Researches of metal texture after friction stir welding

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Abstract. The results of studies of the structure of an aluminum alloy by the method of backscattered electron diffraction (EBSD) are presented, which allows determining disorientation angles between grains, present data in the form of an array of angles and sizes, direct and inverse pole figures and many other types of useful information, as well as identify boundaries and sub-boundaries grains. It has been shown that friction stir welding is a process with a predominance of shear; the observed shear texture can indicate the local orientation of the material flow during welding. The three-dimensional material flow detected by these texture orientations shows important changes, especially on the advancing side of the weld.

1. Introduction

In the process of studying the structure and properties of structural materials, we can arbitrarily distinguish two fundamentally different material science methods for describing the microstructure of polycrystals. One of them consists in describing the morphology of crystallites, measuring their characteristic sizes, etc. These research methods can conditionally be called “metallographic”. Such experiments, as a rule, are carried out using optical, scanning, and, less commonly, transmission electron microscopy. The purpose of another method is to study the crystal lattice: its primary orientation (texture), internal stresses, etc.; studies are usually carried out using x-ray diffractometry and the methods used can conditionally be called "radiographic". These two ways of integral description of the microstructure for a relatively long time existed in parallel, and with their help significant progress was achieved in studying the evolution of the structure under thermal or mechanical action. Until recently, the tasks of studying texture orientation were solved by almost one single method - using transmission electron microscopy (TEM). A fundamentally new way of studying the microstructure by analyzing the patterns of diffraction of backscattered electrons (EBSD analysis) made it possible to measure its qualitatively new characteristic — disorientation of neighboring crystallites. The method developed in recent years for the automatic analysis of backscattered electron diffraction patterns (EBSD analysis) is also to study the spatial distribution of orientations. In this sense, he is a competitor to TEM. The EBSD method is increasingly used to study the microstructure of crystalline materials after various technological methods of exposure: heat treatment, welding, surfacing, pressure treatment, surface plastic deformation, etc.
Friction stir welding (FSW) is a solid-state bonding process that uses a rotating, non-consumable tool [1,2]. On the one hand, the tool serves as a source of heating of the surrounding material. On the other hand, it mixes the plastic material during rotation and forms a weld. Due to the peculiarities of the process and material flow during FSW, the weld is asymmetric with various zones clearly distinguishable under an optical microscope: mixing zone (MZ), thermo-mechanical influence zone (ZTMI), heat influence zone (HIZ) and base material zone (ZBM) [3]. During friction welding with stirring, a rotating tool deforms the surrounding material mainly due to shear, so the resulting shear strain texture stored in the weld metal can serve as an indicator for the flow of material that occurred around the tool during welding. Texture is a measure of the distribution of the orientation of the crystals in the material and may indicate the type, magnitude and orientation of the deformation. The shear deformation created by the rotating tool is oriented parallel to the surface of the tool and, thus, forms concentric stripes behind the tool, which taper from the top of the weld to the base while reducing the influence of the shoulder and reducing the cross of the tool. From numerous foreign and domestic publications, it is known that finely dispersed grains are formed in the center of the weld (core), the formation of which, according to some researchers [2,3], is the result of dynamic recrystallization [4], collective dynamic polygonization [5], or dynamic return [6]. Taking into account the specifics of the friction stir welding process, it is of practical interest to study the microstructure by analyzing backscattered electron diffraction patterns (EBSD analysis) of crystallite disorientation in the weld zone.

2. Materials and research methods
For research, we selected an industrial thermally not hardenable wrought aluminum – magnesium alloy AMg5 (foreign analog - alloy 5083) in the form of rolled plates with a thickness of 5.0 mm. A friction stir welding seam was obtained at a tool rotation speed of 1400 rpm and a welding speed of 100 mm min⁻¹. The tool geometry used for this weld consisted of a cylindrical pin with a thread and three planes, as well as a rotation arm design. Optical microscopy of a mechanically polished weld was performed in cross section, etched with Keller reagent (2 ml of HF, 3 ml of HCl and 5 ml of HNO₃ in 190 ml of water) to reveal the structure of the weld and grains. Electron microscopic studies of the aluminum surface were carried out on the basis of Irkutsk National Research State Technical University using a JIB-4501 JEOL scanning electron microscope with a multipath system equipped with a JIB-4501 electron and ion gun, complete with a nitrogen-free energy dispersive microanalysis system. The analysis of the results of reflected electron diffraction (EBSD analysis) was carried out using the software package for EBSD data processing Channel 5 developed by Oxford Instruments. For these purposes, the studied samples additionally passed the stage of electro polishing. The size of the scanning area for various samples was an area of 100 to 620 μm in size, and the scanning step was from 0.1 to μm. The grain was taken to be a region surrounded by high-angle boundaries, i.e. the value of the limiting angle was set to 15°.

3. Research results and discussion
The alloy structure in the delivery state consisted of grains partially deformed in the rolling direction, while the elongation of the grains is more pronounced for plates of smaller thickness. The average grain size was (25 ± 3) μm.

Figure 1 shows a panoramic survey of the weld, consisting of the area of the core of the weld (1) the mixing zone (MZ), the zone of thermo-mechanical influence (ZTMI) of region 2,3. In region 3, it is clearly seen that the deformation during the friction stir welding proceeds non-uniformly. This allows evaluating the degree of deformation for comparing technological modes and, in particular, to evaluate the geometry of the tool. In Fig. 1, in region 3, regions are observed with layers of the deformable material shifting relative to each other during welding. In the work [7], the adhesive-diffusion nature of the friction stir welding process was shown, when during welding, due to the adhesive-diffusion forces, the material is captured by the tool and moved behind it. In the process of rotation of the tool, the thickness of the layer of the captured material grows to a certain critical mass,
after which the material comes off the tool and the process repeats. Thus, a layered “onion” structure of the mixing zone of the welded joint is formed. These layers of material move relative to each other, and in places of their contact a local temperature increase due to friction forces is possible. Figure 1 illustrates the bands (region 3) along which the material layers moved during deformation and particles of stable secondary phases deposited on these bands under the action of high temperature.

Figure 1. Panoramic shot of the cross section of the weld of the alloy AMG-5.

The mechanism of the formation of a layered structure, as well as during sliding friction, is closely related to the temperature gradient in the surface layer of the deformable material and can be considered on the basis of ideas about the vortex nature of plastic flow. It was shown in the work [7] that such structures are formed during sliding friction as a result of competition between two processes —softening during frictional heating and hardening caused by plastic deformation. A feature of this process is its periodic nature. It is obvious that the heated and plastically deformed weld material was not significantly deformed and any observed deformation texture should reflect the mode, magnitude and orientation of the initial deformation created by the lateral surface of the tool pin. It should be noted that the results of metallographic structural analysis showed that during welding of plates of the AMg5 alloy in the core of the weld (Fig. 1, region 1), a fine-grained structure with the same grain size was formed. To some extent, the grain boundaries were decorated with precipitates of solid particles (oxides).

A detailed analysis of the weld cross section in this regard was carried out by electron backscattering diffraction (EBSD). Scanning a 300 * 600 μm plot is shown in Figure 2.

It should be noted that the study of the processes of texture formation can not be carried out separately from the analysis of the general change in the structure of the material with appropriate thermo-mechanical processing. Thus, the formation of deformation textures depends on the mechanism of plastic deformation. Certain information can be obtained by analyzing the distribution of the preferred orientations of the material in Figure 2. For visualization of orientations of structural
elements (grains), orientation maps were constructed. Such maps can be built both in the space of Euler angles and inverse pole figures Fig. 2, b. In Euler space, each point in the scanning area is assigned a specific orientation, characterized by three Euler angles, which give an unambiguous position of the unit cell in space. An alternative to the space of Euler angles is the space of inverse pole figures. In this space, each scanning point is assigned Miller indices (Fig. 2, c.), which also give an unambiguous position of the unit cell in space. At the vertices of the triangle are Miller indices (111), (101), (001). Thus, if the point (and subsequently the whole grain) on the surface of the studied material has an orientation of (111), then it is colored blue; if (001), then - in red; if (101), then in green. In the case when the point has a different orientation from the data, it is painted in the color corresponding to the legend. Figure 2 shows orientation maps for the samples under study. Figure 2a shows a contrasting image of the grain structure. The analysis of the presented data shows how uniformly the orientations in the material under study are distributed. At first glance, a chaotic distribution of grain orientation is characteristic. At the same time, after friction stir welding, regions of grain orientations of Fig. 2a and grain boundaries that gravitate toward orientation (101), which are colored green (Fig. 2, c.).

This fact is easy to explain: in bcc metals, the main slip plane is (101); therefore, the observed increase in the area of green areas on orientation maps is deformational. For more reliable discussions about the deformation mechanisms in terms of “orientations”, it is necessary to conduct a comprehensive analysis of orientation maps and pole figures (both direct and inverse). As follows from the results of Fig. 2, the average grain size decreased by 4-5 times compared to the initial grain structure of the alloy. The formation of such a structure can be explained by the intense thermo-mechanical action during friction of a rotating tool and the implementation of dynamic recrystallization under these conditions. It should be noted that there is no consensus among researchers on this issue. One of the important evidences of the dynamic recrystallization process is the determination of the disorientation angle of the formed grain boundaries, which gives a reliable idea of what type of these boundaries are - large-angle or small-angle. It is generally accepted that the high-angle boundaries characteristic of recrystallized grains includes those whose disorientation angle exceeds 15°. The backscattered electron diffraction (EBSD) method allows determining orientation angles with an accuracy of ~ 2°.

According to the data obtained, the average grain size in this zone (core) of the weld was 4.3 μm, which is in good agreement with the above results of optical metallography. From Fig. 2, d (highlighted in black) it follows that the fraction of boundaries with small disorientation angles (small-angle boundaries) amounted to about 1 % of the total number of boundaries, which allows concluding that this structure is formed mainly by equiaxed grains with high-angle boundaries (Fig. 2, g red color). The average grain size correlates well with the data obtained by optical microscopy in this zone of the weld. A distinctive feature of the plastic deformation of metals is that the deformation begins only when the external stress in the crystal reaches a critical shear stress or yield strength in the plane and in the direction in which it occurs, which corresponds to the law of critical shear stress (Schmid's law).

At the same time, out of all possible equal systems of directions and sliding planes, the first system to start functioning is the one in which the external voltage component reaches its maximum value first. With certain orientations of the crystal, the shift can begin simultaneously in several slip systems. The backscattered electron diffraction method (EBSD) allows realizing the possibility of constructing micro-stress distribution maps on the surface of the test sample. Such maps are constructed as follows: the program measures the maximum disorientation between two arbitrary points within the same grain. Then, using the mathematical “weighting” procedure, each point is assigned a specific weight value according to the maximum disorientation measured in the previous step. Then, in automatic mode, builds a smoothed map of the distribution of micro-stresses according to the average grain size of the structure of the studied material. Figure 3 shows the results of constructing orientation maps of structural elements (grains) over an area of 50 * 50 μm. The first picture shows the orientation of the grains along the (111) plane in color, it is painted in blue; (001) - in red; (101) green. Blue color and
its combinations have a large area, and the slip plane (101) green color has a smaller area. In the second picture, special twinning borders are plotted. The third picture shows microstresses in the area of grain boundaries. The maximum microstresses are fixed in fine grains.

Figure 2. Results of studying the grain structure of the AMG-5 alloy by electron backscattering diffraction (EBSD).

It should be noted that at present, with the traditional method of aluminum welding [8,9], the mechanism of releasing the weld from the oxide film is not yet fully understood. The role of cathode spots is constantly being reviewed. Many authors base their hypotheses on the basis of evaporation and cathodic cleaning mechanisms. In this regard, in friction stir welding, the role of the oxide film in the structure of the weld and the mechanism for its removal are not taken into account at all. It is quite obvious that at a metal temperature during the friction welding with stirring of 0.8 Tm, the oxide film is not removed, but mixed (moved) with the weld metal. This should affect the mechanical properties of the weld.

Figure 3. Results of the study of the grain structure of AMG-5 alloy by electron backscattering diffraction (EBSD).

In this regard, we must take this factor into account when welding, but for this it is necessary to find in micro-structural studies the areas where this film is concentrated (located). Under certain
conditions of sample preparation by electron backscattering diffraction (EBSD), it is possible to study such areas.

4. Conclusion
In conclusion, the following should be noted: the electron backscattering diffraction method (EBSD) can be used to study aluminum alloys and give a complete overview of the structure features at the micro level, and in combination with texture analysis, it allows establishing the physical mechanisms of structure formation during deformation. At present, a huge number of approaches have been developed to the task of attesting the structural state of deformed materials; however, the choice of various tools affects the results obtained by the researcher. Therefore, the correspondence to the task posed by the researcher and the means for its solution is important.

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