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COMPOSITES THEORY AND PRACTICE

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APPLICATION OF FSP TECHNOLOGY IN FORMATION PROCESS OF COMPOSITE MICROSTRUCTURE IN AlZn5.5MgCu ALUMINIUM ALLOY SURFACE LAYER REINFORCED WITH SiC PARTICLES

In this study an effort was made to form a composite microstructure in the surface layer of the AlZn5.5MgCu aluminium alloy using FSP technology by introducing SiC particles to the alloy. In the experiment the multi-chamber solution was used, which consists in placing SiC powder in chambers separated one from another, cut into the modified material, perpendicular to the sample surface. To perform the friction stir processing a tool with a threaded pin was used. Evaluation of the surface treatment effects was carried out by means of both light and scanning electron microscopy. In addition EDS analysis was performed as well as a comparative measurement of hardness. The conducted examinations showed strong refinement of the of aluminium alloy structure and intense dispersion of the SiC particles in the surface layer of the material, resulting in the formation of a metal-ceramics type composite microstructure. The presence of SiC particles was found in both the stirred zone (SZ) and the thermo-mechanically affected zone (TMAZ). The consequence of the strong refinement of the microstructure and the introduction of SiC particles was a considerable increase in the hardness of the surface layer of the modified material. The performed experiment showed the effectiveness of the applied multi-chamber technology and the possibility to create a composite microstructure in the surface layer of the AlZn5.5MgCu aluminium alloy.

Keywords: friction stir processing, surface layer, composite, AlZn5.5MgCu aluminium alloy

WYKORZYSTANIE TECHNOLOGII FSP W PROCESIE KSZTAŁTOWANIA MIKROSTRUKTURY KOMPOZYTOWEJ W WARSTWIE WIERZCHNIEJ STOPU ALUMINIUM AlZn5.5MgCu WZMACNIANEGO CZĄSTKAMI SiC

W pracy podjęto próbę wytworzenia mikrostruktury kompozytowej w warstwie wierzchniej stopu aluminium AlZn5.5MgCu za pomocą technologii FSP (Friction Stir Processing) poprzez wprowadzanie do stopu cząstek SiC. W eksperymencie wykorzystano technologię wielokomorową FSP, polegającą na umieszczeniu proszku SiC w odseparowanych od siebie komorach wydrążonych w materiale modyfikowanym prostopadle do powierzchni próbki. Do przeprowadzenia obróbki tarciowej zastosowano narzędzie z gwintowanym trzpieniem. Ocenę efektów obróbki powierzchniowej wykonano za pomocą mikroskopii świetlnej i skaningowej mikroskopii elektronowej. Wykonano także analizę EDS oraz porównawczy pomiar twardości. Przeprowadzone badania wykazały silne rozdrobnienie mikrostruktury stopu aluminium oraz intensywne rozproszenie cząstek SiC w warstwie wierzchniej materiału, skutkujące powstaniem mikrostruktury kompozytowej typu metal--ceramika. Obecność cząstek SiC ujawniono zarówno w strefie wymieszania SZ (stirred zone), jak i w strefie odkształcenia termomechanicznego TMAZ (thermo-mechanically affected zone). Konsekwencją silnego rozdrobnienia mikrostruktury oraz wprowadzenia cząstek SiC był znaczący wzrost twardości warstwy wierzchniej materiału modyfikowanego. Przeprowadzone badania wykazały skuteczność zastosowanej technologii wielokomorowej i możliwość wytworzenia mikrostruktury kompozytowej w warstwie wierzchniej stopu aluminium AlZn5.5MgCu.

Słowa kluczowe: modyfikacja tarciowa z mieszaniem materiału, warstwa wierzchnia, kompozyt, stop aluminium AlZn5.5MgCu

INTRODUCTION

Increasingly more often, the decisive factor for the application potential, durability and properties of a product is the surface layer of the material it is made of. The properties of this layer are the result of intentional activities undertaken by mankind or a consequence of the processes occurring during the use of a product [1-7]. Therefore, improvement of the technologies enabling formation of the microstructure and properties of the surface layer is an issue of a particular practical importance. Friction Stir Processing (FSP) is one of the most modern technologies used in surface engineering techniques and it determines new trends in the way of forming the surface layers of metals and their alloys. The FSP technology potential is indicated not only in numerous publications and thematic seminars but also by the rapidly progressing commercialization of respective solutions [8-11]. The FSP method is derived from the welding technology with stirring of the weld material (Friction Stir Welding - FSW) developed by Wayne Thomas from the Cambridge Welding Institute in 1991, and used for joining metallic materials [12]. However, for FSP technology its main purpose is not to join materials but to modify of the microstructure and properties of the material. In FSP technology the basic parameters are: the rotational speed, travel speed, tilt angle and shape of the tool, as well as the dimensions of the tool pin, and the shoulder. These parameters are decisive in terms of the amount of heat generated during the treatment, the material microstructure and its properties [13, 14]. The heat generated during the friction of the tool is necessary to plasticize the modified material. The plasticized material is moved under the shoulder and then mixed and compacted by strain-thickening. The essential advantage of the FSP process is also the possibility to introduce an additional material during the friction modification and to form a composite microstructure in the surface layer of the modified material, and by this to obtain completely new properties that are the outcome of the properties of the individual composite components and their volume share [15-18]. In the course of the treatment using FSP technology, the melting point of the modified material is not exceeded. As a result, some modifying materials which cannot normally be used in technologies with a liquid phase due to adverse interactions between the individual composite components, can now be applied. Within this study, the AlZn5.5MgCu aluminium alloy was subjected to friction modification. This alloy is used, among others, to fabricate particularly important constructions in the automotive and aviation industry.

MATERIALS AND EXPERIMENTAL PROCEDURE

The material for the experimental examination was AlZn5.5MgCu aluminium samples with dimensions of 90x70x15 mm, cut from rolled metal sheets with the thickness of 15 mm. The chemical composition of the aluminium alloy is presented in Table 1.

TABLE 1. **Chemical composition of AlZn5.5MgCu aluminium alloy**

TABELA 1. **Skład chemiczny stopu aluminium AlZn5.5MgCu**

Alloy	Content of elements [% mass.]								
	Zn	Fe	Cu	Mn	Mg	Cr	Si	Ti	Al
AlZn5.5MgCu	5.5	0.3	1.6	0.15	2.4	0.2	0.2	0.1	rest

To form a composite microstructure in the surface layer of the alloy the multi-chamber FSP solution was used, the advantage of which, compared to other competitive solutions, results from the use of only one tool (in competing solutions two tools are required), and a distinctly reduced tendency of the modifying material to move outside of the modified zone. Unlike competing solutions, in the multi-chamber solution the powder is released from a chamber into the metallic matrix cyclically, i.e. chamber by chamber, therefore the risk of uncontrolled spilling of the powder from the modified zone is limited in one cycle to one chamber only. Before the friction stir treatment, the surface of the samples was chemically cleaned to eliminate contaminants that could affect the course of the process. In the experiment technical SiC powder with the average dimension of particles of about 30 μm and a fragmented and multifaceted shape was used. The powder was subjected to comminution in a FRITSCH Pulverisette 6 planetary mill. The comminution process lasted 1 h. The average dimension of the particles after milling was about 4 μm. Then the SiC powder was placed in chambers with the diameter of 2 mm and depth of 4.5 mm cut into the aluminium alloy samples. The thickness of the walls separating the adjacent chambers was constant and amounted to about 0.3 mm. Figure 1 illustrates an exemplary sample after filling the chamber with the SiC powder.

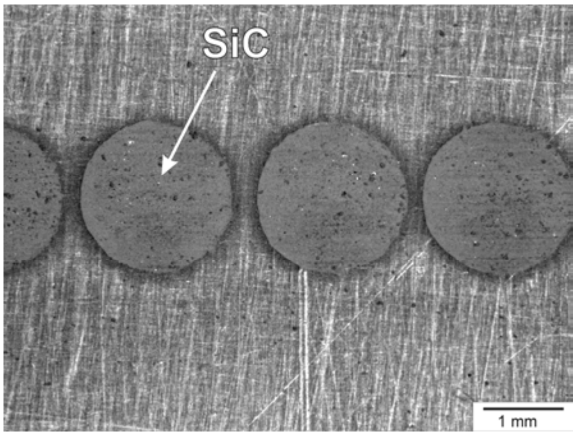

Fig. 1. Sample after filing chambers with SiC powder

Rys. 1. Próbka po napełnieniu komór proszkiem SiC

The tool was made of X37CrMoV5-1 tool steel for hot operations and consisted of a shoulder with the diameter of 18 mm and a pin in the form of a cone with a threaded side surface (Fig. 2a). The stirring pin was 4.5 mm long. During the treatment the operating tool was positioned perpendicular to the sample surface by 2 degrees from the axis. The diagram of the friction treatment is presented in Figure 2b. The friction modification was carried out by means of a CNC vertical milling machine, which made it possible to move the sample in 3 dimensions: X, Y, and Z. The tool travel speed was 40 mm/min, and the rotational speed of the operating tool was 400 rpm. Macroscopic examinations were carried out by means of an Olympus SZ61 stereo microscope, whereas microscopic examinations were performed by means of an Olympus GX41 optical microscope and a JEOL JSM-6610LV scanning microscope. The EDS examinations were carried out using an X-ray microanalyzer EDS Oxford Instruments, operating together with the JEOL JSM-6610LV scanning electron microscope. The hardness measurement was performed by means of a Shimadzu HMV-G20 microhardness meter provided with a Vickers penetrator. The applied load was 980.7 mN and the load time amounted to 10 s.

a)

b)

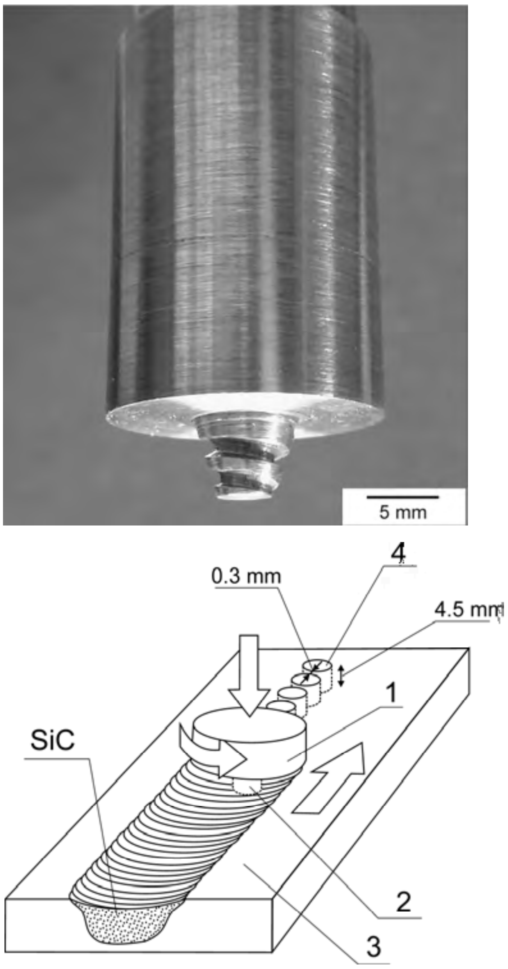

Fig. 2. Shape of tool used in FSP treatment (a), diagram of friction stir welding/processing technique (b); 1 - operating tool, 2 - pin, 3 - region of friction stir treatment, 4 - chambers filled with SiC powder

Rys. 2. Kształt narzędzia wykorzystanego w obróbce tarciowej (a), schemat obróbki tarciowej (b), 1 - narzędzie, 2 - trzpień, 3 - materiał poddany modyfikacji tarciowej, 4 - komory napełnione proszkiem SiC

RESEARCH RESULTS

Figure 3 shows the surface image of the friction stir treatment region with characteristic parallel grooves, which indicate the direction and way of plasticized material movement under the shoulder of the operating tool. The total width of the stirred material is approximately equal to the shoulder diameter of the tool, i.e. about 18 mm.

a)

b)

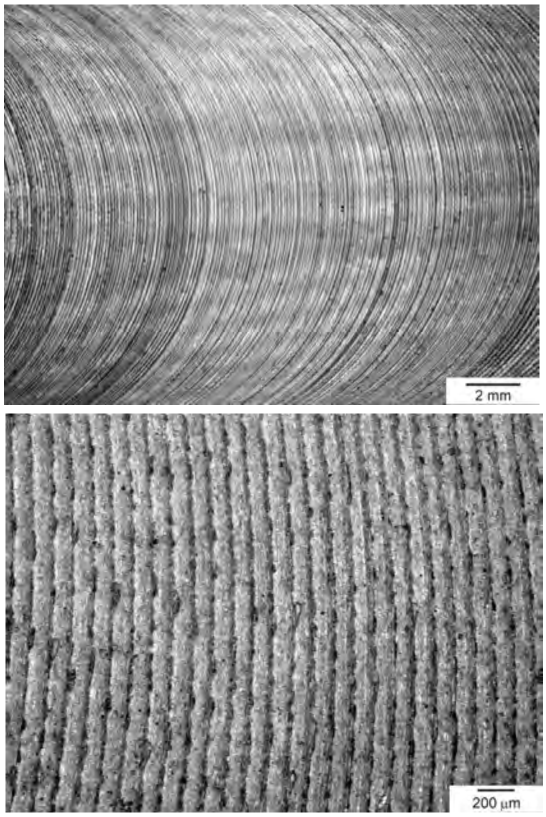

Fig. 3. Image of examined aluminium alloy surface in region of tool working at two different magnifications

Rys. 3. Powierzchnia stopu aluminium w miejscu oddziaływania narzędzia zobrazowana przy różnych powiększeniach

The examination by means of optical microscopy revealed considerable changes in the microstructure of the surface layer of the modified material compared with the state before treatment. The changes occurred not only as the consequence introducing SiC ceramic particles into the plastified matrix but also due to the fact that the features observed in the microstructure of the initial material disappeared. Examples of the microstructure of the AlZn5.5MgCu aluminium alloy in the initial state are shown in Figures 4a and 4b, and after friction modification in Figures 4c and 4d. It can be noted that this aluminium alloy in its initial state had a typical microstructure after rolling with evident elongated grains and numerous precipitations of intermetallic phases situated parallel to the rolling direction and giving the material banding features. The banding of the intermetallic phase precipitations against the background of the grains can be seen particularly in Figure 4a. Moreover, as can be noted, the location of the intermetallic phases did not result from the location of the grain boundaries.

The effect of the banding and rolling structure present in the as-delivered material were completely removed due to the friction modification. Furthermore, in the modified material some zones were found, which are typical for materials subjected to FSP treatment, i.e. the evident prevailing stirred zone (SZ) and the thermo-mechanically affected zone (TMAZ) as well as a heat affected zone (HAZ) adjoining the base material (BM).

Unlike the stirred zone where very fine and in a vast majority equiaxial grains prevailed, in the thermo-mechanically affected zone grains of elongated shapes dominated. The microstructure in the stirred zone proves that dynamic recrystallization of the material occurred. This process is a consequence of strong deformation of the material and an effect of the high temperature that accompanies the treatment. It is worth adding that the material heats up to a temperature ranging between 70÷90% of the material melting point, and the source of the heat is the friction process only. During the friction treatment, no liquid phase comes into being, and only plasticizing of the material occurs. Thus, alternations in the modified material are those in the solid phase only. What is more, the characteristic feature of the stirred zone is the formation of a metal-ceramics type composite microstructure with evenly dispersed SiC particles in the aluminium alloy matrix. An above average share of SiC particles which would prove insufficient stirring of the individual composite components, was not found anywhere in the SZ or TMAZ. Moreover, the adverse effect of contacting adjacent SiC particles was found only in a few places. During the microstructural examinations it was found that the average share of SiC particles in the thermo-mechanically affected zone is almost twice as low as in the stirred zone, and the location of the SiC particles in the TMAZ reflects the direction of the movement of the plasticized material during the treatment. The average share of the strengthening phase was: for the SZ - about 8.5%, for the TMAZ 4.8%.

The characteristic feature of the surface layer is strong refinement of the material microstructure. The average size of the grains in the stirred zone was about 4 μm. The grain size is the consequence of the extent of the heat effect arising due to the friction of the stirring tool. The higher the heat effect, the faster material plasticization occurs, but the material microstructure becomes less refined.

The EDS analysis of the precipitations present in the aluminium alloy showed a few types of intermetallic phases, i.e. those rich in aluminium, iron, copper and zinc (Fig. 5a), precipitations rich in aluminium, silicon, zinc, and copper (Fig. 5b), and precipitations rich in aluminium, copper and zinc. It is worth mentioning that the composition of those precipitations did not change after the friction modification, whereas the way and the place of their layout changed. Although the location of the precipitations in the initial material was typical for rolled materials, the location of the precipitations in the material subjected to the FSP process was more even, without any clear orientation or banding effect.

The changes in the microstructure of the surface layer of the aluminium alloy triggered by the friction modification and introduction of an additional material into the aluminium matrix suggested the possible occurrence of considerable changes in the material properties. The measurements of the material hardness confirmed this assumption. The introduction of hard ceramic SiC particles into the surface layer of the aluminium alloy and very strong refinement of the microstructure of the aluminium alloy triggered by the friction modification caused a considerable increase in the materia hardness.

a) b) c) d)

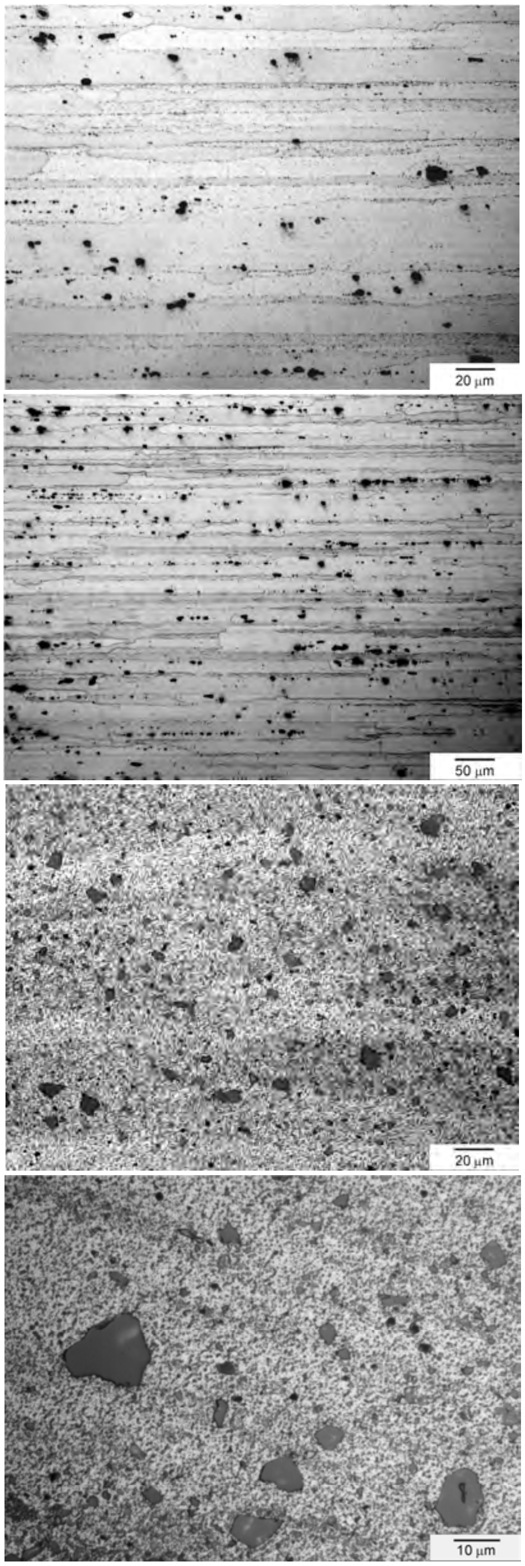

Fig. 4. Microstructure of aluminium alloy in its initial state (a, b) and after friction modification (c, d); etched metallographic microsection

Rys. 4. Mikrostruktura stopu aluminium w stanie wyjściowym (a, b) oraz po modyfikacji tarciowej (c, d), zgład metalograficzny, trawiony

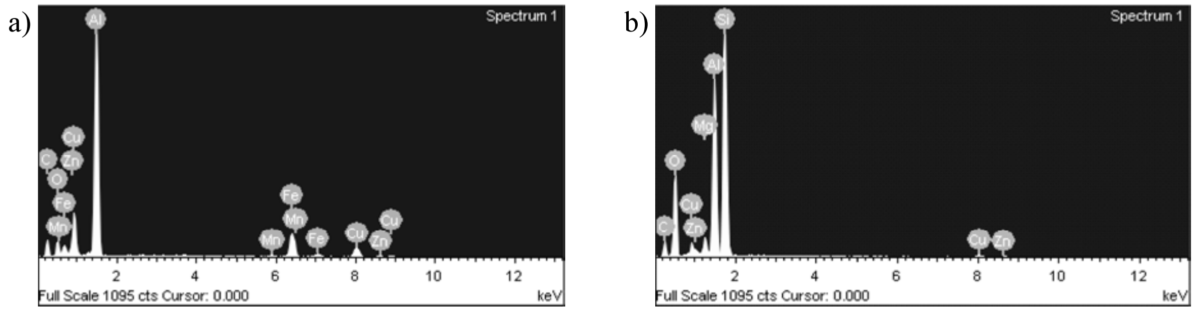

Fig. 5. Results of EDS analysis of precipitations present in aluminium alloy

Rys. 5. Wyniki analiz EDS wydzieleń obecnych w stopie aluminium

An exemplary distribution of the material hardness versus the distance from the surface and the course of changes in the hardnesses measured at the depth of about 3 mm (the total depth of the microstructural changes was about 5÷6 mm) along a line parallel to the sample surface is illustrated in Figures 6a and 6b.

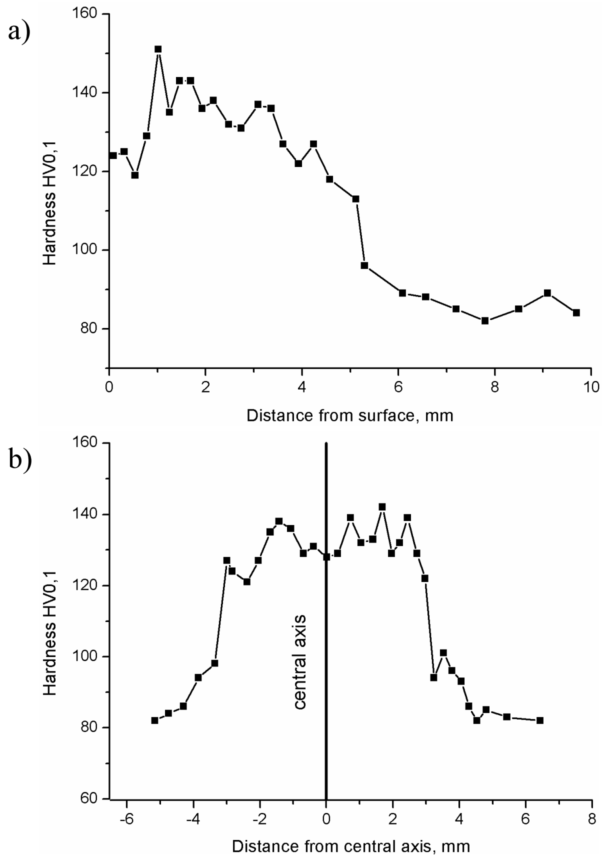

Fig. 6. Course of Vickers microhardness: a) in depth of aluminium sheet after FSP treatment, b) distribution of microhardness at depth of 3 mm against distance from central axis of FSP region; see text

Rys. 6. Przebieg zmian twardości w funkcji odległości od powierzchni modyfikowanej (a), przebieg zmian twardości na głębokości 3 mm w funkcji odległości od centralnej osi obszaru modyfikowanego (b), opis w tekście

The average hardness of the initial material was about 84 HV0.1, whereas the average hardness of the modified material measured in the stirred zone was about 136 HV0.1, which means that it was higher by about 65%. In the subsurface layer the average hardness was lower than in the remaining part of the modified zone and it amounted to about 125 HV0.1, which was caused by a lower share of the strengthening phase in this place.

CONCLUSIONS

Based on the carried-out examinations, the following statements and conclusions were formulated:

1. The use of the multi-chamber solution enables the formation of a metal-ceramics type composite microstructure in the surface layer of the AlZn5.5MgCu aluminium alloy.
2. As a result of the friction stir modification, intensive dispersion of the SiC phase in the stirred zone occurs.
3. During the friction modification, dynamic material recrystallization takes place, leading to the formation of very fine, equiaxial grains in the stirred zone.
4. The consequence of the strong refinement of the microstructure and the introduction of hard SiC particles is a considerable increase in the hardness of the surface layer of the aluminium alloy.

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