



REGULAR ARTICLE

Role of Grain Boundary Strengthening for Increasing the Microhardness of Aluminum Alloys Irradiated by High-Current Pulsed Electron Beam

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The study features the fact shown on the example of a number of aluminum-based alloys (D16AT, 1933 and AA6111) that irradiation with high-current pulsed electron beam (HCPEB) with particle energy of 0.35 MeV, a beam current of 2.0 kA, a pulse duration of 5  $\mu$ s and a beam diameter of 3 cm leads to the formation of a surface layer with a modified structural-phase state. The formation of submicrocrystalline structure with a grain size of less than 1  $\mu$ m is one of the main features of this state. The microhardness of the modified layer for all alloys increases by more than 30%. Based on these studies, the contributions of grain-boundary strengthening to the increase in microhardness of the HCPEB-remelted surface layer of the studied alloys were calculated. It is shown that grain-boundary strengthening is the key factor responsible for the higher microhardness of the surface layers of the studied alloys treated with HCPEB.

**Keywords:** High-current pulsed electron beam, Aluminum alloys, Microhardness, Grain-boundary strengthening.

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1. INTRODUCTION

Aluminum alloys are the most common structural materials right after steel [1]. They are an important construction material not only in aircraft construction but also in the nuclear industry. They are used for producing seamless tubes for reactors and centrifuges used for uranium enrichment, in particular. Therefore, studies aimed at finding new ways to increase the surface strength of aluminum alloys and creating new technological processes for their treatment are relevant.

Recently, high-current pulsed electron beams (HCPEB) have drawn much attention in the field of surface modification of materials [2-5]. In the process of HCPEB-irradiation, high energy ( $10^8$ - $10^9$  W/cm<sup>2</sup>) is applied to the material surface in a very thin layer (a few micrometers) over a short period of time (a few microseconds) while causing ultrafast heating and cooling, uniform melting, vaporization and solidification on the treated surface. Dynamic stress fields arising in the course of these processes can cause intense and ultrafast deformation processes in the material surface layer. The formation of a relatively thick modified surface layer, usually with a submicrocrystalline structure, which has a strong metallurgical bond with the un-treated part (substrate), is considered to be a significant advantage of HCPEB-treatment over other conventional surface

treatment methods.

Structural and phase transformations in metals and alloys caused by the influence of HCPEB shall lead to changes in their strength properties and, above all, their microhardness. It shall be mentioned that the challenge of describing the hardening of polycrystals is very complicated and is far from being solved. Two stages of hardening induced by HCPEB irradiation, namely: surface and subsurface hardening are considered [6, 7]. Surface hardening is the hardening in the beam-remelted top layer. Subsurface hardening is the hardening of the interlayer that follows the upper re-melted layer. The use of HCPEB with different energy parameters leads to the fact that the thickness of the re-melted layer can vary from a few micrometers to 100  $\mu$ m or more. From the point of view of industrial applications, in the case of a sufficiently thick (100  $\mu$ m or more) re-melted layer, it is the fact that this layer becomes stronger than the original material that is important. A number of mechanisms that may be involved in the hardening of the surface HCPEB-modified layer are currently under discussion [8]. However, the fact regarding which of these mechanisms is determinant is still debatable. Based on a number of studies, where both alloys and pure metals were irradiated [7, 9-11], it can be concluded that grain-boundary strengthening, which is implemented due to the formation of a finer-grained structure in this layer, is

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one of the main mechanisms for increasing the microhardness of the HCPEB-re-melted layer. For example, in the study [7], where the Mg-4Sm-2Al-0.5Mn alloy was irradiated, it was shown that the microhardness of the surface re-melted layer increased almost 2-fold mainly due to grain refinement.

The aim of this study is to explore the changes of grain structure in the HCPEB-re-melted surface layer re-melted, as well as to study the changes of microhardness values in the initial and irradiated states on the example of a number of matrix aluminum alloys. The other aim is to calculate the contributions of grain-boundary strengthening to the increase in microhardness of HCPEB-re-melted surface layer of the studied alloys based on these studies, and draw conclusions about the role of grain-boundary strengthening in the overall hardening of HCPEB-re-melted layers.

## 2. EXPERIMENTAL PROCEDURE

Irradiation of alloy sheets was performed by a high-current pulsed electron beam at the TEMP-A accelerator in the NSC KIPT of the NAS of Ukraine [12-14]. The energy flux density at the W target is approximately  $10^9 \text{ W/cm}^2$  (beam energy is  $E \sim 0.35 \text{ MeV}$ , current is  $I \sim 2000 \text{ A}$ , pulse duration is  $\tau_i \sim 5 \cdot 10^{-6} \text{ s}$ , beam diameter is  $D \sim 3 \text{ cm}$ ). Irradiation was produced by a single impulse in a vacuum at  $1.3 \times 10^{-3} \text{ Pa}$ .

Microstructure studies were performed using an Olympus GX51 optical microscope and a SEM Tescan VEGA 3 LMH, a scanning electron microscope. The average grain size  $d$  was determined from microphotographs using the random secant method.

The Vickers microhardness was measured at room temperature in air using the PMT-3 microhardness tester. Microhardness tested with load of 50 g and load duration of 25 s.

## 3. MATERIALS

Industrial aluminum alloys AA6111, 1933 and D16AT were chosen as research materials in this study. The complete chemical compositions of the alloys under study are given in Table 1. In all three investigated aluminum alloys 4 elements, i.e. 3 metals (Mg, Cu, Zn) and semiconductor Si are used as the main alloying elements. Their introduction in relatively large quantities is possible as they have a sufficiently large solubility in aluminum in the solid state. The main function of alloying elements is to increase the strength of aluminum. Strengthening by standard thermomechanical treatment methods is achieved by solid solution formation as well as by dispersion hardening. The main impurities present in all aluminum alloys, iron and manganese, tend to degrade mechanical and corrosion properties. All these alloys have relatively high specific strength and at the same time are sufficiently technological for manufacturing of deformed semi-finished products. The use of different heat treatment modes makes it possible to control the complex of their service characteristics in a wide range in relation to the

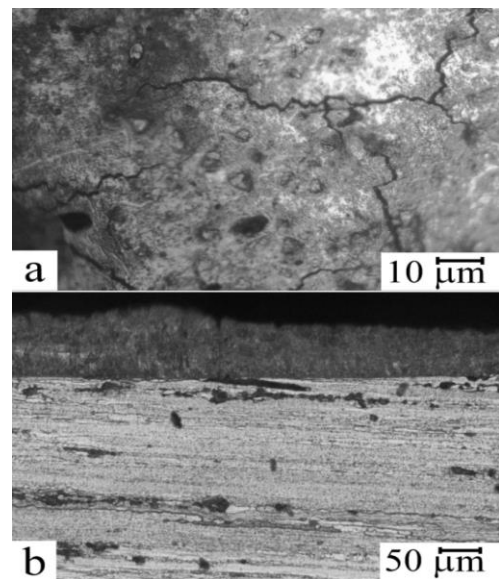
required operating conditions. All this opens up great prospects for processing these alloys with HCPEB to improve their strength surface properties.

**Table 1** – Chemical Compositions of Alloys (Weight Per-cent)

| Alloy  | Mg      | Cu  | Si  | Zn  | Mn  | Al  |
|--------|---------|-----|-----|-----|-----|-----|
| AA6111 | 0.5-1.0 | 0.8 | 0.8 | 0.1 | 0.2 | Bal |
| 1933   | 1.6-2.2 | 1.1 | 0.1 | 6.8 | 0,1 | Bal |
| D16AT  | 1,2-1,8 | 4,7 | 0,5 | 0,2 | 0,6 | Bal |

## 4. RESULTS AND DISCUSSION

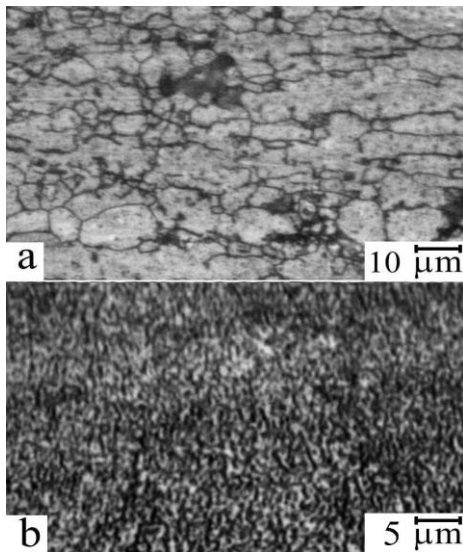
When the plates of the studied alloys are exposed to HCPEB, the surface layer with a thickness of the order of the electron path in the material is rapidly heated to the melting point. Moreover, the electron beam provides localization of the maximum value of the absorbed energy at the depth, which is approximately 1/3 of the electron path in this substance [15]. Since the plate heating rate is greater in the deeper layers, this leads to explosive ejection of part of the molten material and its subsequent rapid cooling by heat dissipation into the main volume of the target. Cooling is accompanied by crystallization of molten material with changed structural-phase state [12-14]. As a result, the properties of the surface layer change. As an example, Fig. 1a shows a fragment view of the irradiated surface of D16 alloy, and Fig. 1b shows a cross-sectional view of a D16 alloy plate after HCPEB treatment. Fig. 1a shows that the surface of the remelted layer is covered with a network of cracks. The upper ball in Fig. 1b corresponds to the remelted layer. The average depth of the remelted layer is approximately 100  $\mu\text{m}$ . This section of the plate was pre-polished and sanded and then etched using an all-purpose etchant.



**Fig. 1** – View of a fragment of the surface of HCPEB-irradiated D16AT alloy (a); view of a cross-sectional section of D16AT alloy in the electron beam treatment zone (b)

As is known [7, 9-11], irradiation with HCPEB creates conditions that can lead to dispersion of the newly formed grain structure. Therefore, the grain structure of the initial and irradiated samples of the studied alloys will be described in detail below.

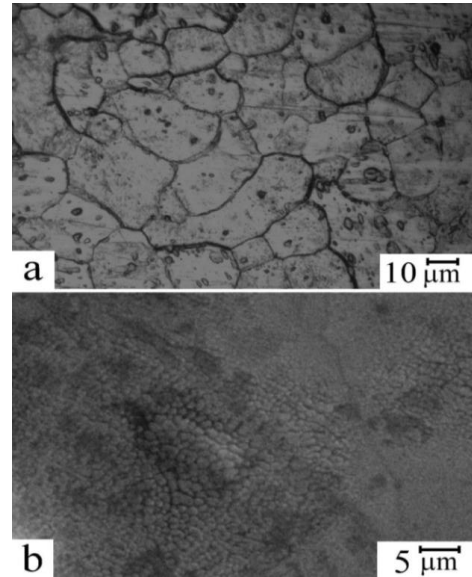
The grain microstructure of D16AT alloy in the initial state is shown in Fig. 2a. It can be seen that the grain structure is almost completely recrystallized. The grains are mostly close to equiaxed, however, there is a slight variation in grain size. The average grain size is 11  $\mu\text{m}$ . It was established that treatment of D16AT alloy with HCPEB leads to grain refinement in the surface irradiated layer. The average grain size in the re-melted layer is approximately 0.8  $\mu\text{m}$  (Fig. 2b). The structure of the modified layer, according to the blurred contrast of grain boundaries, is non-equilibrium.



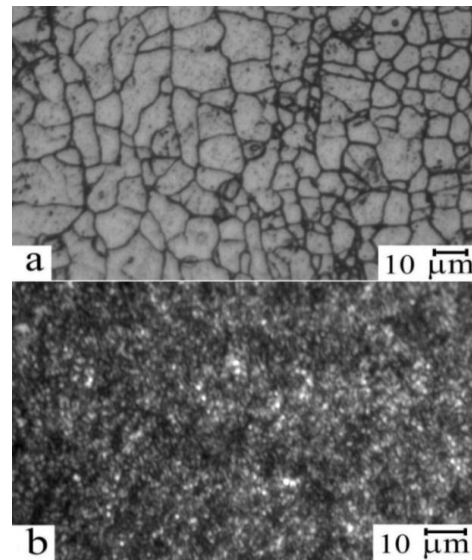
**Fig. 2** – View of original grain microstructure of D16AT alloy (a); view of microstructure of HCPEB-remelted layer of D16AT alloy plate (b)

Fig. 3a shows the initial microstructure of the AA6111 alloy. The average grain size of AA6111 alloy was determined to be approximately 40  $\mu\text{m}$ . Grains have different sizes, but there is no distinct metallographic texture. Fig. 3b shows a view of the grain microstructure of the modified surface layer of this alloy. Comparison of Fig. 3a and Fig. 3b indicates a sharp difference between the grain microstructure of the re-melted layer and the microstructure in the original state. The average grain size of the re-melted AA6111 alloy layer was determined to be 0.7  $\mu\text{m}$ .

Fig. 4a shows the initial grain microstructure of 1933 aluminum alloy. It was determined that the grain size of the initial microstructure of 1933 alloy had a grain size of approximately 15  $\mu\text{m}$ . The structure of the HCPEB-modified surface layer of the studied 1933 alloy, which is shown in Fig. 4b, is dramatically different from the base metal and is characterized by small submicron grain size and non-equilibrium of the structural state.



**Fig. 3** – View of initial of AA6111 alloy (a); view of microstructure of HCPEB-re-melted layer of AA6111 alloy plate (b)



**Fig. 4** – View of initial of 1933 alloy (a); view of micro-structure of HCPEB-re-melted layer of 1933 alloy plate (b)

Fig. 5 shows the ratio of microhardness of the studied alloys in the initial state and after irradiation. The microhardness of the D16AT alloy in the initial state was of approximately 101HV0.50. The microhardness of the HCPEB irradiation-modified layer of D16AT alloy increased significantly to approximately 147HV0.50. Thus, it was established that there is a hardening of the surface layer of aluminum alloy D16AT, which is characterized by an increase in microhardness by 50 % compared to the initial state, as a result of HCPEB-exposure. It was also determined that microhardness values drop after re-melted layer in the heat affected zone of the beam. However, these values are still higher than the microhardness values in the initial state.

Microhardness values correspond to the average value of microhardness in the initial state of the alloy only at a distance of 200 micrometers from the alloy surface.

The microhardness of the AA6111 alloy in the initial state is a 70HV0.50. The microhardness of the irradiated layer of AA6111 alloy is approximately 101HV0.50. Thus, hardening occurs as a result of exposure of the surface layer of aluminum alloy AA6111 with a pulsed electron beam. It is characterized by an increase in microhardness by more than 30 % compared to its value in the initial state of the alloy. After the re-melted layer in the heat affected zone of the beam, the microhardness values decrease. However, these values are still slightly higher than the microhardness values in the initial state. Microhardness values get closer to the average value of microhardness in the initial state of the alloy only at a depth of more than 300 micrometers from the alloy surface.

The microhardness of the 1933 alloy samples examined in this study in the initial state is 105HV0.50. It was determined that the grain size is approximately 0.8  $\mu\text{m}$  for the HCPEB-modified layer of 1933 alloy. The microhardness of the HCPEB-irradiated layer of 1933 alloy is 137HV0.50. Thus, it was established that HCPEB exposure results in surface hardening of 1933 aluminum alloy, which is characterized by an increase in microhardness of the layer re-melted by irradiation by more than 30 % compared to the initial state. The effect of annealing on the microhardness of this alloy was investigated earlier in study [16]. It was determined that after annealing the original sample at 520  $^{\circ}\text{C}$  for 2 hours, the HV value was 56. That is, the heat treatment results in the de-strengthening of the alloy under study. The observed decrease in micro-hardness value happens apparently due to two main factors: dissolution of secondary phase particles and increase in average grain size up to 23  $\mu\text{m}$ .

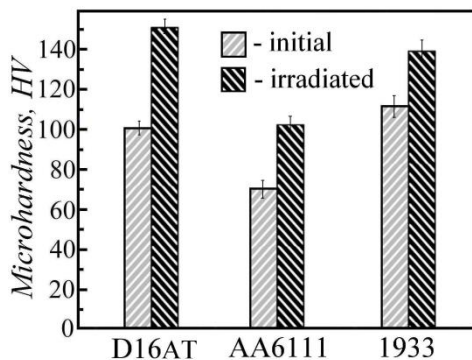


Fig. 5 – Microhardness of alloys in initial and irradiated states

Plastic deformation in aluminum and its alloys in the course of microhardness determination shall be carried out mainly due to dislocation movement only. However, the physical causes leading to hardening are, generally speaking, quite diverse [17, 18]. At the same time, the presence of grain boundaries in the polycrystalline aggregate has a significant effect on hardening. When

dislocations meet an obstacle on their way, their sliding can no longer be continued as before. The constraints imposed on the plastic deformation of the grain by differently oriented neighboring grains occur at grain boundaries. Therefore, the sliding of dislocations in one grain cannot freely continue in its neighboring grain due to the mismatch of crystallographic systems. Consequently, the distance that a mobile dislocation can move before reaching a grain or crystallite boundary decreases with decreasing grain size, resulting in hardening. This type of hardening is called grain-boundary strengthening.

Hardness is the resistance of a material to local plastic deformation resulting from the insertion of a harder body into it. The hardness value can be used for estimating the strength and ductility of a material. Knowing the microhardness of an alloy, one can estimate its other mechanical properties as well. There are ways to approximate the tensile strength and yield strength of a material based on microhardness data [17, 18]. According to [17], the Vickers microhardness value is equivalent to the true stress at 8 % strain and shall be determined using the following equation

$$\sigma_s(\text{MPa}) = 3.27HV \quad (1)$$

For aluminum-based alloys alloyed using Mg, Si and Cu conversion from yield strength  $\sigma_y$  (in MPa) to microhardness  $HV$  (in VPN) can then be done via a simple regression formula [18]:

$$HV = 0.33\sigma_y + 16.0 \quad (2)$$

It is known that the conditional yield strength in polycrystalline materials is related to grain size, and the boundaries are effective barriers for moving dislocations. The finer the grain size, the greater the extent of the boundaries and hence the more often they occur on the path of moving dislocations. According to the Hall-Petch relationship [19], the contribution of grain-boundary strengthening  $\sigma_{GB}$  to the yield strength can be defined as:

$$\sigma_{GB} = Kd^{-1/2} \quad (3)$$

where  $K$  – grain-boundary strengthening coefficient or Hall-Petch constant [18],  $d$  – average grain size.

The values  $\sigma_y$  and  $\sigma_{GB}$  for D16AT, 1933 and AA6111 alloys examined in this study were calculated using relations (2) and (3). Experimentally obtained values of grain size and microhardness of the studied alloys were taken for calculations. The value of grain-boundary strengthening coefficient  $K = 0.10 \text{ MPa} \times \text{m}^{1/2}$  was taken from study [20], where this coefficient was obtained for aluminum-based alloys alloyed with copper and magnesium.

Table 2 shows the values of  $\sigma_y$  obtained from calculations for the D16AT, 1933 and AA6111 alloys examined in this study in the initial and irradiated states. The values of  $\sigma_{GB}$  contributions to the strengthening of these alloys in the initial and irradiated state are also given in this table.

**Table 2** – Values  $\sigma_y$  and  $\sigma_{GB}$  for D16AT, 1933 and AA6111 Alloys in Initial and Irradiated States

| Alloy  | $\sigma_{GB}$ , MPa |            | $\sigma_y$ , MPa |            |
|--------|---------------------|------------|------------------|------------|
|        | initial             | irradiated | initial          | irradiated |
| AA6111 | 16                  | 120        | 152              | 257        |
| 1933   | 26                  | 100        | 270              | 367        |
| D16AT  | 30                  | 112        | 326              | 452        |

The analysis of the results given in Table 2 shows the leading role of grain-boundary strengthening mechanism in increasing the overall hardening of the re-melted layer of the studied alloys. The whole increase in the  $\sigma_y$  value in the irradiated state of the alloys can be obtained due to the increase in the  $\sigma_{GB}$  value. As is known, according to [18] the contribution to the overall hardening of the solid-solution hardening mechanism also increases in the irradiated layer, since the aluminum-based solid solution is enriched with alloying elements. However, this increase occurs due to a reduced contribution to the overall hardening of the dispersion mechanism, since the vast majority of intermetallic phase particles dissolve in the course of irradiation. All this suggests a dominant role in increasing the strength of the HCPEB-re-melted layer precisely due to the mechanism of grain-boundary strengthening.

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## 5. CONCLUSION

It was established that HCPEB-treatment of the surface of D16AT, 1933 and AA6111 aluminum alloys leads to the formation of a surface layer about 100  $\mu\text{m}$  thick with a modified structural-phase state, the distinctive feature of which is the presence of submicrocrystalline grain structure with an average grain size of 0.7  $\mu\text{m}$  for AA6111 alloy and 0.8  $\mu\text{m}$  for D16AT and 1933 alloys, whereas in the initial state the grain size of all alloys exceeded the value of 10  $\mu\text{m}$ .

It was established that the surface layer of D16AT, 1933 and AA6111 aluminum alloys is strengthened as a result of the action of HCPEB. It is characterized by an increase in its microhardness by more than 30 %.

The contribution of grain-boundary strengthening to the overall hardening of the surface HCPEB-irradiated layer of the studied alloys was calculated. It was concluded that it is the grain-boundary strengthening mechanism that is dominant in increasing the strength of the HCPEB-re-melted layer of these alloys.

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**Роль зернограничного зміцнення в збільшенні мікротвердості алюмінієвих сплавів, опромінених сильнотрумовим імпульсним пучком електронів**В.В. Брюховецький<sup>1</sup>, В.В. Литвиненко<sup>1</sup>, Д.Є. Мила<sup>1</sup>, О.Л. Рак<sup>2</sup><sup>1</sup> *Інститут електрофізики і радіаційних технологій НАН України, 61002 Харків, Україна*<sup>2</sup> *Національний науковий центр «Харківський фізики-технічний інститут» НАН України, 61108 Харків, Україна*

У роботі на прикладі ряду сплавів на основі алюмінію (Д16АТ, 1933 і АА6111) показано, що опромінення сильнотрумовим імпульсним пучком електронів з питомою енергією 0.35 MeV, силою струму 2.0 кА, тривалістю імпульсу 5 мс та діаметром пучку 3 см призводить до формування поверхневого шару з модифікованим структурно-фазовим станом. Однією з головних особливостей такого стану є утворення субмікрокристалічної структури з розміром зерна меншим за 1 мкм. Мікротвердість модифікованого шару для всіх сплавів збільшується більш ніж на 30 %. На підставі цих досліджень було розраховано вклади зернограничного зміцнення у збільшення мікротвердості переплавленого поверхневого шару досліджуваних сплавів. Показано, що саме зернограничне зміцнення є ключовим фактором, відповідальним за більш високу мікротвердість поверхневих опромінених шарів сплавів.

**Ключові слова:** Сильнотрумовий імпульсний електронний пучок, Алюмінієві сплави, Мікротвердість, Зернограничне зміцнення.