic torsion straining (SPTS) (Mandal *et al*. 2007), multi-axis forging (MAF) and accumulative roll-bonding (ARB) (Sedighi *et al*. 2016a, b). Saito *et al*. (1999) invented a new nobel process, named accumulative roll bonding (ARB), to achieve ultra high strains in metallic materials without changing the specimen dimensions. The ARB technique is an SPD procedure to get large production quantities of strip metals. Usually, the ARB process consists of roll bonding of cleaned and stacked strips by 50% thickness reduction, cutting them into stacks and then roll bonding again (Sedighi *et al*. 2016a, b). The aim of this study is the production of AA1060/TiO_2 composites via ARB process at 300°C. Finally, mechanical, bonding properties and fracture surface of MMCs fabricated by ARB are compared versus different amounts of TiO_2 particle volume percent.

2. Experimental procedure

Aluminium alloy 1060 strips with the length of 200×50×1 mm annealed at 400°C in the ambient atmosphere. The chemical composition of 1060 alloy is presented in table 1. TiO_2 particles with the average size of 1 micrometer were used as raw materials. The ARB experiments were carried out using a laboratory rolling mill without a lubricant. A rolling machine with 170 mm diameter, 36 RPM of rotational speed and a power capacity of 35hp is used, in this study. To start the ARB process, the strips were degreased in acetone bath and scratch brushed with a 90 mm diameter stainless steel circumferential brush. To fabricate the reinforced composites by the ARB process, TiO_2 particles were dispersed between each two of the layers by 0, 1, 5 and 10% volume content in 4 samples and the two strips were stacked together to achieve 2 mm thickness. Both ends of the stacked strips were fastened by steel wire to make it ready for the accumulative roll bonding process. Then, the strips were roll-bonded by 75% reduction at 400°C to achieve o.5 mm thickness. We call this composite as “primary composite” and this cycle as “cycle # 0 or zero cycle”, in this study. So, the thickness reduces to 1 mm (abigureout 75% reduction in thickness), Fig. 1(a). To remove the effect of work hardening in the zero cycle, the sample annealed at 400°C in the ambient atmosphere again and. In the next step, The Warm-ARB process repeated up to four cycles on each of the 4 sample again and for the last time without annealing between each of two cycles with 50% reduction in thickness at 300°C, Fig. 1(b). After the four ARB cycles in total, the AA1060 matrix composites containing the well-dispersed TiO_2 reinforcements were fabricated. Finally, in all samples, the number of the powder layers after the 4th cycle, is 32 within the composite layers. The steps of the production process of the Al/TiO_2 composites have been summarized in Table 2. ASTM-E8M is used to prepare the tensile test specimens were machined along the longitudinal direction of the rolled samples. Tensile tests were conducted at the ambient temperature on a Hounds field H50KS testing machine at a strain rate of 1.67×10^{-4} /sec. For each sample, the tensile test was repeated 3 times.

Table 1 Chemical composition of aluminum alloy1060

Element	Al	Si	Fe	Mg	Zn	Ti	Cu
Wt%	balance	0.25	0.03	0.03	0.05	0.03	0.05

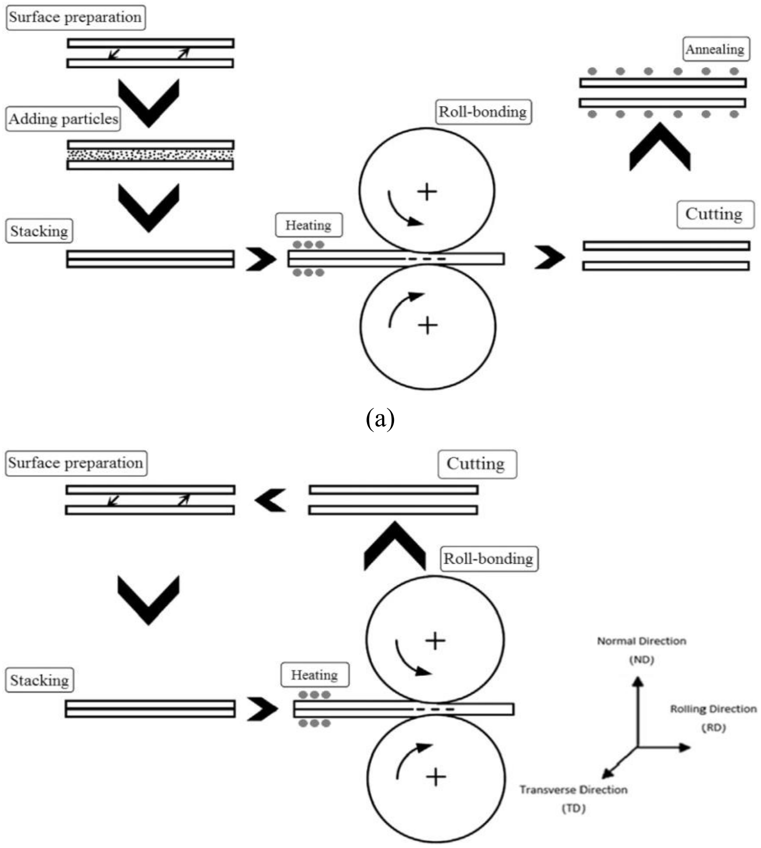

Fig. 1 Schematic illustration of the production process of Al/TiO$_2$ composite sheets (a) primary cycle (cycle#0) and (b) main cycles

Table 2 Specifications of the WARB process for production of Al/TiO$_2$ composite

No. of cycles	Rolling temperature (°C)	No. of Al-layers	No. of TiO$_2$ layers	Reduction in each cycle (%)	The Al-layers thickness (μm)	Total reduction (%)	Effective strain (ε_{ef})
0	400	2	1	75	500	75*	1.6*
1	300	4	2	50	250	50	0.8
2	300	8	4	50	125	75	1.6
3	300	16	8	50	62.5	87.5	2.4
4	300	32	16	50	31.25	93.75	3.2

*After the zero cycle, the sample is annealed at 400°C for 1 hour; therefore, the strain exerted in the zero cycle would be removed.

The Vickers hardness test was done by standard ASTM-E384 under the load of 500 gr force in 15s on each composite. Also to measure the the average Vickers microhardness, the hardness test

Fig. 2 Schematic illustration of the peeling test fixture

was done on more than five points and the average amount was reported (Eizadjou *et al*. 2008). Finally, using an Instron tensile testing machine with 100 kg load cell, the bond strength of the Al/TiO_2 composites was measured. The mean peeling force was measured by a clamping configuration shown in Fig. 2. The speed of the crosshead in the peeling test was 20 mm/min. Note that the average peel strength is taken as

$$\text{Average peel strength} = \frac{\text{Average load}}{\text{Bond width}} \tag{1}$$

3. Results and discussion

3.1 Tensile strength and average vickers microhardness

Tensile tests have been carried out for MMC samples with different TiO_2 contents fabricated after 4 cycles, (Fig. 3). By increasing the plastic strain, the flow stress rapidly reaches to its maximum value. According to Fig. 3, increasing the TiO_2 volume percent in the MMCs leads to the enhancement of the engineering stress–strain curve of samples. As seen in Fig. 4, the tensile strength of the ARBed MMCs is 142.85 MPa and 181.27 MPa for samples produced with 1% and 10% of TiO_2 particles which are 1.1 and 1.5 times higher than those obtained for the monolithic sample, respectively. Strain hardening around the TiO_2 particles during the rolling process activites the slip systems which generate additional dislocations around the particles and decreases the mobility of dislocations (Ng, Li, Fan, Gao, Smith, Ehmann, Cao, 2015). Also, because the ARB done in this study is a warm forming process, there is grain refinement during all the ARB cycles. Both of these effects lead to increasing in the strength and decreasing in the ductility of samples which decrease the bonding strength. According to Fig. 4, by increasing the TiO_2 content, the elongation of samples decreases considerably which attributed to the weak interface of TiO_2 clusters with the AA1060 matrix. TiO_2 particles layer include porosities which affect the reduction of the elongation. The harder TiO_2 particles impede the motion of Al grains which increases the dislocation density in the matrix near the matrix-reinforcement interfaces (Emadinia *et al*. 2016).

All of these effects enhance the strength and reduce the elongation of the AA1060/TiO_2 composites according to Fig. 4 due to higher volume fraction of particles which leads to more strain hardening around the particles. So, the tensile strength increases while ductility decreases. As seen in Fig. 4, the ductility of the ARBed MMCs is 3.71% (1% TiO_2) and 1.79% (10% TiO_2) which are 0.93 and 0.45 times higher than those obtained for the monolithic sample.

As mentioned before, work hardening and reduction of dislocation mobility leads to creation of this behavior. TiO_2 particles layer includes porosities which reduces the amount of elongation. So, the mechanisms that enhance the elongation during the ARB are (I) increasing the uniformity of TiO_2 particles inside the aluminum matrix, (II) increasing the bond strength between the Al matrix and TiO_2 particles which decreases the voids among them, (III) decreasing the porosities in the clusters and dispersing them inside the metal matrix. According to Fig. 5, the toughness value of the composites decreases considerably from 4.28 j.m^{-3}×10^{4} (monolithic sample) to 2.73 j.m^{-3} ×10^{4}(10% TiO_2) which is due to the stress hardening and the less mobility of dislocations. TiO_2 particles are the barriers against the movement of dislocations. So, increasing the amount of TiO_2 particles in the AA1060 matrix decreases the elongation severely which reduces the toughness amplitude.

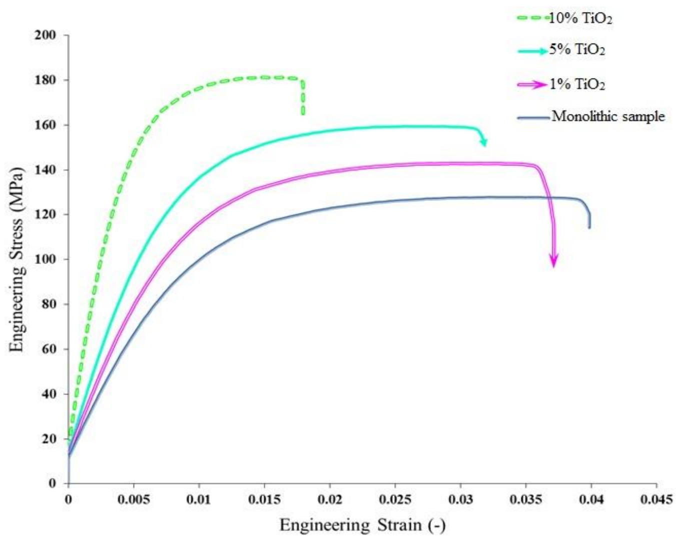

Fig. 3 Engineering stress-strain tensile test curves for AA1060/TiO_2 composites

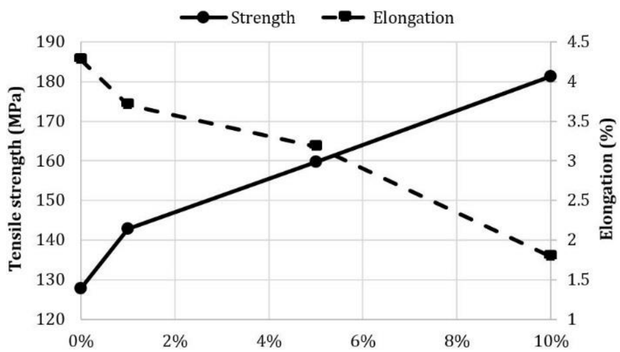

Fig. 4 Tensile strength and elongation of the Al/TiO_2 composites with the TiO_2 volume contents

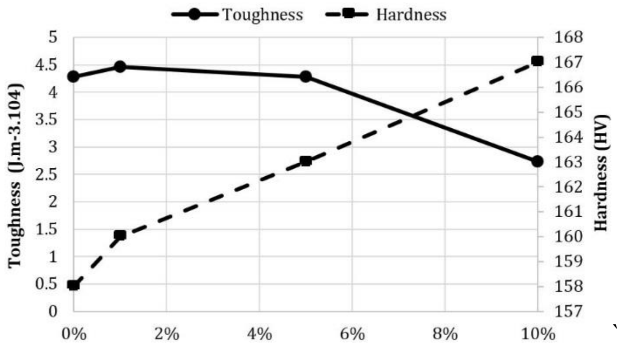

Fig. 5 Variations of toughness and hardness of composite samples with the TiO_2 volume percentages

The hardness values of the samples after ARB increase with the content of particles, (Fig. 5). As mentioned before, TiO_2 particles are the barriers in the way of dislocations. According to Fig. 5, average Vickers hardness amount increases from 158 up to 167 that is about 5.7% improve for samples produced with the content volume fraction of the hard phase TiO_2 particles from 0% up to 10%, respectively. During the forming process, the first reason for increasing of micro hardness seems to be related to the initial reduction of the grain size and growth of dislocation density inside the crystalline lattice which leads to initial strain hardening (Jamaati *et al*. 2011). By increasing the ARB cycles up to four cycles, the materials reach to a certain steady state density of dislocations and the distribution of particles in the matrix becomes more and more uniform. In the next stage, the mechanism which increases the hardness is local strain hardening around the particles. So, the higher amont of TiO_2 particles inside the AA1060 matrix leads to more local strain hardening and as a result, the average Vickers hardness improves considerably.

3.2 Fractography

To clarify the rupture mechanisms in the AA1060/1vol% TiO_2, and Al/10vol% TiO_2 composites, an SEM study is conducted. Fracture surfaces after the tensile test are shown in Fig. 6. The dimples of the AA1060/1% TiO_2 sample (Fig. 6 (a)) are very deep, which clearly exhibit a typical ductile fracture. Also, Fig. 6(a) reveals that the AA1060/1% TiO_2 composites exhibit a typical

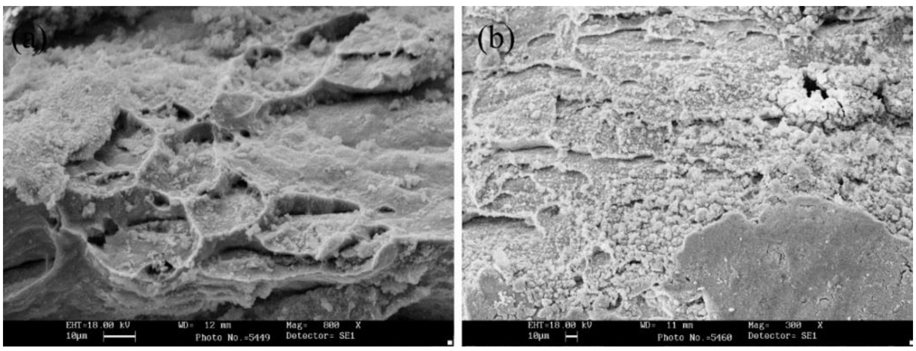

Fig. 6 Fracture surfaces after tensile test: (a) Al/1 vol%; and (b) Al/10 vol% TiO_2

ductile fracture with dimples and shear zones. Fig. 6(a) reveals a fracture model that occurs through the formation and coalescence of micro voids crossing the entire sample. The depth of the dimple in Al/10 vol% TiO_2 (Fig. 6(b)) is smaller than that of Al/1 vol% TiO_2. These particles have a significant effect on the fracture surface of composites. Presence of particles on the core and walls of dimples which makes agglomeration and particle-matrix interface that provides suitable areas for crack initiation and propagation.

3.3 Peeling test

The variation of bond strength among the composite layers as a function of volume content of TiO_2 particles is shown in Fig. 7. As can be seen in Fig. 7, increasing the volume content of TiO_2 particles leads to decreasing the bond strength from 0% up to 10%. Increasing the volume content of TiO_2 particles leads to generation a harder condition for flowing the virgin material through the particles and as a result, the width of the bonds decreases. So, the roll bonding mechanism of AA1060/TiO_2 composites can be proposed as the extruding of underlying virgin and clean base metal through the cracks in the work hardened layer and TiO_2 particles. However, the extruded metals cannot create a strong bond at deformations lower than a threshold thickness reduction. The roll bonding threshold is evidently a key factor to create a successful metallurgical bond which is defined as the minimum percentage reduction required to generate a successful bonding.

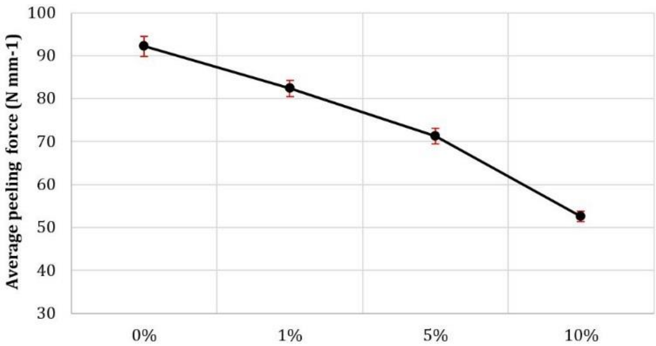

Fig. 7 Average peeling force versus the TiO_2 volume contents

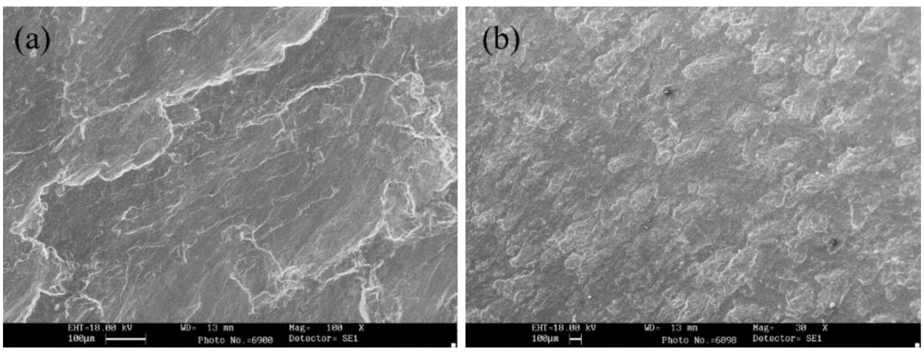

Fig. 8 SEM image of a peeled surface of samples with (a) 0%; and (b) 10% of TiO_2 particles volume contents

3.4 Peeling surface

The cracks and the extruded virgin metal (bonded areas) through them are obviously observed in Fig. 8. By continuing the ARB process, the virgin metal flows through the cracks in the work hardened layer under the action of normal rolling pressure and forms metallic bonds looks like the isolated islands, Fig. 8.

As mentioned before, by increasing the volume content of TiO_2 particles, the bonding strength decreases from 0% up to 10%. In this state, due to severe work hardening of the Al matrix, there are more barriers for extruding the virgin metal. So, as can be seen in Fig. 8, the bonding areas decrease drastically which leads to decreasing the amount of bonding strength.

4. Conclusions

In this study, AA1060/TiO_2 composites were produced in the form of strips through the Warm-ARB process successfully. The influence of TiO_2 particles on the mechanical, bonding properties and microstructural evolution of AA1060/TiO_2 composites has been investigated. The following results can be concluded:

- Warm accumulative roll bonding is a successful process that can be used to produce MMCs with higher tensile toughness than conventional.
- Increasing the volume percentage of TiO_2 particles enhances the strength of composites. So, it reaches to a maximum value of 181.26 MPa with 10% volume content of TiO_2 particles which is about 1.42 times higher than the strength of the monolithic sample.
- The maximum elongation of the monolithic sample is 3.98% while it reaches to 3.71% (AMMC with 1% of TiO_2) and 1.79% (AMMC with 10% volume of TiO_2). This means that TiO_2 particles have an enhancing effect on the elongation after a certain number of cycles (four cycles, in this study).
- The average Vickers hardness amount of the samples improves from 158 to 167 which is about 5.7% for samples fabricated with 0% up to 10% of TiO_2, respectively.
- By increasing the TiO_2 volume content, the bond strength among the layers decreases slightly. The bond strength of monolithic sample exceeds 1.75 times of AMMC with 10% volume percentage of TiO_2 particles which is due to lower forming ability of virgin metals among the TiO_2 particles.

References

Baazamat, S., Tajally, M. and Borhani, E. (2015), "Fabrication and characteristic of al-based hybrid nanocomposite reinforced with wo3 and sic by accumulative roll bonding process", *J. Alloys Compounds*, **653**, 39-46. http://dx.doi.org/10.1016/j.jallcom.2015.08.267

Chin, E.S. (1999), "Army focused team on functionally graded armor composites", *Mater. Sci. Eng. A*, **259**, 155-161. https://doi.org/10.1016/S0921-5093 (98)00883-1

Darmiani, E., Danaee, I., Golozar, M.A. and Toroghinejad, M.R. (2013), "Corrosion investigation of Al - Sic nano-composite fabricated by accumulative roll bonding (ARB) process", *J. Alloys Compounds*, **552**, 31-39. https://doi.org/10.1016/j.jallcom.2012.10.069

Eizadjou, M., Manesh, H.D. and Janghorban, K. (2008), "Investigation of roll bonding between aluminum alloy strips", *Mater. Des.*, **29**(4), 909-913. https://doi.org/10.1016/j.matdes.2007.03.020

Emadinia, O., Simões, S., Viana, F., Vieira, M.F., Cavaleiro, A.J., Ramos, A.S. and Vieira, M.T. (2016), "Cold rolled versus sputtered Ni/Ti multilayers for reaction-assisted diffusion bonding", *Weld. World*, **60**(2), 337-344. https://doi.org/10.1007/s40194-015-0282-8

Heydari Vini, M., Sedighi, M. and Mondali, M. (2016), "Mechanical properties and microstructural evolution of AA5083/Al2O3 composites fabricated by warm accumulative roll bonding", *J. Adv. Des. Manuf. Technol.*, **9**(4), 13-22.

Jamaati, R., Amirkhanlou, S., Toroghinejad, M.R. and Niroumand, B. (2011), "Effect of particle size on microstructure and mechanical properties of composites produced by ARB process", *Mater. Sci. Eng. A*, **528**(4-5), 2143-2148. https://doi.org/10.1016/j.msea.2010.11.056

Kaczmar, J.W., Pietrzak, K. and Włosiński, W. (2000), "The production and application of metal matrix composite materials", *J. Mater. Process. Technol.*, **106**(1-3), 58-67. https://doi.org/10.1016/S0924-0136(00)00639-7

Mandal, D., Dutta, B.K. and Panigrahi, S.C. (2007), "Dry sliding wear behavior of stir cast aluminium base short steel fiber reinforced composites", *J. Mater. Sci.*, **42**, 8622-8628. https://doi.org/10.1007/s10853-006-1271-5

Ng, M.K., Li, L., Fan, Z., Gao, R.X., Smith III, E.F., Ehmann, K.F. and Cao, J. (2015), "Joining sheet metals by electrically-assisted roll bonding", *CIRP Annals – Manuf. Technol.*, **64**(1), 273-276. https://doi.org/10.1016/j.cirp.2015.04.131

Reihanian, M., Hadadian, F.K. and Paydar, M.H. (2014), "Fabrication of Al–2 Vol% Al_2O_3/SiC hybrid composite via accumulative roll bonding (ARB): an investigation of the microstructure and mechanical properties", *Mater. Sci. Eng. A*, **607**, 88-196. https://doi.org/10.1016/j.msea.2014.04.013

Saito, Y., Utsunomiya, H., Tsuji, N. and Sakai, T. (1999), "Novel ultra-high straining process for bulk materials development of the accumulative roll-bonding (ARB) process", *Acta Mater.*, **47**, 579-583. https://doi.org/10.1016/S1359-6454(98)00365-6

Sedighi, M., Heydari Vini, M. and Farhadipour, P. (2016a), "Effect of alumina content on the mechanical properties of AA5083/Al_2O_3 composites fabricated by warm accumulative roll bonding", *J. Powder Metallur. Metal Ceram.*, **55**(7-8), 413-418. http://dx.doi.org/10.1007%2Fs11106-016-9821-0

Sedighi, M., Heydari Vini, M. and Farhadipour, P. (2016b), "Investigation of roll bonding between AA5083 strips", *Adv. Mater. Process.*, **4**(3), 33-42.

CH