

Available online at www.jourcc.comJournal homepage: www.JOURCC.com

Journal of Composites and Compounds

Characterization of TiB_2 reinforced aluminum matrix composite synthesized by in situ stir casting method

Fereshteh Barragh Jam ^a , Hadi Bangi Houri ^{b*} , Morteza Ferdosi ^c

^a Department of Chemical Engineering, Faculty of Engineering, University of Maragheh, Maragheh, Iran

^b Department of Materials and Metallurgical Engineering, Ferdowsi University of Mashhad, Mashhad, Iran

^c Faculty of Materials and Metallurgical Engineering, Semnan University, Semnan, Iran

ABSTRACT

In this study, TiB_2 reinforced Al-matrix composite was fabricated by in situ stir casting route, and the effect of processing parameters was investigated. X-ray diffraction (XRD) and field emission scanning electron microscopy (FE-SEM) equipped with energy-dispersive X-ray spectroscopy (EDX) were used to study the composition and microstructure of the samples. Finally, to investigate the tribological and electrochemical behavior of the samples, wear tests (pin-on-disk) and potentiodynamic polarization tests (PDP) were used, respectively. Results showed that by increasing stirring time, both tribological and mechanical behavior of the samples improved. Also, it was found that by increasing the stirring speed of the melt to 180 rpm, the mechanical and tribological behavior of the samples improved, and by further increasing the stirring speed to 300 rpm, they were decreased. Consequently, samples containing lower than 7 wt. % TiB_2 showed better metallurgical properties, due to lack of agglomeration.

©2020 JCC Research Group.

Peer review under responsibility of JCC Research Group

ARTICLE INFORMATION

Article history:

Received 17 September 2020

Received in revised form 25 November 2020

Accepted 07 December 2020

Keywords:

In situ

Composite

TiB_2

Wear resistance

Stir casting

1. Introduction

One of the most abundant metallic element in nature, after O_2 and Si , is aluminum, which makes up 8.13% of the Earth's crust. Aluminum is a white-silvery and soft metal that is a chemical element with an atomic number of 13. Also, under normal conditions, it is insoluble in water [1]. This metal is found in combination with other elements such as phosphate [2], sulfate [3], silicate [4], and hydroxide [5], and there is no pure state of this metal due to its high reactivity with other chemical elements. Al can also be found in clays, soils, minerals and rocks, and even in food and water. The outstanding features of this metal are its malleability and ductility. The Greek word alum that means astringent in medicine, is the root of the name aluminum [6]. During the reaction of Al with O_2 , water, and other oxidants, an Al_2O_3 film is formed on its surface which prevents corrosion of the metal [7]. Al_2O_3 is insoluble in water while easily soluble in strong alkalis and mineral acids, unlike aluminum sulfate, nitrate, and chloride that are dissolved in water [8, 9].

Due to the good wear resistance, high specific strength-to-weight ratio, superior damping capacity, and excellent dimensional stability of Al-based metal matrix composites (MMCs) that are reinforced with ceramic particles, it has been noticed by researchers [10-12]. The incorporation of externally synthesized reinforcements, like TiC , Al_2O_3 , and SiC , to the matrix alloys, is commonly applied for preparing Al-based composites (ex-situ composites) [13-16]. In this method, without proper modifica-

tion of the ceramic particle, the addition of reinforcements could cause poor adhesion at the interface, thermodynamic instability, and segregation of the reinforcements [17, 18]. The superior bonding in the interface of matrix and reinforcement, uniform distribution of smaller particles, process economy, and thermodynamical stability of the reinforcing particles are the advantages of the in-situ synthesized MMCs because the reinforcement formation takes place within the matrix [4, 19, 20]. For the fabrication of the Al-based MMCs, many in-situ formed ceramic particulates including TiC , TiB_2 , and Al_2O_3 can be used [21-23].

Due to the outstanding wear resistance, high hardness (3400 HV), high elastic modulus (534 GPa), and high melting point (3225 °C) of TiB_2 , it is an advanced strengthening phase for Al matrix [24]. Moreover, TiB_2 does not show any reaction with Al to form detrimental products at the interface of the matrix and reinforcement and this is a special reason for their application [18, 25]. The mechanical erosion resistance, oxidation stability, and excellent heat conductivity are the properties of TiB_2 as a hard ceramic. TiB_2 reacts with sulfuric acid, nitric acid, and alkalis, however, it is not oxidized in the air above 1100 °C and also in hydrofluoric and hydrochloric acids [26-29].

To prepare in-situ composites of Al/ TiB_2 with enhanced dispersion of the reinforcing agent, a new method of mechanical stirring at the interface of Al and the salts was developed by Chen et al. [30]. They reported that the improvement in the distribution of TiB_2 particles in the aluminum matrix was due to the interface mechanical stirring. The speed of 180 rpm stirring was applied for both first 15 min and last 15min of the

* Corresponding author: Hadi Bangi Houri; E-mail: Pers73hadi@gmail.com

DOI: 10.1001.1.26765837.2020.2.5.1.5

<https://doi.org/10.29252/jcc.2.4.1>

This is an open access article under the CC BY license (<https://creativecommons.org/licenses/by/4.0>)

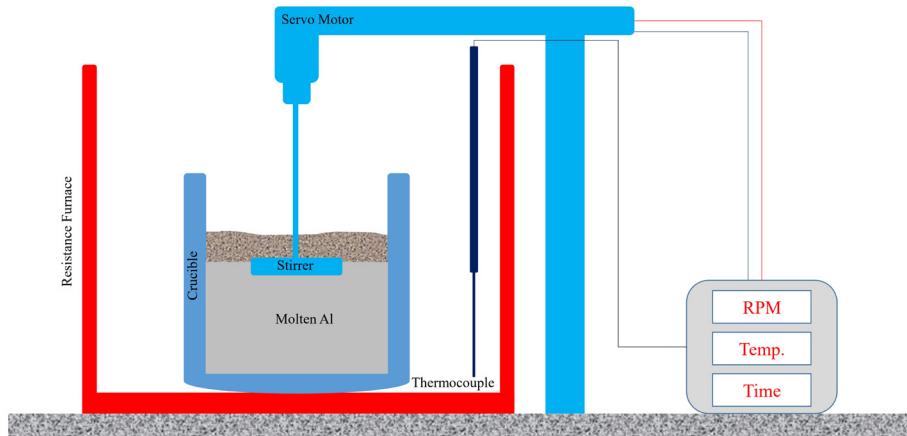


Fig. 1. The schematic representation of the installed equipment.

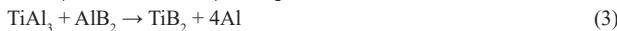
60 min for the synthesis process.

Pazhouhanfar et al. [31] synthesized the composite of Al-TiB₂ with 3, 6, and 9 wt. % of TiB₂ reinforcement by the method of stir casting. They analyzed the mechanical and microstructure properties of the composites. They reported that using the optimized process parameters leads to a uniform distribution without agglomeration for reinforcements in the aluminum matrix. Furthermore, preheating of TiB₂ powders and the addition of K₂TiF₆ results in a strong bonding.

The clusters of particles in the cast of MMCs are formed due to the combination of reinforcement settling effect and the particle rejection by the matrix dendrites while growing into the remaining melt during the solidification process [32]. In this work, the vortex casting method was applied for in-situ preparation of Al/TiB₂ composites which is an attractive method because of flexibility in using raw materials and the condition of processing. Also it has economic benefits of large-scale manufacturing. The amount of reinforcing particles, sample cooling rate, stirring start time, and stirring time are the process parameters.

2. Materials and methods

Aluminum matrix composite reinforced with TiB₂ particles has been produced through exothermic reactions, using Halide salts of KBF₄ – K₂TiF₆ within molten aluminum. This composite is produced through the following reactions.



The aluminum used here was %99.8 pure aluminum. %99 pure KBF₄ and K₂TiF₆ have been used as well. Fig. 1 displays the schematic representation of the equipment used for composite production. For any experiment, 1.5kg of aluminum has been melted and maintained in a graphite-clay crucible under a temperature of 850 °C. The powders used in this study have been dried up for two hours under a temperature of 200 °C. The powders have been mixed afterward with a Ti/B stoichiometric ratio of 1.2 and have been added to the molten mixture. After the powders have been fully solved in the molten mixture, a pre-heated four-bladed stirrer has been used to fully mix the molten material and composite-maker powders and stirred the mixture under different conditions. To prevent the entry of impure elements including Fe inside the molten mixture, the stirrer blades have been coated with nickel.

Different stirring velocities of the molten mixture were 0, 60, 180, and 300 rpm for 5, 15, 30, and 60 minutes respectively. To study the

impacts of the time starting from the rest time until the start of stirring on mechanical and tribological properties of the samples, 0, 15, 30 and 45 minutes have passed after adding the reinforcing particles to the molten mixture and stirring operation has been conducted then. It's noteworthy that the molten mixture's temperature has been conserved throughout these time durations to prevent the solidification of the mixture. To study the impact of reinforcing particle's amount on the properties of composites being produced, samples with different percentages of reinforcing particles (0, 1, 4, 7, and 9 wt. %) have been produced. The first series of samples being investigated were samples with different percentages of reinforcing materials which have been investigated after identifying the best reinforcing percentage of other remaining samples. Table 1 displays the characteristics of samples under investigation.

To study the phases involved in composites being produced, X-Ray Diffraction (XRD) procedure has been used. The device used here was Empyrean with Cu- K α and its radiation operated at 40 Kv and 40 mA. To study the microstructures, the samples have been sanded with 200-2000 sand and they have been polished then using felt and alumina powder (in micrometer scale) for 10 minutes. Finally, the FESEM device, Quanta 200 model has been used for imaging purposes.

Study the mechanical behavior of the samples, hardness and tensile assays have been used. Hardness testing has been undertaken using the Brinel device, made in Koopa. The study samples selected for hardness

Table 1.
The properties of samples produced under different conditions

Sample	TiB ₂ wt. %.	RPM	Time (min)	Time to Mix (min)
S1	Al Pure	-	-	-
S2	1	180	15	0
S3	4	180	15	0
S4	7	180	15	0
S5	9	180	15	0
S6	7	0	15	0
S7	7	60	15	0
S8	7	300	15	0
S9	7	180	5	0
S10	7	180	30	0
S11	7	180	60	0
S12	7	180	15	15
S13	7	180	15	30
S14	7	180	15	45

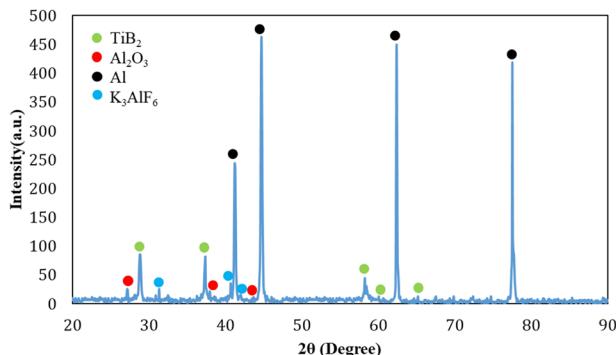


Fig. 2. XRD test results for S4 sample

testing have been all prepared based on the ASTM E8M-04 standard. Finally, to study the tensile behavior of the samples, Universal tensile testing device, the TB model has been used. The tensile strength of the samples has been tested under a velocity of 0.05 mm/s.

To study the tribological behavior of the samples, a pin on disk wear test has been used. The device used to conduct this test was the wear device, the Bong Shin model. This test has been conducted under room temperature and humidity. The wear velocity was 0.125 m/s and the wear distance was 200 m. the pin used in this study was made up of hardened steel with a hardness ratio of 50 HRC/ finally to test for the impact of reinforcing particles on the electrochemical behavior of the composites being produced, potentiodynamic polarization (PDP) test has been conducted in 3.5 wt. % NaCl solution. This test has been implemented using Origa Flex, made in France with a three-electrode system. The reference electrode used in this test was SCE. The scanning rate in this test was 0.5 mv/s. To develop balance in the Open Circuit Potential (OCP) of the samples, the samples have been immersed in a test solution for one hour.

3. Result and discussion

3.1. Characterization of composites with different TiB₂ content

3.1.1. XRD Analyses

Fig. 2 displays the XRD analysis of the S4 sample. This figure contains the peaks related to TiB₂ as a reinforcing particle. This peak illustrates that the produced composite is an in-place type of composite because the intended phase has been produced inside the molten material and through the reactions between the particles being added and

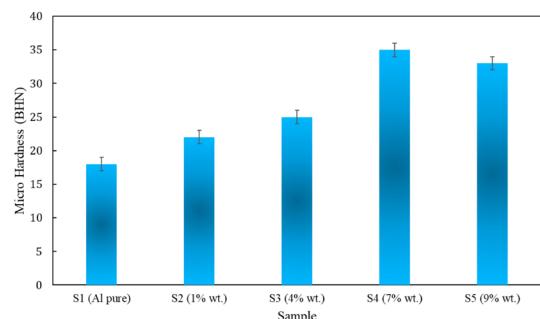


Fig. 4. The results of hardness testing for composites with different percentages of reinforcing particles

the matrix. The rest of the results illustrate that the main phases being produced are related to TiB₂ and Al phases. Considering the aluminum matrix used in this study, the presence of the main Al peak is not surprising. The presence of TiB₂ as the main peak suggests that the generated Gibbs energy involved in the formation of TiB₂ is far less than the Gibbs energy involved in AlTi₃ and AlB₂ formation [25, 33]. The emergence of this peak confirms reactions 1-3. The present study didn't report any intermediate product of TiAl₃ or AlB₂. Considering the Ti-B proportion, only 2.1 will be inferred which suggests that no significant portion of KBF₄ or K₂TiF₆ will be synthesized or oxidized during the reactions.

3.1.2. Morphology

Fig. 3 displays the FESEM images produced from S2, S3, S4, and S5. It can be inferred from these figures that reinforcing particles are present within the aluminum matrix. The presence of such particles leads to significant changes in the engineering properties of the samples. According to these figures, it can be concluded that in percentages higher than %7 of TiB₂ particle, agglomeration and clotting behavior occurred within the system. This may lead to the disappearance of sales' engineering behavior because pieces' properties are different in various parts of the samples and we can't record a uniform behavior of the sample. Considering the figures, it can be inferred that the matrix's particles became finer upon the presence of reinforcing particles. The further fineness of the matrix under the influence of reinforcing particles can be studied in two different parts. The first part concerns the initiation of solidification in which the reinforcing particles act as germinating particles and result in further fineness of the system's particles. The second part concerns the process of solidification itself through which the presence of reinforcing particles inhibits the growth of germinations being developed.

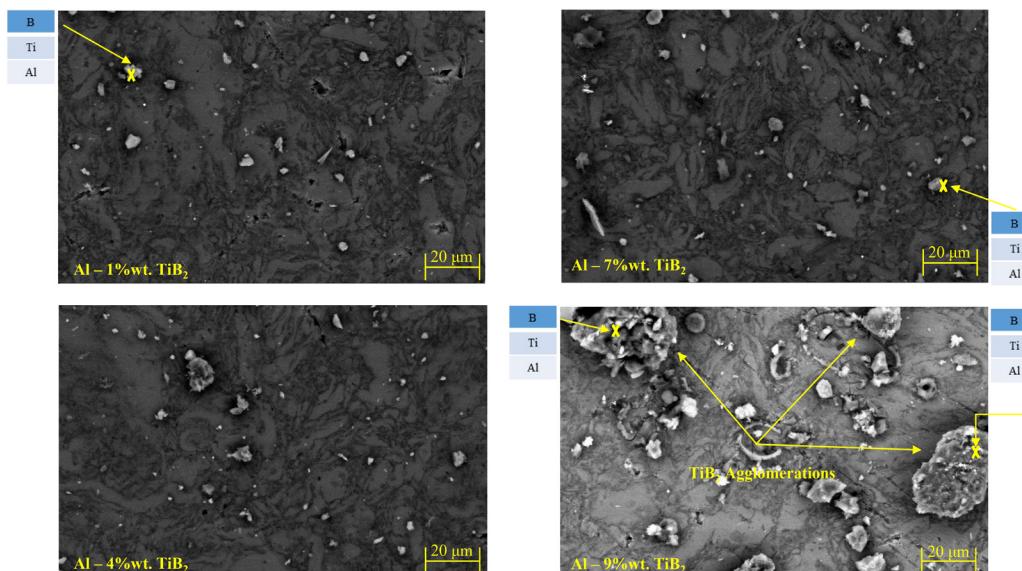


Fig. 3. FESEM images taken from aluminum matrix composites reinforced with different wt. % of TiB₂

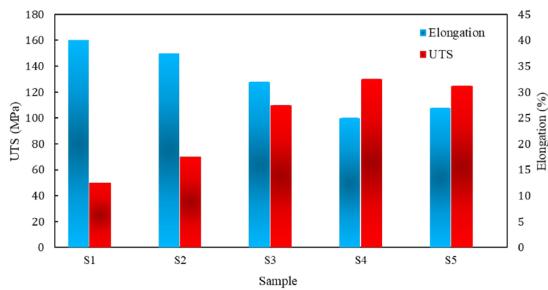


Fig. 5. The results of tensile strength tests for composites including different percentages of reinforcing particles

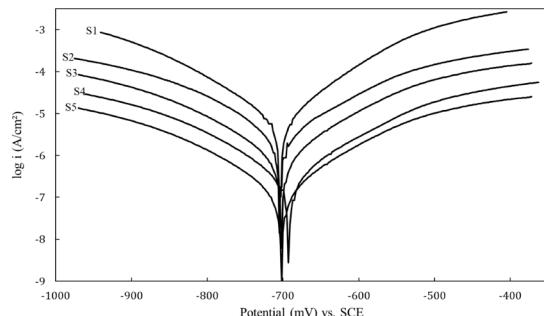


Fig. 7. Polarization diagram related to PDP test

To put it differently, the developed particles are surrounded by these particles and can't grow any further. It's crystal clear that the fineness of the matrix will contribute to improving the mechanical properties of study samples [34, 35].

3.1.3. Hardness

Fig. 4 displays the hardness test results. From this figure, it can be inferred that the presence of reinforcing particles resulted in the higher hardness of study samples compared with pure aluminum. This suggests that the presence of reinforcing particles under any conditions results in increased hardness of the study samples. Besides, the increased number of these particles resulted in increased hardness of the samples. The results of hardness tests illustrated that for values higher than 7 wt. % of reinforcing particles, composites' hardness has been reduced. This may be related to agglomeration and clotting of the particles the hardness reduction and weakening of composites' mechanical behavior upon the presence of reinforcing particles' agglomeration has been proved in the literature as well [29-32]. The presence of reinforcing particles resulted in the development of a higher energy level within the system which will finally result in increased hardness upon locking up the misplacements and development of blockages against misplacements. The mechanism involved in hardness testing is based on the plastic deformity on the surface of the study samples. The plastic deformation required the motion of misplacements; meanwhile, in case any factor prohibits the motion of misplacements, it will result in plastic deformity which will finally contribute to increased hardness.

3.1.4. Tensile

Fig. 5 displays the variations of tensile behavior of the composite samples with different percentages of reinforcing particles. It can be inferred that the presence of reinforcing particles up to 7% wt. of TiB_2 resulted in increased strength of the study samples and upon increasing the amount of reinforcing particles to 9% wt. resulted in decreased strength of the amole. The main reason behind the weakness of the sample's mechanical properties for higher percentages of reinforcing particles can be

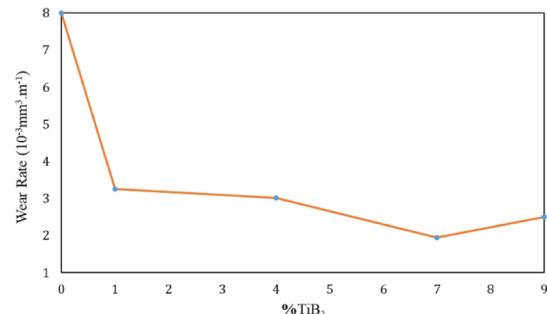


Fig. 6. Wear rate for composites containing different percentages of reinforcing particles

traced back to the agglomeration of reinforcing particles, which has been approved in FESEM images for 9% wt. of reinforcing particles. It can be inferred from Fig. 5 that the use of reinforcing particles resulted in the deceased elongation of the composites being produced. It's clear that the presence of reinforcing particles resulted in misplacements being locked up and therefore their motion potential will be reduced and this was the main reason for higher strength, misplacement lock up and finally decreased composite formation.

Finally, to study the mechanical behavior of the composites being produced (such as hardness and strength) which vary according to the presence of reinforcing particles can be discussed in two main parts [21]. First, based on the (continuous) transfer of matrix load to particles which has been developed through the favorable linking between the soft matrix and TiB_2 particles as strong and non-deficient particles. Whenever tension is incurred on a composite, it will be transferred to reinforcing particles from the matrix. As a result of such transfer of force, the soft and formable matrix will tolerate fewer loads and the reinforced particles will bear a significant portion of the force which will finally lead to improved behavior of the composite behavior. Another related factor is the reinforcing impact of TiB_2 particles on the surrender strength of the aluminum matrix. The impact of reinforcing particles on the composite strength (i.e. σ_{ys}) can be analyzed based on the following equation [11, 21].

$$\sigma_{yc} = \sigma_{ys} [V_p (1+S/2) + (1-V_p)] \quad (4)$$

Where σ_{yc} is the matrix's strength, V_p is the volumetric fraction of the reinforcing particles, and S is the superficial proportion of reinforcing particles which will be considered as equal to 1 for similar particles. The micromechanics involved in the increased strength of the composites may be originated from the following.

Any variation in the size of matrix particles (further fineness of the matrix) which based on Hall-Petch equation, the boundary of the increased particle will result in the development of obstructions against the motion of misplacements and will finally lock up the misplacements and decrease their motion which is itself the main origin for increased hardness and strength. The second point concerning the boundaries is the resource for misplacement generation. In other words, the boundary itself results in the development of misplacements and increased misplacement density which will lead to misplacement jungle, and locking up the misplacements will result in increased strength as well

$$(\Delta\sigma_{\text{Hall-Petch}}).$$

Orowan mechanism, based on the Orowan mechanism, the use of reinforcing particles will itself contribute to misplacement lock up and obstructs their motion. The pinning of the misplacements through using reinforcing particles will result in increased strength of the composites $\Delta\sigma_{\text{Orowan}}$ [33, 34].

The difference in heat expansion coefficient between reinforcing particles and the matrix leads to irregularities in the crystal and will

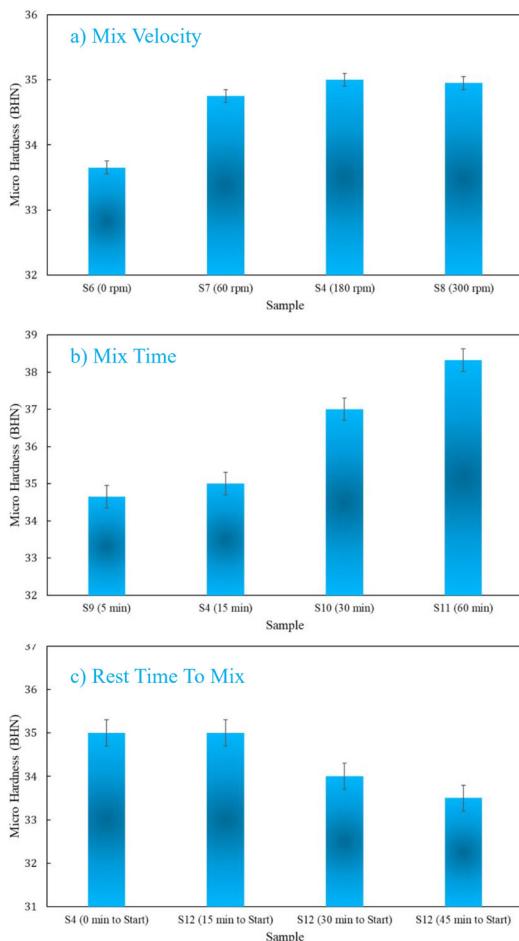


Fig. 8. Variations in samples' hardness under different conditions for the production of Al - 7% wt. TiB₂ composite; a) different stirring velocities; b) different stirring durations; c) different resting times and reactions between molten mixtures and aluminum

generate misplacements. The increased density of misplacements will lead to the formation of different misplacements locks which will finally contribute to decreased formability and strength of the samples ($\Delta\sigma_{CTE}$).

3.1.5. Wear

Fig. 6 displays the tribological behavior of the composites being produced using different percentages of reinforcing particles. It can be inferred from this figure that composites containing different percentages of reinforcing particles displays different tribological behavior. The figure suggests that the presence of reinforcing particles resulted in a decreased wear rate of the composites. Besides, increasing the amount of reinforcing particles up to 7 wt. % resulted in decreased wear rate and improved tribological behavior of the composite. Decreased rate of wear and improved tribological behavior of aluminum matrix composites has been proved in other studies available in the literature. A review of the related literature suggests that TiB₂-reinforced composites displayed favorable wear properties compared with composites reinforced with SiC [36-39]. Considering the study conducted by Prasada Rao et al.[40], It has been proved that the presence of TiB₂ particles resulted in decreased wear rate of aluminum matrix composites. According to Prasada Rao et al., the wear rate of the Al-7Si / TiB₂ composite depends upon the size of particles, the distance between dendrites, and the size of Si particles. In other words, in case any factors result in further fineness of system's particles during solidification process and finally, particles in lower dimensions will be developed, the wear properties will be improved and wear rate will be decreased. In this study, the use of reinforcing particles resulted in a decreased wear rate which will consequently lead to a reduction of grains' size. In other words, the presence of TiB₂ particles

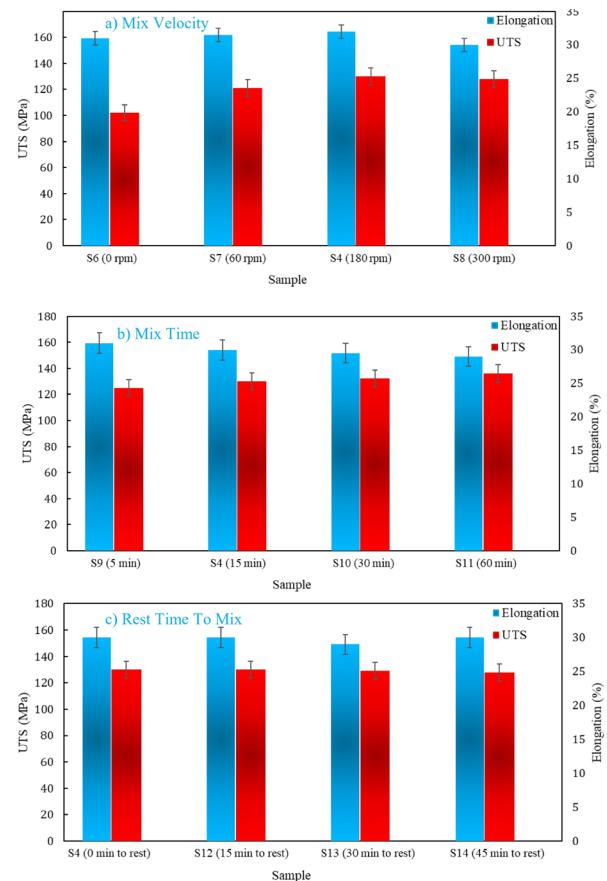


Fig. 9. The tensile behavior of the sample under different conditions for the production of Al - 7% wt. TiB₂ composite; a) different stirring velocities; b) different stirring durations; c) different resting times and reactions between molten mixtures and aluminum

modifies the particles' morphology and makes them smaller. Finally, the produced composite displays improved wear properties. Meanwhile, increasing the number of particles to higher than 7-9 wt. % results in higher wear rate and diminished sample's tribological behavior. The reason behind the increased wear rate in higher percentages of TiB₂ particles can be traced back to the agglomeration of the particles.

Considering the results obtained from mechanical and tribological tests, it has been suggested that the best parentage to produce aluminum matrix composite reinforced with TiB₂ particles, is 7 wt. % of TiB₂.

3.1.6. Corrosion

The potentiodynamic polarization (PDP) electrochemical test was used to study the effect of TiO₂ particles on the electrochemical properties of the produced composite, which the results are presented in Fig. 7. As shown, the presence of reinforcing particles results in changing the electrochemical behavior of particles. Also, their existence and an increase in their amount in composites lead to the formation of Tafel branches with low flows. In other words, their presence results in decreasing corrosion flow density (Table 2). Based on Fig. 7 and Table 2, adding the reinforcing particles fails to change the slope of Tafel branches and they are produced in lower flow density in parallel, which indicates no change in corrosion mechanism in the existence of the reinforcing particles. Their presence leads to no change in the mechanism of cathodic (e.g., hydrogen reduction) and anodic reactions (e.g., aluminum dissolution) although it only results in decreasing their rate and kinetics [41]. As represented in Table 7, the change in the amount of the reinforcing particles, and their presence or absence fail to vary the corrosion potential of the samples significantly, which represents no change

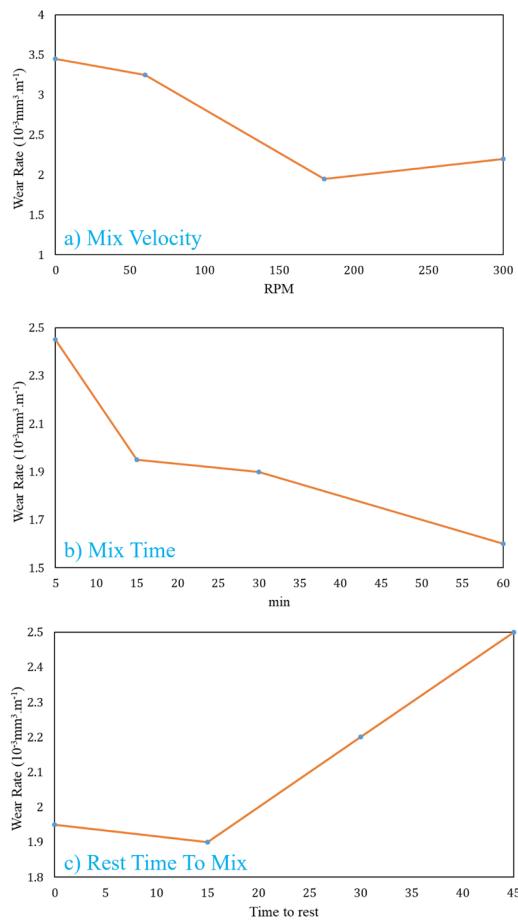


Fig. 10. Wear rate of the study sample under different conditions of Al - 7% wt. TiB_2 composite production; 1) different stirring velocities; b) different stirring durations; c) different resting times and implementation of reactions between molten mixture and aluminum

in the thermodynamic of electrochemical reactions in the presence of the particles [42-44]. Finally, their existence only alters the kinetics of electrochemical reactions. The blockage of the cavities and micro-cracks existing in the structure of aluminum-based by TiO_2 is considered as a reason for reducing the rate of electrochemical reactions (decreasing corrosion flow density) in the existence of the particles. The dangerous ions such as Cl^- can easily pass through each micro-crack due to their small radius (angstrom), which leads to corrosion in the system. The reinforcing particles reduce the diffusion rate of aggressive ions such as Cl^- because of blocking the micro-cracks, leading to a decrease in corrosion rate.

3.2. Characterization of composites with the different condition of fabrication

3.2.1. Mechanical Properties

Figs. 8-9 display the tensile and hardness behavior of the samples produced with different stirring velocity (a), stirring duration (b), and the resting time until the beginning of stirring (c). According to this figure, one can declare that making changes in any of the above parameters resulted in different tensile and hardness behavior of the samples. In other words, making changes in composite production conditions resulted in different mechanical properties of the composites.

To study the impact of stirring speed on the properties of the composites being produced, a reinforced version of the composite containing 7 wt. % of TiB_2 has been produced under different stirring velocities. According to Figs. 8-a,b, stirring contributes to higher hardness and

Table 2.
Results obtained from PDP test through tafel method

Sample	i_{Corr} ($\text{A} \cdot \text{cm}^2$)	E_{Corr} (mV)	β_a (mV.decade $^{-1}$)	β_c (mV.decade $^{-1}$)
S1	2.7×10^{-4}	-701	78	-90
S2	7.9×10^{-5}	-700	75	-89
S3	1.36×10^{-5}	-703	74	-91
S4	9.32×10^{-6}	-695	75	-88
S5	1.02×10^{-6}	-699	75	-92

mechanical strength of the composites which suggests that stirring and complete mixture of the molten material and reinforcing particles results in optimized mechanical properties of the samples. It's also clear that upon increasing stirring velocity up to 180 rpm, samples' hardness and strength have been increased. On the other hand, increased stirring velocity didn't affect hardness and samples' strength significantly. Improved hardness and strength of the study samples as a result of increased stirring velocity up to 180 rpm can be analyzed through two different points; first, under higher stirring velocities, more uniform scattering of the reinforcing particles will be developed in the matrix which will finally contribute to improved mechanical properties of the samples. Second, higher stirring velocities lead to the formation of finer-grained structures within the matrix. This fine-grained structure results in improved mechanical properties of the composite (Hall-Petch). It has been turned out that increased stirring velocity up to 180 rpm results in decreased mechanical properties of the samples which may be due to excessive turbulence and absorption of developed oxides on the surface of molten material and their entrance into that, such that under higher stirring velocities, the developed oxides on the surface of the molten material will be absorbed and imprisoned which finally results in the production of a deficient piece, with its mechanical properties being weakened. Accordingly, it has been made clear that the best stirring speed for the production aluminum matrix composite reinforced with 7% wt of TiB_2 is 180 rpm.

To study the impact of stirring duration, the sample was reinforced with 7 wt. % of reinforcing particles has been stirred for different time durations. Figs. 8-a, b display the samples' varied hardness and strength upon changing stirring time required for molten material. According to this figure, increased stirring duration resulted in higher hardness and strength of the composites. Improved strength and hardness of the composites upon increased time duration for stirring can be discussed in two levels. First, increasing stirring duration will contribute to better scatteredness of the particles within the system which will finally lead to better mechanical properties of the composite being produced. Second, fine-grained particles affect the system to a great extent. It has been found that upon increasing stirring duration, the microstructure, as well as the matrix's morphology, became finer and therefore the density of particles located on the boundaries will be higher. It's clear that a higher density of the boundary particles results in decreased motion of misplacements and therefore sample's strength will be increased as well. Accordingly, it's expected that samples' deformity and length increase will decrease during the tensile tests. The results of tensile tests all confirm this phenomenon.

To study the rest duration and the reactions that occurred between the added particles and molten material, different time durations have been established and samples have been produced under different resting times. In other words, the samples have been located inside the kiln under different time intervals of 0, 15, 30, and 45 minutes of stirring after the addition of reinforcing particles to the molten mixture. After these times, the mixture has been stirred once again and the molten mixture including the reinforcing particles has been loaded. Figs. 8-c and 9-c

display the hardness and strength variations of the composites produced under different resting times for the molten mixture and addition of reinforcing particles. It has been found that during the primary minutes, the molten mixture undertook required reactions and after some time has been passed, i.e. from 0-15 minutes, no significant changes have been made in samples' mechanical properties. Increased reaction duration between reinforcing particles and the matrix resulted in decreased mechanical properties of the samples. It suggests that increased duration of the reaction between reinforcing materials and the molten mixture results in unwanted reactions inside the molten mixture which will consequently lead to the production of an unfavorable phase. Finally, the presence of such unfavorable phases results in the devastation of the mechanical properties of the samples being produced.

Upon the comparison made between the hardness test results in Figs. 8b-c, it's clear that higher durations of stirring didn't result in a significant change in mechanical properties. Meanwhile, over time in static mode, the composite's hardness is decreased. It suggests that higher stirring durations prohibit the formation of unfavorable phases as well as the development of unwanted reactions and will finally contribute to the formation of a structure with unfavorable phases.

3.2.2. Wear

Fig. 10 displays the variations in wear rate for the samples being produced under different loading conditions. According to Fig. 10-a, it can be inferred that increased velocity for stirring the molten mixture resulted in lower wear resistance. Besides, increased stirring velocity of the molten mixture up to 180 rpm resulted in an increased wear rate. Weakened mechanical properties of the samples under increased stirring velocities have been confirmed in hardness and tensile tests. The reason behind the weakened wear behavior of the composites being produced under higher stirring velocities can be related to the ceramic and fragile phases which have entered inside the molten mixture from the slag due to turbulence. Molten mixture's turbulence will be increased under higher stirring velocities and the paths for oxygen penetration inside the molten mixture will be increased. Therefore, unfavorable oxidized phases will be formed inside the molten mixture which will finally contribute to lowered wear quality of the composite.

It's clear from Fig. 10-b that increased stirring duration resulted in a lower wear rate of the composite. The results of mechanical tests illustrated that increased mixing time for molten mixture and reinforcing particles will contribute to the development of a more uniformly scattered matrix of the reinforcing materials. This will also lead to a decreased risk of formation of reinforcing clots within the matrix and will lower down agglomeration risk as well.

Finally, Fig. 10-c displays that increased resting time results in weakened wear properties of the sample and increases the wear rate. This can be due to the formation of unfavorable phases under higher resting times, such that increased resting time and incomplete mixing of the molten mixture through this low time duration results in the formation of unfavorable phases which will consequently lead to weakened engineering properties of the samples including their wear properties.

4. Conclusions

1. Production of aluminum matrix composite reinforced with TiB_2 particles through the in-place method is possible through using $KBF_4 - K_2TiF_6$ Halide salts.
2. The presence of reinforcing particles within aluminum matrix composite resulted in heightened engineering properties (including hardness, wear, tensile, and corrosion) of the sample

composites being produced.

3. The best combination protocol for the production of aluminum matrix composite reinforced with TiB_2 particles is Al / 7% wt. TiB_2 .
4. It has been found out that increasing the percentage of TiB_2 particles up to 7 wt. % resulted in the agglomeration of the particles within the composite and weakened its engineering properties.
5. The reinforcing particles obstruct the entrance of dangerous ions such as Cl^- upon the obstruction of micro-cracks and pores existing on the surface of the composites and improved samples' electrochemical properties.
6. TiB_2 particles resulted in improved engineering properties of the samples by blocking the misplacements and blocking the particles' growth during solidification.
7. The mechanisms governing the improved strength and hardness of the composites reinforced with TiB_2 particles include Orowan and Hall-Petch.
8. Upon increasing stirring velocity, samples' engineering properties have been weakened as a result of the formation of oxidized phases and turbulence and solution of oxygen within the molten phase.
9. Increased stirring duration improved composites' engineering properties due to uniform scattering of the reinforcing particles inside the composite matrix.
10. Upon increasing the time interval between the moment of adding reinforcing particles to the loading moment (up to 15 minutes), samples' engineering properties will be weakened due to the occurrence of unwanted reactions and the formation of unfavorable phases.

Acknowledgments

The authors would like to acknowledge the Ferdowsi University of Mashhad for the financial support towards this research.

Conflict of interest

The authors declare that there is no conflict of interest.

REFERENCES

- [1] A.M. Davitoiu, E. Truta, A.M. Mitu, A.A. Bojescu, F. Grama, M. Ionica, The Role of Aluminum in the Symptomatology of Attention Deficit Hyperactivity Disorder Children, *Research and Science Today* 1 (2018) 115.
- [2] C.C.T. Leussa, L. Libessart, C. Djelal, C.N. Djangang, A. Elimbi, Pozzolanic activity of kaolins containing aluminum hydroxide, *Scientific Reports* 10(1) (2020) 1-12.
- [3] A. Bigham, A.H. Aghajanian, M. Movahedi, M. Sattary, M. Rafienia, L. Tayebi, A 3D nanostructured calcium-aluminum-silicate scaffold with hierarchical meso-macroporosity for bone tissue regeneration: Fabrication, sintering behavior, surface modification and in vitro studies, *Journal of the European Ceramic Society* 41(1) (2021) 941-962.
- [4] L.S. Fard, N.S. Peighambardoust, H.W. Jang, A. Dehghan, N.N.K. Salighi, M. Iranpour, M.I. Rajabi, The rechargeable aluminum-ion battery with different composite cathodes: A review, *Journal of Composites and Compounds* 2(4) (2020) 138-146.
- [5] D.-L. Vu, N. Kim, Y. Myung, M. Yang, J.-w. Lee, Aluminum phosphate as a bifunctional additive for improved cycling stability of Li-S batteries, *Journal of Power Sources* 459 (2020) 228068.
- [6] J.L. Domingo, J. Llobet, M. Gomez, J. Tomas, J. Corbella, Nutritional and toxicological effects of short-term ingestion of aluminum by the rat, *Research communications in chemical pathology and pharmacology* 56(3) (1987) 409.
- [7] V.T. Targhi, H. Omidvar, F. Sharifianjazi, A. Pakseresht, Hot corrosion behavior of aluminized and Si-modified aluminized coated IN-738LC produced by a novel hot-dip process, *Surfaces and Interfaces* 21 (2020) 100599.
- [8] M.G. Soni, S.M. White, W.G. Flamm, G.A. Burdock, Safety evaluation of dietary aluminum, *Regulatory toxicology and pharmacology* 33(1) (2001) 66-79.

[9] E.H. Jazi, R. Esalmi-Farsani, G. Borhani, F.S. Jazi, Synthesis and Characterization of In Situ Al-Al₁₃Fe₄-Al₂O₃-TiB₂ Nanocomposite Powder by Mechanical Alloying and Subsequent Heat Treatment, *Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry* 44(2) (2014) 177-184.

[10] Y. Zhang, N. Ma, H. Wang, Y. Le, X. Li, Damping capacity of in situ TiB₂ particulates reinforced aluminium composites with Ti addition, *Materials & design* 28(2) (2007) 628-632.

[11] S. Jayalakshmi, S. Gupta, S. Sankaranarayanan, S. Sahu, M. Gupta, Structural and mechanical properties of Ni₆₀Nb₄₀ amorphous alloy particle reinforced Al-based composites produced by microwave-assisted rapid sintering, *Materials Science and Engineering: A* 581 (2013) 119-127.

[12] S. Rahimi, F. SharifianJazi, A. Esmaeilkhani, M. Moradi, A.H. Safi Samghabadi, Effect of SiO₂ content on Y-TZP/Al₂O₃ ceramic-nanocomposite properties as potential dental applications, *Ceramics International* 46(8, Part A) (2020) 10910-10916.

[13] S.A. Sajjadi, H.R. Ezatpour, M. Torabi Parizi, Comparison of microstructure and mechanical properties of A356 aluminum alloy/Al₂O₃ composites fabricated by stir and compo-casting processes, *Materials & Design* 34 (2012) 106-111.

[14] P. Poddar, V.C. Srivastava, P.K. De, K.L. Sahoo, Processing and mechanical properties of SiC reinforced cast magnesium matrix composites by stir casting process, *Materials Science and Engineering: A* 460(461) (2007) 357-364.

[15] S. Gopalakrishnan, N. Murugan, Production and wear characterisation of AA 6061 matrix titanium carbide particulate reinforced composite by enhanced stir casting method, *Composites Part B: Engineering* 43(2) (2012) 302-308.

[16] B. Leila, S. Mostafa, S. Arman, A review of carbon nanotube/TiO₂ composite prepared via sol-gel method, *Journal of Composites and Compounds* 1(1) (2019) 1-9.

[17] S. Kumar, M. Chakraborty, V. Subramanya Sarma, B.S. Murty, Tensile and wear behaviour of in situ Al-7Si/TiB₂ particulate composites, *Wear* 265(1) (2008) 134-142.

[18] C.S. Ramesh, A. Ahamed, Friction and wear behaviour of cast Al 6063 based in situ metal matrix composites, *Wear* 271(9) (2011) 1928-1939.

[19] M. Emamy, M. Mahta, J. Rasizadeh, Formation of TiB₂ particles during dissolution of TiAl₃ in Al-TiB₂ metal matrix composite using an in situ technique, *Composites Science and Technology* 66(7) (2006) 1063-1066.

[20] S. Lakshmi, L. Lu, M. Gupta, In situ preparation of TiB₂ reinforced Al based composites, *Journal of Materials Processing Technology* 73(1) (1998) 160-166.

[21] Q. Zhang, B.L. Xiao, W.G. Wang, Z.Y. Ma, Reactive mechanism and mechanical properties of in situ composites fabricated from an Al-TiB₂ system by friction stir processing, *Acta Materialia* 60(20) (2012) 7090-7103.

[22] D.G. Zhao, X.F. Liu, Y.C. Pan, X.F. Bian, X.J. Liu, Microstructure and mechanical properties of in situ synthesized (TiB₂+Al₂O₃)/Al-Cu composites, *Journal of Materials Processing Technology* 189(1) (2007) 237-241.

[23] A. Masoudian, A. Tahaei, A. Shakiba, F. Sharifianjazi, J.A. Mohandes, Microstructure and mechanical properties of friction stir weld of dissimilar AZ31-O magnesium alloy to 6061-T6 aluminum alloy, *Transactions of nonferrous metals society of China* 24(5) (2014) 1317-1322.

[24] M.F. Heragh, S. Eskandarnezhad, A. Dehghan, Ni-Cu matrix composite reinforced with CNTs: preparation, characterization, wear and corrosion behavior, inhibitory effects, *Journal of Composites and Compounds* 2(4) (2020) 123-128.

[25] S. Suresh, N. Shenbag, V. Moorthi, Aluminium-Titanium Diboride (Al-TiB₂) Metal Matrix Composites: Challenges and Opportunities, *Procedia Engineering* 38 (2012) 89-97.

[26] A.C. Reddy, Investigation of the Clustering Behavior of Titanium Diboride Particles in TiB₂/AA2024 Alloy Metal Matrix Composites, 4th International Conference on Composite Materials and Characterization, Hyderabad, India, 2003, pp. 216-220.

[27] B. Basu, G. Raju, A. Suri, Processing and properties of monolithic TiB₂ based materials, *International materials reviews* 51(6) (2006) 352-374.

[28] A. Abuchenari, M. Moradi, The Effect of Cu-substitution on the microstructure and magnetic properties of Fe-15%Ni alloy prepared by mechanical alloying, *Journal of Composites and Compounds* 1(1) (2019) 10-15.

[29] E. Sharifi Sedeh, S. Mirdamadi, F. Sharifianjazi, M. Tahriri, Synthesis and evaluation of mechanical and biological properties of scaffold prepared from Ti and Mg with different volume percent, *Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry* 45(7) (2015) 1087-1091.

[30] F. Chen, Z. Chen, F. Mao, T. Wang, Z. Cao, TiB₂ reinforced aluminum based in situ composites fabricated by stir casting, *Materials Science and Engineering: A* 625 (2015) 357-368.

[31] Y. Pazhouhanfar, B. Eghbali, Microstructural characterization and mechanical properties of TiB₂ reinforced Al6061 matrix composites produced using stir casting process, *Materials Science and Engineering: A* 710 (2018) 172-180.

[32] A.C. Reddy, Fracture behaviour of brittle matrix and alumina trihydrate particulate composites, NISCAIR-CSIR, India (2002) 365-368.

[33] Z. Sadeghian, M.H. Enayati, P. Beiss, In situ production of Al-TiB₂ nanocomposite by double-step mechanical alloying, *Journal of Materials Science* 44(10) (2009) 2566-2572.

[34] L. Lu, M.O. Lai, F.L. Chen, Al-4 wt% Cu Composite reinforced with in-situ TiB₂ particles, *Acta Materialia* 45(10) (1997) 4297-4309.

[35] H.B. Michael Rajan, S. Ramabalan, I. Dinaharan, S.J. Vijay, Synthesis and characterization of in situ formed titanium diboride particulate reinforced AA7075 aluminum alloy cast composites, *Materials & Design* 44 (2013) 438-445.

[36] M. Huang, X. Li, H. Yi, N. Ma, H. Wang, Effect of in situ TiB₂ particle reinforcement on the creep resistance of hypoeutectic Al-12Si alloy, *Journal of alloys and compounds* 389(1-2) (2005) 275-280.

[37] P.L. Schaffer, L. Arnberg, A.K. Dahle, Segregation of particles and its influence on the morphology of the eutectic silicon phase in Al-7 wt.% Si alloys, *Scripta materialia* 54(4) (2006) 677-682.

[38] J. Wood, P. Davies, J. Kellie, Properties of reactively cast aluminium-TiB₂ alloys, *Materials science and technology* 9(10) (1993) 833-840.

[39] M. Zhao, G. Wu, L. Jiang, Z. Dou, Friction and wear properties of TiB₂P/Al composite, *Composites Part A: Applied Science and Manufacturing* 37(11) (2006) 1916-1921.

[40] A.P. Rao, K. Das, B. Murty, M. Chakraborty, Effect of grain refinement on wear properties of Al and Al-7Si alloy, *Wear* 257(1-2) (2004) 148-153.

[41] M. Behpour, S. Ghoreishi, M. Khayatkashani, N. Soltani, The effect of two oleo-gum resin exudate from Ferula assa-foetida and Dorema ammoniacum on mild steel corrosion in acidic media, *Corrosion science* 53(8) (2011) 2489-2501.

[42] M.F. Heragh, S. Eskandarnezhad, A. Dehghan, Ni-Cu matrix composite reinforced with CNTs: preparation, characterization, wear and corrosion behavior, inhibitory effects, *Journal of Composites and Compounds* 2(4) (2020) 123-128.

[43] M.F. Heragh, H. Tavakoli, Electrochemical properties of a new green corrosion inhibitor derived from *Prosopis farcta* for St37 steel in 1 M hydrochloric acid, *Metals and Materials International* 26(11) (2019) 1-10.

[44] A. Satapathy, G. Gunasekaran, S. Sahoo, K. Amit, P. Rodrigues, Corrosion inhibition by *Justicia gendarussa* plant extract in hydrochloric acid solution, *Corrosion science* 51(12) (2009) 2848-2856.