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Microhardness of eutectic Al-Si alloy after friction stir processing and annealing

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The plate of eutectic Al-Si alloy (AK12D) with a thickness of about 5 mm was subjected to friction stir processing (FSP). Samples for the study were cut perpendicular to the welding direction. Next, the samples were annealed for 1 h in the temperature range $150-500^{\circ}$ C, quenched in water, and subjected to natural aging for 3 months. Thermal stability of the microstructure in various zones after FSP and subsequent annealing was studied by scanning electron microscopy. The volume fractions of dendrites and thickness of the eutectic interlayers were calculated depending on the annealing temperature. It was found that in none of the zones did annealing up to $T=500^{\circ}$ C lead to a noticeable change in the microstructure of the alloy. It has been established that the temperature dependences of the microhardness of the stir zone and base material are different: for the base material it is of a "V-shaped" form with a minimum of 85 HV after annealing at $T=350^{\circ}$ C, while for the stir zone it remains constant in the range of 95 – 98 HV after annealing up to $350-400^{\circ}$ C, then increases and practically coincides with the values of the microhardness of the base material. The microhardness behavior of the stir zone is explained by the fact that the effect of FSP is similar to the effect of incomplete quenching.

Keywords: friction stir processing, annealing, microhardness, microstructure, aluminium alloy.

1. Introduction

Al-Si alloys are widely used in various industries because of their low cost, low density and ability to operate at high temperatures combined with excellent casting properties. However, in the as-cast state, Al-Si alloys exhibit low mechanical and wear-resistant properties due to the dendritic structure, irregularly distributed coarse and needle-like silicon particles, and porosity [1-4]. A promising way to improve the structure and properties of such alloys is friction stir processing (FSP), which refines the microstructure of the material, thereby improving many of its performance characteristics [4-9]. It was found that FSP led to the elimination of porosity, fragmentation of α -Al dendrites, refinement and redistribution of silicon particles, and decrease in the grain size. This results in an increased hardness, yield strength, ultimate tensile strength, ductility, wear resistance, and damping properties [10-16].

In a fatigue cracking study of a hypoeutectic cast alloy A356 subjected to FSP, it was found that at ambient temperature the fatigue life increased by almost an order of magnitude. However, with increasing temperature, the difference in durability decreased and at 200°C the fatigue characteristics became the same [17]. At the same time, FSP changes the fracture pattern of the A356 alloy from a brittle mode in the original material to a very ductile mode in the FSPed samples [14].

FSP simultaneously enhances the ductility and the ultimate tensile stress of the A390 hypereutectic alloy [18]. The simultaneous increase in ductility and strength

was explained by the formation of ultrafine recrystallized grains and the refining of silicon and secondary particles in the microstructure. In the as-cast alloy, with an increase in the strain rate, the crack propagation path changed from primary silicon particles to intermetallic and silicon eutectic particles. Ultimately, at the highest strain rate, it moved to the α -Al dendrites and was distributed along the aluminum boundaries, resulting in an intergranular fracture. Meanwhile, in the FSPed alloy the crack propagation path was mainly along the aluminum grain boundaries rather than being completely along the silicon particles [18].

Thus, FSP has the potential to locally improve the microstructure in the highly stressed regions of as-cast parts for various applications. Since many items are operated over a wide temperature range, it is very important that the changes introduced by FSP are maintained at elevated temperatures. The purpose of this work was to study the effect of annealing in the temperature range $150-500^{\circ}$ C on the microstructure and microhardness of eutectic Al-Si alloy pretreated by FSP.

2. Experimental

Commercial AK12D eutectic Al-Si alloy with a nominal composition (in wt.%) 12.6%Si, 1.91%Cu, 0.979%Mg, 0.945%Ni, 0.494%Fe, 0.353%Mn, 0.0646%Ti, 0.0026%Zr, <0.0250%Ca and 82.32%Al was subjected to FSP. The alloy was used in the form of plates with a thickness of about 5 mm. The shape and dimensions of the tool to perform FSP are described in [19]. One-pass FSP was performed under the following regime: pressure 16 MPa, linear speed of tool

movement 3 cm/min, rotational frequency 1000 rpm. After FSP, the plates were cut by an electric wire-cutting machine perpendicular to the processing direction. The cut samples were grouped in pairs. The pair included two adjacent samples. In the pair, one sample was subjected to annealing and the other served as a reference sample. The surfaces of the samples in the pairs that were on different sides of the common cut were examined. This was done in order that the compared surfaces of the samples were as close to each other as possible. Further, the FSPed samples were annealed in the temperature range of 150-500°C for 60 minutes followed by quenching in water. After annealing, the samples were subjected to natural aging at room temperature for three months. Metallographic samples were prepared by polishing with diamond pastes of different grits and colloidal suspension based on silicon oxide OP-S (Struers). Microhardness was measured by the Vickers method (HV) at a load of 0.5 N and a duration of its application of 10 s. The microhardness was measured along a straight line located at a distance of 1 mm from the contact surface of the sample with the tool shoulder. The microstructure was studied with a scanning electron microscope, Tescan Mira, in the mode of back-scattered electrons. The volume fraction of α -solid solution of silicon in aluminum was measured by the grid method, and the thickness of eutectic interlayers was measured by the directed sectioning method.

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3. Results

3.1. Microstructure

Figure 1a shows the microstructure of alloy after FSP and natural aging. Three characteristic zones can be observed: the stir zone (1), which has ultrafine grained microstructure formed as a result of dynamic recrystallization, the thermomechanically affected zone (2), characterized by elongated grains of initial as-cast microstructure and the heat affected zone (3), which retained the as-cast microstructure.

Figure 1b shows the microstructure of the sample after annealing at 500° C. No significant changes in microstructure occurred in any zone of the sample after annealing. The ultrafine grained microstructure was preserved in the stir zone. The microstructures in the heat affected zone and the base material zone after annealing in the temperature range of $150-500^{\circ}$ C practically coincide: the volume fraction of aluminum solid solution remains at 50-65%, and the thickness of eutectic interlayers is $10-20~\mu m$.

3.2. Microhardness

Figure 2 shows the microhardness distribution in the sample as a function of annealing temperature (Fig. 2 a), as well as its schematic representation (Fig. 2 b).

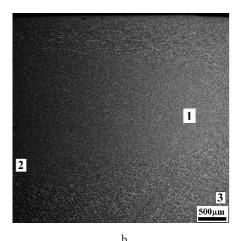


Fig. 1. Alloy Al-12.6% Si after FSP (a) and subsequent annealing at 500° C (b): $1 - \sin 2 - \sin 2$ the thermomechanically affected zone; $3 - \sin 2$ the heat affected zone.

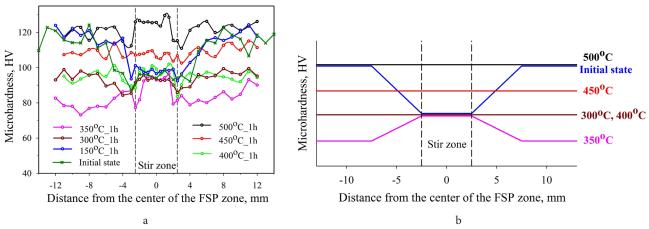


Fig. 2. (Color online) Distribution of microhardness over the sample cross section depending on the annealing temperature (a) and its schematic representation (b).

As the annealing temperature increases up to 400°C, the microhardness in the stir zone remains constant, while it decreases in the heat affected zone and base material. After annealing at 350°C, the microhardness in the heat affected zone and base material is minimal. After annealing at 400°C the microhardness of all zones becomes equal. After annealing at temperatures above 400°C the microhardness of all zones is equalized and grows, reaching a maximum after annealing at 500°C.

Figure 3 shows a comparison of temperature dependences of microhardness of the stir zone and base material. It can be seen that the temperature dependences of microhardness have different characters. In the base material, the microhardness is about 120 HV up to 150°C. Above 150°C microhardness drops, reaching a minimum of 85 HV after annealing at 350°C. As annealing temperature increases further, hardness of the base material increases monotonically up to 120 HV after annealing at 500°C. The microhardness of the stir zone behaves differently: it remains at 95-98 HV at annealing to 350 and 400°C. With further increase in annealing temperature the microhardness increases monotonically, reaching a maximum of 125 HV after annealing at 500°C. After annealing in the temperature range 400 – 500°C microhardnesses of the stir zone and base material are almost the same.

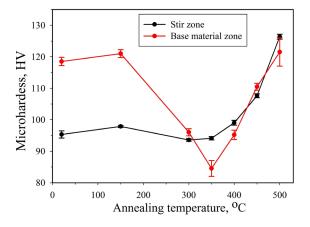


Fig. 3. (Color online) Dependence of microhardness in the stir zone and base material on the annealing temperature.

4. Discussion

Due to the presence in the composition of such elements as Cu, Mg, Ni, Fe, Mn, the AK12D alloy should be conditionally considered double. The alloy is dispersion-hardenable due to the precipitation of the following intermetallics during ageing: (Cr, Ni)₂Al₃, CuAl₂, Mg₂Si, and Q (Al₅Cu₂Mg₈Si₆) [20,21]. After annealing, the alloy underwent 3 months of natural aging at room temperature and reached a hardness of 120–125 HV. It is noteworthy that the temperature dependences of the microhardness of the stir zone and base material are significantly different. Let us consider the behavior of the microhardness of the base material zone. As the temperature increases, the microhardness decreases, reaching a minimum after annealing at 350°C. This circumstance can be explained by the fact that heating dissolves some hardening particles and enlarges others. As the

annealing temperature increases above 350°C, the remaining particles dissolve and, during subsequent natural aging at room temperature, are precipitated as smaller particles with coherent boundaries and provide higher microhardness. After annealing at 500°C and subsequent natural aging, the microhardness of the base material reaches the initial value (before FSP).

Let us now consider the behavior of microhardness in the stir zone. During FSP, due to intense plastic deformation and heating in the stir zone, there was a partial dissolution of the small particles and enlargement of the large ones. As the simulation shows, in the aluminum alloy 2024, the temperature in the FSPed area reaches the value of about 400°C in this mode of FSP [19]. It can be assumed that in the Al-12.6%Si alloy as well, the temperature in the FSPed area is at least 400°C. In this sense, FSP is similar to incomplete hardening in its effect on the microstructure. Therefore, annealing at temperatures below 350-400°C did not lead to changes in the microhardness of the stir zone, and the "plateau" of constant hardness is maintained. As the annealing temperature increases above 400°C, the particles undissolved during FSP dissolve and are precipitated again during subsequent natural aging at room temperature, providing a higher microhardness. As the annealing temperature increases above 400°C, the microhardness of the stir zone and base material are almost equal.

It is noteworthy that after annealing at 350°C the microhardness of the stir zone is about 10 HV higher than the microhardness of the base material. This can be explained by the fact that other hardening mechanisms, in particular grain boundary and dislocation hardening, contribute to the microhardness of the stir zone along with age hardening [22].

Thus, the stirred zone of the AK12D alloy is characterized by a stable ultrafine-grained microstructure up to an annealing temperature of 500°C and a stable hardness up to 400°C. This combination of properties allows the use of FSPed parts in applications requiring high thermal stability of microstructure and microhardness.

5. Conclusions

- 1. In the FSPed samples of AK12D alloy, several zones with different microstructures were found: the stir zone (zone of dynamic recrystallization) with ultrafine grained microstructure, the thermomechanically affected zone with elongated grains of the initial microstructure and the heat affected zone. The heat-affected zone and the zone of base material cannot be separated with the scanning electron microscope.
- 2. It was found that annealing to a temperature of 500°C in none of the FSP zones did not lead to a noticeable change in the microstructure. In particular, after annealing in the temperature range $150-500^{\circ}\text{C}$, the microstructure is almost identical in the heat-affected zone and the basic material: the volume fraction α -solid solution of silicon in aluminum remains at 50-65%, and the thickness of eutectic interlayers is $10-20~\mu\text{m}$.
- 3. Temperature dependences of the microhardness of the stir zone and base material have different forms. Temperature dependence of microhardness of the base material has

"V-shaped" form with a minimum at 85 HV after annealing at 350°C. In contrast, the microhardness of the stir zone remains constant at the level of 95–98 HV after heat treatment up to 350–400°C, further microhardness increases and almost equal to values of microhardness of the base material. Such a behavior of the microhardness of the stir zone is explained by the fact that the effect of FSP is similar to the effect of incomplete hardening.

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