

EFFECT OF CARBONITRIDING ON TRIBOMECHANICAL AND CORROSION-RESISTANT PROPERTIES OF FRICTION STIR WELDED ALUMINIUM 2024 ALLOY

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Abstract: Friction stir welding (FSW) is extensively used to join aluminium alloys components in space and aircraft industries. Al 2024 is a heat-treatable aluminium alloy with copper as the primary alloying element which has good strength and fatigue resistance. This paper investigates the effect of carbonitriding surface modification on the hardness, tensile strength and impact strength of FSW welded Al 2024 joints. The friction stir welding was performed on three different sets of aluminium alloy (Al2024:Al2024, Al2024: carbonitrided-Al2024, carbonitrided-Al2024: carbonitrided-Al2024) at two different tool rotation speed (TRS) and two welding speed using cylindrical pin tool. The carbonitriding pre-treatment of Al-2024 alloy demonstrated significant improvement in the tensile strength, percentage elongation, abrasion wear resistance and corrosion resistance with the sacrifice of impact strength. The maximum tensile strength of all three sets of samples after FSW was recorded in descending order of (i) carbonitrided-Al2024:carbonitrided-Al2024 (ii) Al2024:Carbonitrided-Al2024 and (iii) Al2024:Al2024. The friction stir welded joint of carbonitrided aluminium alloy exhibited best abrasive wear resistant and corrosion resistant properties.

KEYWORDS: Friction stir welding, carbonitriding, hardness, tensile strength, impact strength

1 Introduction

Although more than 2000 years back, joining process can be traced, welding processes emerged in late 1800s. This occurrence invented various methods of welding e.g. gas welding, arc welding etc, and these types of welding came up with high level of gas, solidification defects because of the melting of the material and solidification process, and low density heat generation. Considering these issues, friction stir welding (FSW) was invented and patented in 1991 at The Welding Institute UK to avoid the common fusion weld problems such as weld solidification cracking, porosity, segregation, brittle intermetallic formation, and heat affected zone liquation cracking, which could come up with significant technological development in industries as solid-state welding of aluminium, magnesium steels in industries like automotive, ship and aerospace [1]. The necessity for greater fuel efficiency in automotive and aerospace sector have motivated the increased use of aluminium alloys and aluminium foam for lightweight designs in recent years [2, 3]. Friction stir welding is one of the most preferred welding technique for the welding of aluminium alloys as it is free from major weldability problems such a porosity and cracking. Friction stir welding has the upper edge of number of benefits including mechanical properties, improved process quality, no consumable, lower health and environmental issues, and reduced operating costs [4].

In FSW, weld is produced by rotating non consumable tool which plasticizes the work-pieces by friction and joins those together [3]. The FSW consists of mainly four zones such as stir zone, thermo-mechanically affected zone (TMAZ), heat affected zone (HAZ) and base material (BM) having deferent micro-structures [5-8]. The strength of friction stir welding mainly depends on tool tilt angle, tool pin profile, tool material, time of indentation of tool, tool rotation and traverse speed, axial force as FSW parameters [9-12]. Unlike the complex fusion welding processes involving heat [13, 14], the solid state welding, there has been limited literatures on the investigation of metallurgical/mechanical processing of materials before/after the friction stir welding of the aluminium sheet [15-19]. Aydin [19] performed the friction stir welding on different heat-treated Al-2024 alloys and demonstrated an increment in tensile properties after precipitation hardening of Al-2024 alloy. In a continued effort, the authors investigated the effect of surface hardening *via* carbonitriding of Al2024 alloy on the tribo-mechanical and corrosion resistant properties.

Kumar and Patel [20] analysed the microstructures and measured the mechanical, and abrasive wear properties of AA6061-T6 aluminium alloy by inter-plate reinforcing of the silicon carbide microparticles (μSiC) and silicon nitride microparticles ($\mu\text{Si}_3\text{N}_4$) and established that the reinforcement of μSiC and $\mu\text{Si}_3\text{N}_4$ decreased the tensile properties of the FSW joint but improved the abrasive wear and micro-hardness properties considerably in the stir zone (SZ). Bahuguna et al. [21] studied the effect of prior Ni coating of AA6061-T6 plates on mechanical and abrasive wear properties of FSW joints and found that the prior Ni coating significantly improved the tensile and abrasive wear properties. Das et al. [22] studied the effects of various parameters such as tool shoulder diameter, tool pin profile, plunge depth, tool rotation speed and welding speed on joint properties of FSWed joint of Al2014 alloy and demonstrated that the square pin profile of 24 mm shoulder diameter with a plunge depth of 0.06 mm and tool rotating at 815 rev/mm with welding performed at a speed of 63 mm/min delivered the best tensile and microstructural properties of FSWed joint due to its unique stirring effect by the pulsating action of the sharp edge of the pin and metal propagation process at the stir area. Ratna and Reddy [23] reported the corrosion behaviour of Mg-alloy and Al-alloy rolled sheets joined by friction stir welding and revealed that the joints had severe corrosion compared with base materials. The predominant corrosion mechanism behind the high corrosion rate of the joint was found to be high galvanic corrosion. They strongly suggested that Mg-Al joint was not suitable for applications where corrosion was suspected. Ganesh and Kumar [24] investigated the superplastic forming of friction stir welded AA6061-T6 alloy sheet at various tool rotation speeds. Experimental results indicated that the tool rotation speed was the critical parameter during friction stir welding that had a greater influence on superplastic forming and suggested that super-plasticity could be significantly enhanced by friction stir welding by varying the FSW tool rotation speed. Sajuri et al. [25] performed post-weld cold rolling (PWCR) on FSWed AA5083 joint at different thickness-reduction percentages (i.e., 10%, 20%, and 40%) to identify the effect of strain hardening on microstructure and mechanical Properties. The tensile strength of the PWCR treated FSWed joint increased with the increased cold-rolling percentage with greatest tensile strength observed at 403 MPa for PWCRed-40% due to reduced grain size in nugget zone and base metal. Further, the literature review reveals that there has been continued efforts towards improving the corrosion resistant behaviour of FSW joint of aluminium [26-29].

Most of the studies, an effort has been made to improve the mechanical properties of FSW joint by variation in feed and RPM and tool geometry, rather than studying the positive impact of prior surface hardening techniques for improved tribo-mechanical and corrosion resistant properties. Hence, the authors first time investigated the effects of carbonitriding of aluminium sheets before applying the friction stir welding on their tribo-mechanical and corrosion resistant properties. Al2024 is heat-treatable aluminium alloy with copper as the primary alloying

element. It is used in applications requiring high strength to weight ratio, as well as good fatigue resistance. Due to its high strength and fatigue resistance, Al2024 is widely used in aircraft structures. Carbonitriding is a surface hardening technique by thermal diffusion of active carbon and nitrogen atoms simultaneously onto the substrate material at required temperature. Carbonitriding of Al2024 was performed by applying unconventional environmentally friendly method of devised for low-melting-point materials. A plasma electrolysis saturation process (PESP) was reported to improve the corrosion resistance of aluminium and its alloy [30, 31]. In this paper, carbonitrided Al 2024 plates were joined by friction stir welding at varying RPM (spindle speed) and welding speed. The effect of carbonitriding surface hardening treatment on the hardness, tensile strength, impact strength, wear resistance and corrosion resistance of the FSW joints of Aluminium-2024 carbonitrided plates were reported.

2 Materials and Methods

2.1 Materials and Processing

Aluminium 2024 was purchased from Gayitri Industry, Roorkee, Uttarakhand. The composition of Al 2024 is presented in Table 1. The carbonitriding process was used for the surface hardening of aluminium 2024. The carbonitriding process was done at Dhruv Enterprises, Pune, India by using plasma assisted carbonitriding process on Al-2024 up to a depth of 0.3 mm thickness.

Table 1 Composition of Al 2024

Constituents	Al	Cr	Cu	Fe	Mg	Mn	Si	Ti	Zn	Other
Weight %	90.7-94.7	0.1	3.8-4.9	0.5	1.2-1.8	0.3-0.9	0.5	0.15	0.25	Max 0.20

2.2 Experimental Measurements

The carbonitrided Al-2024 plates were cut into 100 mm × 60 mm × 6.5 mm size for samples preparations. The cylindrical pin profile FSW tool made of H13 steel was used for the welding. The specification of tool is shown in figure 1. The FSW setup was prepared in the vertical milling machine to carry out welding.

The friction Stir Welding was carried out at two different tool rotation speeds of 1000 rpm and 1400 rpm and at two different welding speed of 14 mm/min and 20 mm/min on each sample of both plates without carbonitriding (Al2024:Al2024) process, one plate without carbonitrided and another carbonitrided (Al2024: carbonitrided-Al2024), and both plate carbonitrided (carbonitrided-Al2024: carbonitrided-Al2024) with total set of 12 experiment as represented by Table 2.

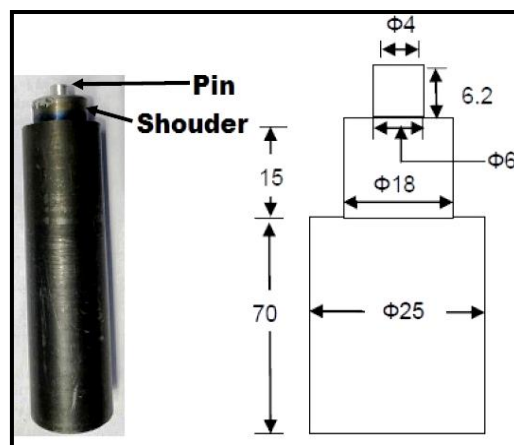


Fig. 1 FSW Tool (all dimensions are in mm)

After successful welding, the welded samples plates were cut from the welded joint as per ASTM standard for various testing to evaluate microstructure, tensile strength, hardness and wear resistance of the FSW joint. The tensile testing specimens of dimension 12 mm width 120 mm length and 6.5 mm thickness were prepared for the tensile test according to ASTM E-8 standard. The tensile was conducted on Computerized Universal Testing machine (UTM) and results are analysed to calculate the tensile strength of FSW joint. The images of the tensile tested samples are depicted in figure 2.



Fig. 2 Tensile tested samples

The Vickers hardness test was conducted at 5kgf minimum load and 200 kgf maximum loads for 15 sec. In each sample hardness was observed in different zones at 8 different points. Charpy impact test was carried out on digital impact testing machine (Veekay: Model-I 91). The standard size of the specimens 55mm×10mm×6.5mm and the depth of the notch is about 2mm were prepared as per ASTM A 370 standard as shown in figure 3.



Fig. 3 Charpy Test Samples

The surface of the wear specimens was cleaned with a soft paper soaked in acetone before the test. The specimen weight was measured using a digital electronic balance (0.001 mg accuracy) before it was mounted in the specimen holder. The difference between initial and final weight of the specimen was a measure of wear loss. As per ASTM G65 standard, the FSW specimen of size (75mm x 25mm) is pressed against a rotating wheel with a chlorobutyl rubber tyre of specified hardness. The abrasive was fed over the contact portion between the rotating rubber wheels and test specimen and the rate of feeding the abrasive was 365 g/min. The abrasion tested samples are shown in figure 4.

The material losses from the welding surface are measured by using precision electronic balance and then specific wear rate (SWR) was calculated by following equation (1).

$$W_s = \frac{\Delta m}{\rho l F_n} \quad (1)$$

Where, W_s = specific wear rate (mm^3/Nm); Δm = change in weight; ρ = density of the specimen (g/cm^3); F_n = normal load (N).

A metallurgical microscope (Banbros India, Model: BMI-101A) with digital camera attached to the computer was used for capturing the optical image at a magnification of 400x of microstructures. Microstructure was observed on cross-section of friction stir welded plate. For microstructural observation, the surface was ground first with emery papers of 120, 240, 320, 400 grit sizes followed by fine grit papers of size 0, 2, 3, 4. The direction of sample rubbing was changed 90° every time with changing grit paper size. Finally, the sample was polished on Hifin diamond compound hifinator polishing machine for half an hour. The Poultan etchant (2 ml HF, 3 ml HCL, 20 ml HNO_3 and 175 ml water) was applied for observing the grains distribution.



Fig. 4 FSW samples after sand abrasion test

Since aluminium 2024 is prone to corrosion, the corrosion test was performed. To perform the corrosion test, first 3.5 grams of NaCl and 100 ml distilled water solution was prepared. Then samples of FSW joint of Al-2024 of size 20×20×6 mm dimension was dipped in the solution for one week. After every one week the samples were taken off from the solution and kept in an oven for about an hour at 110 °C. Furthermore, samples were kept in desiccator for one hour to remove moisture contents. The weight loss was measured. The corrosion wear rate can be calculated by form following formula (2)

$$\text{Corrosion rate (Mils/year)} = \frac{534 W}{DAT} \quad (2)$$

Where 534: constant; W: the loss in weight in grams; A: area of specimen in inch; D = metal density in g/cm^3 , T: the exposure of time to the corrosion in hours.

3 Results and Discussion

The carbonitriding process was done to achieve anti corrosion layer on Al 2024 surface due to the formation of Al_4C_3 (aluminium carbide), AlN (Aluminium nitride) and $Al_7C_3N_3$ (aluminium carbonitride) phase on the surface of aluminium alloy [30].

3.1 Tensile Strength

The RPM (spindle speed) and feed has effect on tensile strength. Tensile strength and percentage elongation of with and without carbonitriding process samples after friction stir welding at various TRS and welding speed are shown in Table 2 and figure 5. From the figure, it can be revealed that the pre-treatment by carbonitriding of single-plate or both-plate before friction stir welding results in significant improvement in tensile properties over the untreated sample. The FSW joint of both plate carbonitrided delivered best tensile property of 320.90 MPa among all samples at tool rotation speed of 1000 rpm and welding speed of 14 mm/min. Table 2 and Figure 5 reveal that the tensile strength of aluminium 2024 without carbonitriding FSW joint attained maximum tensile property of 279.87 MPa at tool rotation speed of 1000 RPM and welding speed of 14mm/min welding speed, whereas the single-plate carbonitrided exhibited the maximum tensile property when friction stir welded at tool rotation speed of 1400 RPM and welding speed of 20 mm/min welding speed. The tensile strength of single as well as both-plate after carbonitriding exhibited almost similar tensile behaviour after friction stir welding at 1400 RPM and 14 mm/min. From elongation data reflected in Table 3, both single-plate and double-plate carbonitrided FSW joints exhibited better ductility behaviour than untreated aluminium alloy at all welding parameters (tool rotation speed/welding speed) with best elongation behaviour (21.08%) shown by double-plate carbonitrided FSW joint at TRS of 1000 rpm and welding speed of 20 mm/min. Hence for structural applications demanding both higher tensile strength and elongation, carbonitriding as pre-treatment before FSW is greatly beneficial imparting significant improvement in tensile strength (1.15 times higher @ TRS of 1000 rpm and welding speed of 14 mm/min) and elongation properties (1.77 times higher @ TRS of 1000 rpm and welding speed of 20 mm/min) than the untreated Al2024 alloy.

Table 2 Tensile strength and Percentage elongation of FSW joints

S. No	FSW Joint Samples	RPM	Welding Speed (mm/min)	UTS (MPa)	Percentage Elongation
1	Both plates without carbonitriding	1000	20	218.91	6.8
2		1000	14	279.872	11.88
3		1400	20	226.026	10.12
4		1400	14	275.962	11.12
5	With-without carbonitriding plates combination	1000	20	279.038	16.72
6		1000	14	296.859	17.12
7		1400	20	311.394	16.72
8		1400	14	291.294	14.20
9	Both plates carbonitrided	1000	20	303.846	21.08
10		1000	14	320.897	19.72
11		1400	20	296.21	16.32
12		1400	14	289.487	10.60

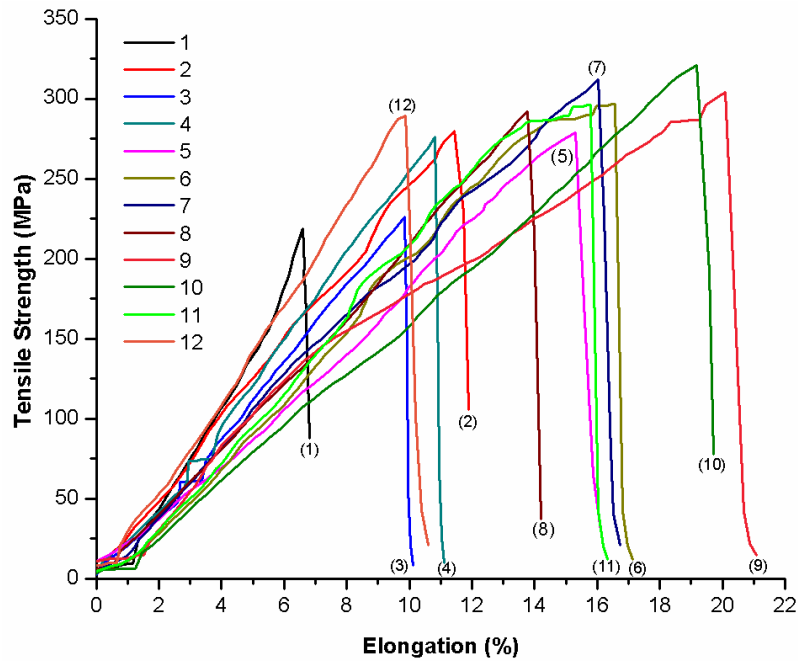


Fig. 5 Tensile strength vs percentage elongation of FSW joint at varying TRS (rpm) and welding speeds (mm/min).

3.2 Hardness

Hardness of joints was measured at 2mm and 4mm from the weld center in both directions. The comparison of hardness of FSW joints of untreated sample, single-plate carbonitrided, and both-plate carbonitrided at different welding speed and RPM are depicted in figures 6, 7, 8 and 9. In all hardness plot i.e figures 6, 7, 8 and 9, the center line “0” denotes the nugget zone, the data points with respect to the “-2 and +2” denote the thermo-mechanical affected zone (TMAZ) and the data points with respect to the “-4 and +4” denote the heat affected zone (HAZ). For welding parameter of 1000 rpm and 20 mm/min, the carbonitrided sample exhibits greater hardness with highest hardness in TMAZ than both with/without-carbonitrided and untreated aluminium alloy FSW joints. For rest of the welding parameters, the with/without-carbonitrided samples exhibit greater hardness than carbonitrided and untreated FSW aluminium alloy joints. In all samples, the base metals reflect the greatest hardness over the welded zones. After observing the hardness pattern of all 12 samples from fig. 6, 7, 8 and 9, it can be interpreted that the hardness of FSW joint of both-plate carbonitrided decreases with increment in friction deformation energy input exhibiting greatest hardness at least frictional energy input (i.e. @ TRS of 1000 rpm and welding speed of 20 mm/min) and least hardness at maximum frictional energy input (i.e. @ TRS of 1400 rpm and welding speed of 14 mm/min). However, in the FSW joint of single-plate carbonitrided and untreated samples, a similar trend of minor improvement in hardness was revealed with friction deformation energy input. It may be envisioned that the negative effect of frictional deformation energy input on hardness of single carbonitrided plate is counterbalanced by the positive gain in hardness of its adjoining untreated plate.

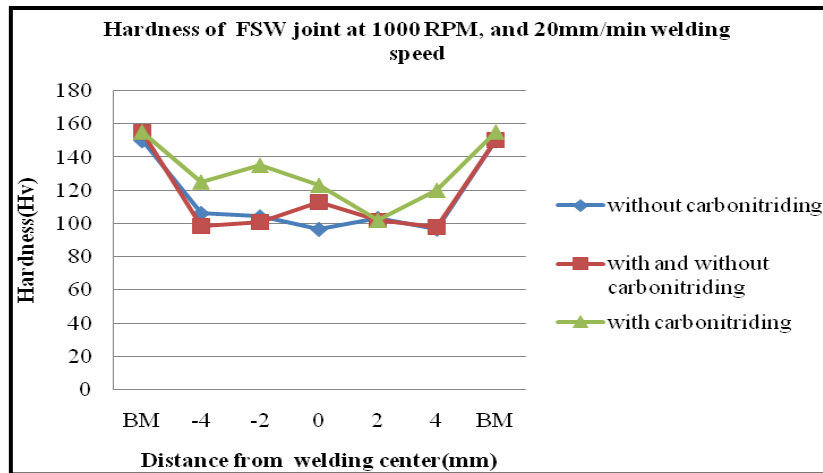


Fig. 6 Hardness of FSW joint at 1000 RPM and 20 mm/min welding speed.

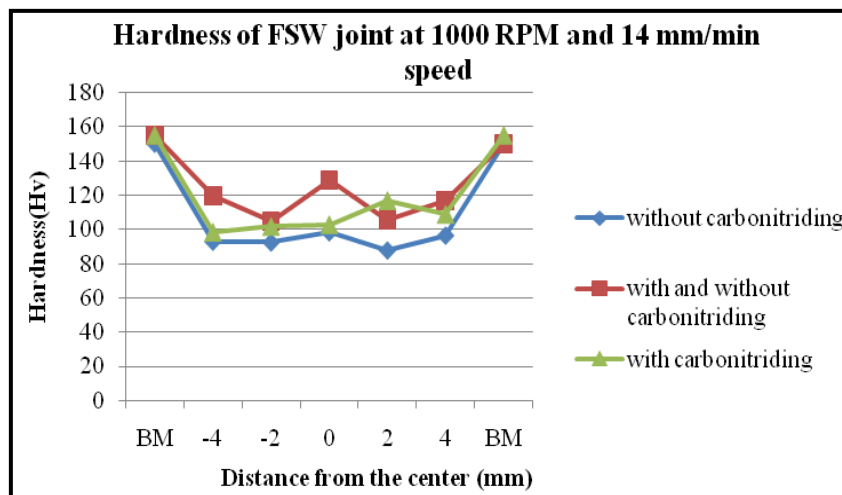


Fig. 7 Hardness of FSW joint at 1000 RPM and 14 mm/min welding speed.

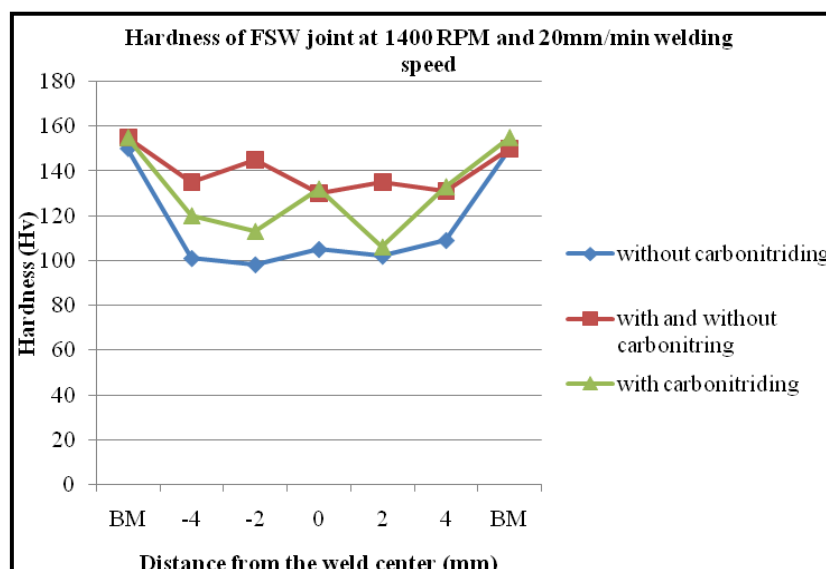


Fig. 8 Hardness of FSW joint at 1400 RPM and 20mm/min welding speed.

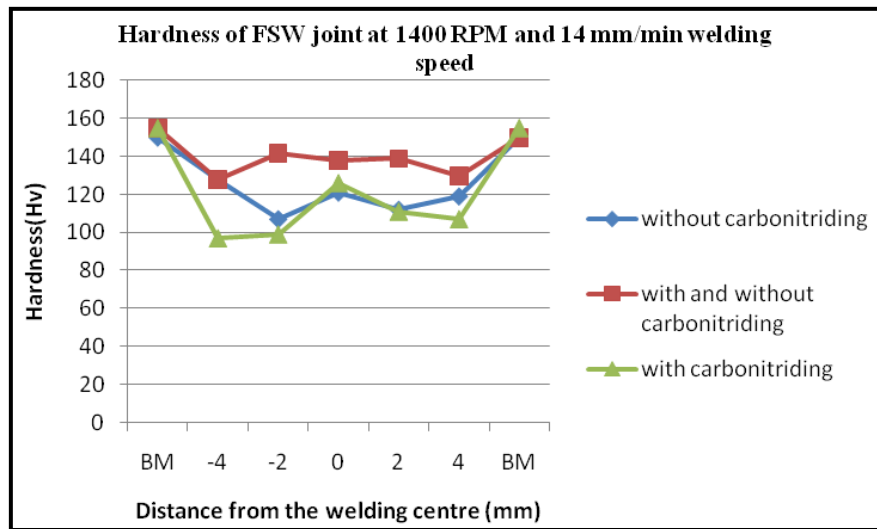


Fig. 9 Hardness of FSW joint at 1400 RPM and 14 mm/min welding speed.

3.3 Impact Strength

The Charpy impact test was conducted and the result is plotted in figure 10. From the figure, it can be interpreted that the greatest impact strength was displayed by the FSW joint of untreated sample at welding parameter of 1000 rpm and 14 mm/min. The descending order of impact strength was measured in terms of rank as (i) 9.4 J/cm² with FSW of untreated sample at welded at 1000 rpm and 14 mm/min, (ii) 6.4 J/cm² with FSW of untreated sample at welded at 1000 rpm and 20mm/min, (iii) 6.1 J/cm² with FSW of untreated sample at welded at 1400 rpm and 20mm/min, and (iv) 4.9 J/cm² with FSW of untreated sample at welded at 1400 rpm and 20mm/min. Hence, it is evident that carbonitriding degrades the impact properties and with/without-carbonitrided sample after FSW exhibits better impact properties in most of the cases (except at 1400 rpm and 14 mm/min) than both-plate carbonitrided FSW joints.

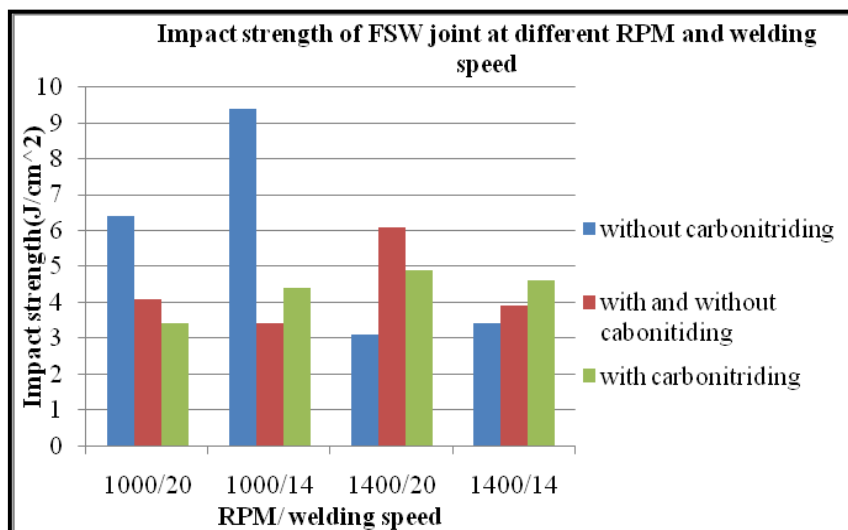


Fig. 10 Impact strength of FSW joint at different RPM and welding speed.

3.4 Microstructural Properties

The microstructure of the weld zone obtained for the welded joint delivering greatest tensile strength (here FSW of both plate carbonitrided) at its respective tool rotational speed of 1000 rpm and weld speed of 14 mm/min and the welded joint delivering least tensile strength (here the FSW joint of both plate untreated) at tool rotational speed of 1000 rpm and weld speed of 20 mm/min is presented in Figure 11 (a) and (b) respectively. A clear interface between stir zone (SZ) and thermos-mechanical affected zone (TMAZ) was more sharply evident in FSW joint of carburized plate than the FSW joint of untreated plate. The grain size of all zone of FSW joint of carburized plate is observed to be larger and distributed more uniformly than the FSW joint of untreated plate and this larger grain size combined with the strengthening influence of aluminium carbide, aluminium nitride and aluminium carbonitrided through surface carburizing process might be envisioned to impart better tensile properties with higher tensile strength and greater percentage elongation to friction stir welded carburized plate.

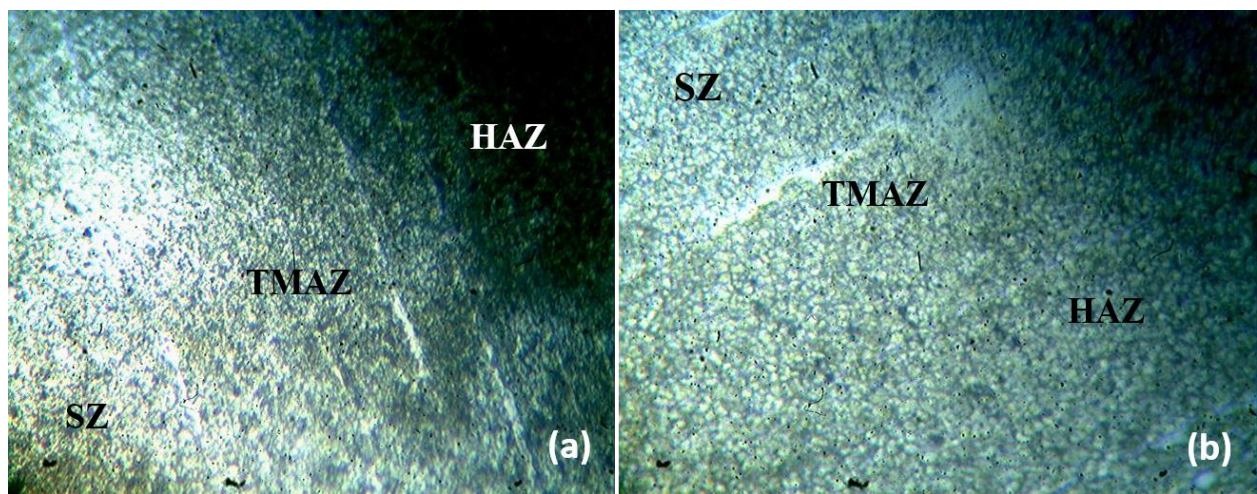


Fig. 11 Optical microstructures at magnification of 400x zoom of friction stir welded joints at tool rotational speed of 1000 rpm and weld speed of (a) 14 mm/min (b) 20 mm/min.

3.5 Abrasion Wear Properties

The specific wear rate of welded joints was calculated of all samples at constant sliding distance of 1435.608m, wheel rotational speed of 200 rpm, applied load of 79.086N and 2000 counter as shown in figure 11. Figure 11 showed that abrasion wear rate was maximum for the untreated sample at each welding parameter with greatest wear observed at 1000 rpm and 14 mm/min welding speed. Friction stir welded joints exhibited least wear rate at welding parameter of 1400 rpm and at welding speed of 20 mm/min with less variance in specific wear rate among all treated as well as untreated samples. The highest wear resistant property was revealed by the FSW joints of double-plate treated samples followed by one-plate carbonitrided sample and least wear resistant by untreated samples. The greatest abrasion wear resistant properties reflected by the FSW joint of double-plate carbonitrided aluminium alloy can be attributed to the presence of hard phase Al_4C_3 (Aluminium carbide), AlN (Aluminium nitride) and $\text{Al}_7\text{C}_3\text{N}_3$ (aluminium carbonitride) among the welded zone offering greater resistance to the sand abrasion wearing process.

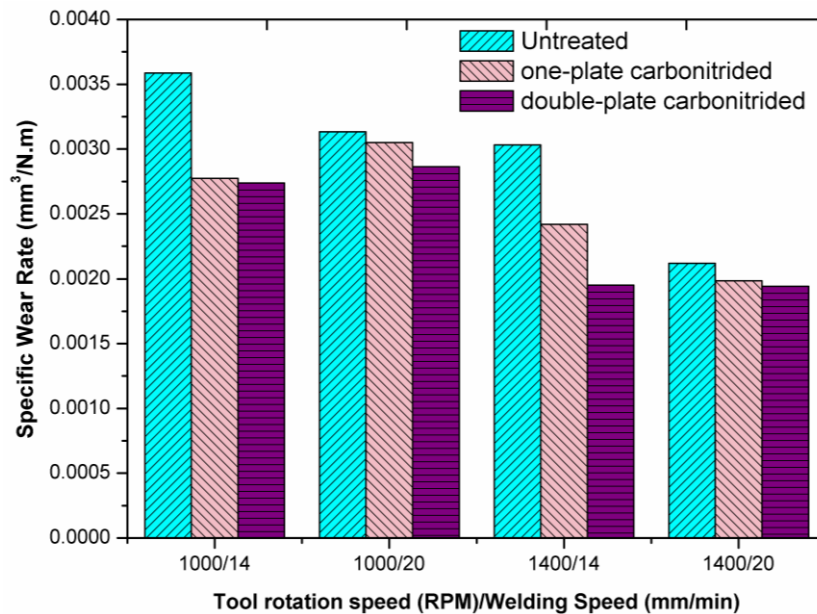


Fig. 12 Wear rate of different FSW samples.

3.6 Corrosion Properties

The four different test sample (as-procured Al2024 alloy, carbonitrided Al2024 alloy, FSW welded joint of untreated Al2024 alloy and FSW welded joint of carbonitrided Al2024 alloy) of constant surface area of 0.620 inch² and density of 2.78 g/cm³ were dipped separately into salt solution for 4 week durations to carry out the corrosion wear test. Different corrosion wear rates were observed for samples and plotted in figure 12. Figure 12 showed that corrosion wear rate of pure aluminium alloy was greatest amongst all samples. Furthermore, the FSW joint of carbonitrided Al2024 alloy exhibited least corrosion rate amongst all samples.

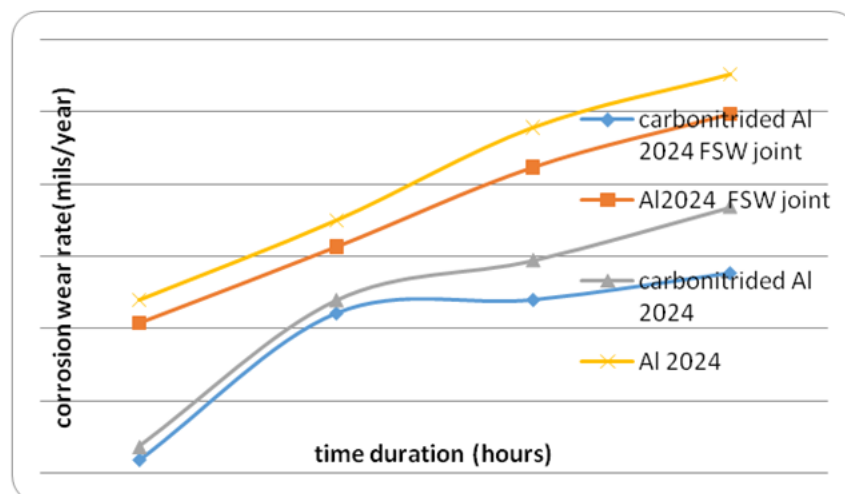


Fig. 13 Time vs Corrosion wear rate.

CONCLUSIONS

The effect of carbonitriding process on aluminium 2024 was observed on mechanical properties of welded joints. The following conclusions were drawn for the study:

- The FSW joint of carbonitriding pre-treated aluminium alloy exhibited superior tensile strength (1.15 times) and elongation properties (1.77 times) than the FSW joint of untreated aluminium alloy.
- The FSW joint of untreated and double-plate treated aluminium alloy attained maximum tensile strength at 1000 RPM and 14mm/min welding speed while the one-plate treated FSW joint attained the maximum tensile strength at TRS of 1400 RPM and welding speed of 20 mm/min.
- The carbonitriding pre-treatment followed by FSW imparted better elongation properties (1.77 times higher @ TRS of 1000 rpm and welding speed of 20 mm/min) than the FSW joint of untreated Al2024 alloy.
- The impact strength of carbonitrided FSW joint exhibited significant reduction in impact strength at TRS of 1000 RPM and welding speed of both 14 and 20 mm/min as compared to FSW joint of untreated one.
- Abrasion wear rate was minimum for the carbonitrided samples at all welding parameters with least wear rate at TRS of 1400 rpm and welding speed of 20mm/min feed. The corrosion wear rate was least for carbonitrided FSW joint amongst all samples.
- Overall, it can be recommended to carbonitride the material before FSW for imparting improved tensile, elongation, abrasion wear resistant and corrosion resistant properties.

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