Modification of 5083 Aluminum Alloy with Graphene Via Friction Stir Processing

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Abstract. Graphene-modified layer is obtained on 5083 aluminum alloy sheet material via friction stir processing. A special groove is cut in the aluminum plate and filled with graphene. The processing is carried out using an innovative technology with an appropriate tool. The temperature at a chosen point in the heat affected zone is measured in real-time by a remote-control system. Test specimens were prepared from the processed plates and metallographic analysis was carried out. The microhardness of the modified layer is measured perpendicular to the direction of processing and in depth. An increase in microhardness relative to that of the base material is found.

Keywords: friction stir processing (FSP), friction stir welding (FSW), metallography, microhardness, parameters.

1. INTRODUCTION

Friction stir welding (FSW), and friction stir processing (FSP) in particular, are relatively new, successful methods for bonding or surface processing of solid-state materials. FSP has some advantages over fusion welding methods since the issues associated with cooling the liquid phase are avoided. Increasingly more studies are being conducted in order to further develop the FSP technology and to implement it faster in industrial production in order to exploit its advantages. The scientists found improved mechanical properties after FSP and FSW, such as hardness, tensile strength, bending strength, torsion strength, fatigue strength, and ductility [1] - [3]. Grain refinement is also observed in the processed zone, which leads to a correspondingly higher strength of the weld [4] - [7]. This also applies to aluminum materials and alloys. One of the main advantages of the process FSW is conducting welding at temperatures below the melting point of the materials, thus reducing the resulting stresses and deformations [8]. Moreover, the addition of graphene in the weld improves the properties of the processed area [9] - [13]. This research examines FSP method with addition of graphene in the zone of processing. The microhardness of the modified layer transverse to the processing direction and in debth is measured and analyzed and metallographic analysis was carried out. The remote temperature measurements in the FSP area are made using the measuring system developed at IMSETCH-BAS.

2. 2. MATERIALS AND METHODS

2.1. Experimental

The experiments were conducted with addition of graphene using an innovative technology. The graphene has purity of 99.9+%, size: 5nm, S.A:170 m²/g and Dia: 30µm. The experimental blanks are made of A5083 aluminum alloy sheet material with thickness of 10mm. A groove with width of 0.5 mm and depth of 3 mm was cut at angle 45° lengthwise on the test plate of size 150x50 mm as shown in Fig. 1. The plate was

Print ISSN 1691-5402 Online ISSN 2256-070X <u>https://doi.org/10.17770/etr2023vol3.7196</u> © 2023Radostina Zaekova, Plamen Tashev, Yasen Hadjitodorov, Deyan Gradinarov Published by Rezekne Academy of Technologies. This is an open access article under the <u>Creative Commons Attribution 4.0 International License.</u> mechanically cleaned and degreased with 5% acetone solution. The groove was filled with graphene and then pressed to seal it and to avoid loss of material during the subsequent processing.



Fig. 1. Experimental plate.

The as-prepared plate was subjected to FSP of the graphene addition zone (Fig. 2). The FSP was carried out on a HURCO VMX30i vertical machining centre. A specially designed fixture was attached on the machine table for positioning and clamping the plates on a thermo-insulation pad.



Fig. 2. FSP of the experimental plate.

The tool for FSP was designed and fabricated at IMSETCHA-BAS. It is shaped as truncated cone with length 4 mm, diameter at the base 6 mm and diameter at the tip 4 mm. A spiral groove with pitch 1 mm and three axial flutes at 120° are cut on the tapered surface. The smoothing shoulder has diameter of 13 mm. In order to better homogenize the material, the tool rotates counterclockwise.

The processing is carried out with progressive movement of the tool, first in a linear and then in a clockwise cycloid-like trajectory with offset 2 mm to the left and right from the central axis. The cycloid-like curve has radius 2 mm and pitch 2 mm.

The parameters of FSP are:

- Rotational speed 1300 rpm;
- Transverse (welding) speed 45 mm/min.

2.2. Remote measurement system of the temperature in the FSP-ed zone

A schematic view of the experimental setup for remote measurement of the temperature in FSP-ed zone is shown in Fig. 3. The processed sample (5) is positioned and clamped in the fixture. The temperature is measured with eight thermocouples type B (4) connected to an eight-channel temperature recorder (1). The thermocouples (4) are spark-welded to the FSP-ed sample (5) and their ends are insulated with glass tubes. The data from the thermocouples (4) is received, visualized, analyzed and stored on a laptop (2). The recorder (1) and the laptop (2) are connected via USB cable. The data from FSP is transmitted in real time via internal wireless network created by TeamViewer between WiFi router (3) and a remote computer, where they are visualized, processed and stored.



Fig. 3. Schematic view of the experimental setup.

The eight-channel temperature recorder Pico TC 08 (1) is shown in Fig. 4.



Fig. 4. Temperature recorder Pico TC 08.

The following software products are used:

- PicoLog 6 installed on laptop (2) to monitor the temperature of the thermocouples;
- TeamViewer installed on laptop (2) to establish a connection over the Internet as well as to visualize in real time the parameters and to and store them on the computer.

In Fig. 5 a macro section of the processed sample is shown, cut off in the place where the thermocouple monitoring the temperature is attached at a certain depth under the FSP-ed zone. The thermocouple is sparkwelded to the bottom of a hole in the opposite side to the side of FSP. The measurement location is shown in the photo with a yellow arrow.



Fig. 5. Location of the thermocouple.

The temperature was measured during the processing as the tool approaches and moves away relative to the thermocouple location and the measured values were recorded (Fig. 6).



The system for remote measurement of the temperature in the FSP-ed zone enables successful remote monitoring and control of this important parameter of the processing, as well as achieving predictable end results. The system also provides high degree of automation of the research work and easy and reliable archiving of the obtained data.

2.3. Microhardness measurements and metallography

Specimens were cut from the FSP-ed sample with added graphene and were prepared for metallographic analysis according to the standard procedure, i. e. wet grinding with sandpapers from No 400 to No 2400. The metallographic sections were developed by immersion in aqueous solution of 0.5% hydrofluoric (HF) acid. The observations with were made "PolyvarMet" metallographic microscope. The microstructure was captured using ProgRes CT3 USB digital camera with licensed software ProgResCapturePro. The analysis was conducted at temperature 24°C. The microhardness was measured with MicroDuromat 4000 microhardness tester with load 10 kg, time to reach the load 10 s and time to hold the load 10 s.

3. RESULTS AND DISCUSSION

The microhardness of the base material A5083 was measured. The measurement locations with the

respective values are shown on the metallographic section (Fig. 7). The measured values for Vickers microhardness (HV) of the A5083 base material are 85.2 кg/mm², 88.5 кg/mm² and 86.0 кg/mm². The average HV value is 86.6 kg/mm².



Fig. 7. Microhardness of the base metal.

Fig. 8 (a, b) shows the measured microhardness of the FSP-ed material and the measurement locations (pointed with arrows) in two characteristic zones:

- Stir zone with visible graphene piling in wavy parallel stripes (Fig. 8a);
- Thermo-mechanically affected zone (TMAZ) (Fig. 8b).

The measurements in both zones are made in direction transverse to the direction of FSP.



a) Stir zone



b) TMAZ Fig. 8. Microhardness of the FSP-ed material.

In the stir zone the highest microhardness value 159.3kg/mm² is measured in the vicinity of graphene stripes. In the TMAZ the measured microhardnesses are lower (85.5 kg/mm² to 90.7 kg/mm²).

The microhardness was also measured in depth in the same section, along a vertical line above the hole for thermocouple. Twenty-five measurements were taken at 240 μ m intervals. The curve of microhardness change in depth is shown in Fig. 9.



Fig.9. Curve of microhardness change in depth.

Close to the surface the measured microhardness is 119.0 kg/mm² and the maximum measured value is 120.7 kg/mm². The observed decreases of the values can be explained with the uneven distribution of graphene confirmed by the metallographic analysis. After leaving the zone limited by the pin length of the tool, which defines the depth of FSP, the hardness is approximates to that of the base material.

The microhardness on the surface of the FSP-ed zone with added graphene is also measured. The measurements were made in the area of intersections of lines 1 to 7 parallel to the direction of FSP at 2.2 mm from each other, and a line transverse to the direction of FSP. The middle line 4 is located along the axis of FSP. Microhardness was measured in three points from each area.

The values of Vickers microhardness (HV, kg/mm²) on the surface of the FSP-ed area with added graphene are graphically visualized in Fig. 10.



Fig. 10. Distribution of Vickers microhardness (HV, kg/mm²).

The analysis of the measurement results suggests that the microhardness increases when approaching the axis of the FSP zone, i.e. the axis of the graphene groove (Fig. 10). Therein are also located the points where the highest deviations from the average microhardness value are measured, which is explained both with the higher graphene concentration and its more uneven distribution proved by the wavy parallel stripes of graphene piling observed during the metallographic analysis.

Obviously, the points of the highest measured microhardnesses are shifted from the middle line 4, i.e. from the axis of FSP zone (axis of graphene groove). We suggest that this is due to the asymmetric movement of the tool which results in uneven stirring of graphene.

4. CONCLUSIONS

An innovative technology for adding graphene to aluminum alloy during FSP has been implemented. To our knowledge, there are no publications on FSP with addition of graphene using our proposed technology.

The maximum measured microhardness after FSP with added graphene is 1.8 times higher than the maximum measured microhardness of the base metal before FSP.

In the FSP areas adjacent to the graphene insertion area the reported microhardness is more than 25% higher than that of the base metal.

The largest deviations of the microhardness from the average value are observed in the areas with higher concentration of graphene and are due to the uneven distribution of graphene, confirmed by the metallographic study, which showed wavy parallel stripes of graphene.

The aims of the further research are related to both improving the technology to achieve a more homogeneous distribution of graphene and optimizing its amount.

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