Effect of heat treatment on AA2014 alloy processed through multi-pass friction stir processing

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Abstract. In this paper, the ultrafine grain (UFG) structure was prepared in AA2014 via multi-pass friction stir processing (FSP). Post-processing heat treatment was carried out in order to enhance the mechanical properties. For achieving this aim, trail experiments have been performed to opt the process parameters and number of passes. 3D optical microscopy, Vickers’s microhardness tester, and universal tensile testing (UTM) machine were employed to study the microstructural features and mechanical properties and the results have been reported. Scanning electron microscope (SEM) was used to examine the microstructures of fracture features of tensile specimens. The properties of the heat treated (HT) sample was found better than the non-heat treated (NHT) sample. Fracture surfaces of both samples show the presence of large dimples which indicates ductile failure.

Key words: Multi-pass friction stir processing; Ultrafine grain structure; AA2014.

1. Introduction

The demand for UFG structure has been increasing for the last two decades in the industrial fields and aerospace due to its versatile properties like enhanced superplasticity, high strength to weight ratio and improved mechanical properties. A wide variety of methods are used for producing UFG, yet FSP is the best method for producing UFG in aluminium alloys [1-2]. The working method of FSP is same as friction stir welding (FSW). A rotating non-consumable tool with designed shoulder and pin is plunged into the processed specimen and traversed along the processed line. The frictional heat is generated at the tool-work junction leads to intense plastic deformation in the penetrated zone [3-4]. AA2014-T6 is Al-Cu precipitation hardened alloy of 2xxx series. It finds enormous applications in aerospace, automobile filed, structural components and shipbuilding industries due to an advantage high strength to weight ratio [5]. Few kinds of literature are available on the influence of heat treatment on FSP of AA2014. However, the heat treatment improves the mechanical properties. Devaraju et al. [6] studied the influence of post welding rapid cooling on AA 2014. He found that the grain size is further decreased and mechanical properties are improved due to the sudden cooling in dry ice with ethanol. Yu Chen et al. [7] made an attempt to investigate the influence of post-welding heat treatment on the hardness of the stir zone of friction stir welded AA 2024 and he concluded that averaging is responsible for the decrease of hardness in stir zone. Charith et al. [8] carried out subsequent aging heat treatment for ultrafine grained friction stir processed Al-Zn-Mg-Si and reported that there is a slight improvement in mechanical properties after heat treatment without a change in microstructure. The influence of post-
processing heat treatment on microstructure and mechanical properties of multi-pass FSP of AA2024/SiC composites was investigated by Ghanbari et al. [9]. The results indicate that the hardness is decreased in the stir region with an increase in a number of passes without heat treatment. However, the hardness is increased for HT samples due to the uniform distribution of S-phase precipitates. In this, the effect of post-processing heat treatment on ultrafine grained AA2014 processed by multi-pass FSP was studied and results have been reported.

2. Experimental procedure

4mm thick-AA2014-T6 aluminium alloy was used as base metal for the present study. The detailed chemical composition of the alloy is given in Table 1. These experiments were performed on a 3-Ton capacity CNC based Friction stir welding machine. The tool material selected for FSW/FSP must have wear resistance, low coefficient of thermal expansion, high fracture toughness and it can withstand strength at elevated temperatures. Based on the existing literature AISI H13 tool steel was selected as tool material for FSP. The chemical composition of H13 tool is given in Table 2. The geometry of tool plays a crucial role in FSP and it must be selected carefully as to achieve the fine grains. For achieving fine grains in AA2014, a taper threaded tool with concave shoulder along with optimum tool geometry was selected for performing FSP is shown in Fig. 1.

Trial experiments were performed to fix the optimum process parameters. The parameters considered for the FSP are rotational speed and traverse speed. The other parameters, axial load, and tilt angle are kept constant at 1000 kg and \(2^\circ\) respectively. From the microstructure analysis of trial experiments, the sample processed with the rotational speed of 1100 rpm and traverse speed of 30 mm/min was found to be defect free and it is selected for further multi-pass experiments. Several multi-experiments were conducted and the third pass was found to be more grain refinement. So multi-pass with three-pass experiments were conducted in room temperature to get UFG structure and this sample is considered as non-heat treated (NHT) sample. The sample was kept in heat treatment furnace at 160\(^\circ\)C for 16 hours to improve the mechanical properties. The samples for microstructure analysis were sectioned by using a power hacksaw. The specimens were polished with different grades emery papers and then polished using disc polishing machine to have a scratch free surface. Then, the polished surface is cleaned with acetone and etched in Keller’s reagent for 20 seconds to reveal the microstructure. Microstructures were observed for various samples and different zones by using an optical 3D microscope. Hardness was measured along the processed zone with an interval of 0.5 mm using microhardness tester with a diamond indenter. Hardness test was done at load of 100 gf and 10 s dwell time. The sample for tensile testing was cut by wire-cut electrical discharge machine (EDM) as per the ASTM standards (Fig. 2). The tensile testing was performed in room temperature with a universal testing machine (UTM) with a crosshead speed of 0.5 mm/min. The fractography samples were analysed with scanning electron microscopy (SEM).

| Table 1. Composition of AA2014. |
|-----------------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| Element Comp (Wt%)          | Cu     | Mg     | Si     | Mn     | Fe     | Zn     | Ti     | Cr     | Al     |
|-----------------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
|                            | 4.25   | 0.80   | 0.69   | 0.64   | 0.23   | 0.17   | 0.023  | 0.029  | Remainder |

| Table 2. Composition of AISI H13. |
|----------------------------------|--------|--------|--------|--------|--------|--------|--------|--------|
| Element Comp (Wt%)               | Cr     | Mn     | C      | Mo     | P      | S      | Si     | V      |
|----------------------------------|--------|--------|--------|--------|--------|--------|--------|--------|
|                                | 5.1    | 0.4    | 0.39   | 1.3    | 0.02   | 0.02   | 1.05   | 1.1    |
3. Results and discussion

3.1 Microstructure

The microstructures of the base metal, non-heat treated stir zone (NHTSZ), non-heat treated heat affected zone (NHTHAZ), heat treated stir zone (HTSZ) and heat treated heat affected zone (HTHAZ) are shown in Fig. 3 (a)-(e). The optimized process parameters resulted in defect-free microstructure. The microstructure of base metal displays coarse grains of aluminum matrix along with small volume fraction of precipitates at grain boundaries with a grain size of 107 μm (Fig. 3 (a)). The microstructure of NHTSZ reveals the presence of small recrystallized grains of 3.87 μm (Fig. 3 (b)). These results attributed to the occurrence of intense plastic deformation at tool-work interface leads to dynamic recrystallization in the stir zone of NHT specimen and resulted in fine grains. From the microstructure of HTSZ, it was observed that there is no much grain coarsening after heat treatment (Fig. 3 (d)). The grain size of HTSZ was measured to be 3.9 μm which is almost similar to the NHTSZ. As expected, the heat-affected zones of heat treated and non-heat treated specimens show the marginal increment in grain size and approximately same as the grain size of the base metal (Fig. 3 (c & d)). This is due to the excess heat input and absence mechanical stirring.
Fig. 3. Microstructures of a) base metal, b) NHT stir zone, c) NHT heat affected zone, d) HT stir zone, e) HT heat affected zone.

3.2 Mechanical properties

The microhardness and tensile properties of the NON-HT and HT specimens are given in Table 3. The average hardness in the stir zone was found to be 102.3 HV and 110.6 for NON-HT and HT sample respectively. This is due to the fact that, during heat treatment, the precipitates become coarsened and the size of the precipitates is sufficient to stop the dislocations, hence resulted in better hardness for HT sample. But the hardness (151.3 HV) of the base sample is more compared to other two. The hardness distribution along the processed region of NON-HT and HT samples was shown in Fig. 4. There was a drop in hardness for HAZ region compared to SZ region. The HAZ experience high temperature during stirring action leads to a reduction in hardness. The tensile properties (Table 3) such as ultimate tensile strength (UTS), yield strength (YS), the percentage reduction in area and percentage elongation were calculated from the tensile data. From the results, it is clear that the UTS and YS were 15.2%, and 6.2% more for HT sample compared to NON-HT sample due to the precipitates behaviour during heat treatment as mentioned in hardness analysis. As we know that, the strength and elongation are
intentionally proportional to each other. So there was a marginal drop of 24.8% and 23.05% in elongation and reduction in area for HT samples than the HT samples.

The fracture features of the NON HT and HT samples were examined using SEM to know the fracture behaviour. The fractographs of two tensile samples display the existence of large dimples, which indicates the ductile failure (Fig. 5 (a & b)). The density of dimples was little more in NON-HT samples compared to HT sample (Fig. 5 (a)). Due to the precipitation hardening in HT samples, the fracture occurred earlier than NON-HT sample resulted in less density of dimples (Fig. 5 (b)). The fracture surface of HT samples shows the flat region could be attributed to lack of metallic bonding.

![Hardness profile of non-heat treated and heat treated specimens along the processed zone.](image)

**Fig. 4.** Hardness profile of non-heat treated and heat treated specimens along the processed zone.

![Fracture surfaces of a) NHT specimen, b) HT specimen.](image)

**Fig. 5.** Fracture surfaces of a) NHT specimen, b) HT specimen.
Table.3 Tensile properties of non-heat treated and heat treated specimens.

| Material condition | Yield strength (MPa) | Ultimate tensile strength (MPa) | Reduction in the area (%) | Elongation (%) | Hardness (HV) |
|--------------------|----------------------|-------------------------------|----------------------------|----------------|---------------|
| NHT                | 178.74               | 247.4                         | 35.99                      | 12.9           | 102.3         |
| HT                 | 210.71               | 263.6                         | 27.7                       | 9.7            | 110.6         |

4. Conclusions
The influence of post-FSP heat treatment on microstructural and mechanical properties of ultrafine-grained AA2014 processed through Friction stir processed was studied and following conclusions were drawn.
- From the multi-pass experiments, it was found that the grain size is decreased with increase in a number of passes up to the third pass.
- Of three zones of FSP, the stir zone experiences the intense plastic deformation leads to more grain refinement. Due to the fine grain size, the stir region resulted in better hardness compared to other zones.
- It was seen that the mechanical properties of heat treated and non-heat treated samples were less than the base metal.
- Due to well precipitation behavior, the mechanical properties were improved in heat treated sample compared to the non-heat treated sample without a change in grain size.

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