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Treatment of alumina ceramics by intense electron and ion beams

The paper investigated modification of the microstructure of the surface layers of alumina ceramics under exposure to electron and ion beams. Electron beam irradiation was performed at accelerating voltage U = 15 kV and beam current of J = 70 A and J = 100 A. Ion irradiation was performed with carbon ions at accelerating voltage of U = 180 keV. The current density and energy density varied in the range of 15–85 A/cm^2 and 0.3–1.5 J/cm^2, respectively. The amount of energy acting on the ceramic surface depended on the number of pulses N. It is shown that exposure to electron and ion beams changes the microstructure of the irradiated ceramic layer. In general, the effect of exposure is similar for electron and ion irradiation, and it is characterized not only by surface melting, but also by formation of a finer microstructure through the depth of the irradiated layer, which is oriented in the direction of the electron and ion beam exposure. It is shown that crystallization processes in overheated layers of ceramics depend on its type and melting point.

Keywords: electron and ion beams, alumina ceramics, microstructure.

Introduction

Ceramic materials based on alumina ceramics are used in various industries [1–3], including the telecommunications, automotive, aerospace, and electrical engineering industries. This type of ceramics is also used for wear-resistant parts of mechanisms, as an abrasive material for machining, etc. A variety of applications of alumina ceramics are popular due to a combination of its physicomechanical, optical, and chemical properties. It is known that all the combined properties of finished ceramic materials depend on technological conditions of manufacture, their microstructure and surface condition [4–6].

Alumina ceramics is widely used as a dielectric substrate for production of hybrid integrated circuits in the electronics industry [7, 8]. Substrates for integrated circuits are expected to ensure low energy losses, show physical stability and chemical resistance under high-temperature effects, catalytic and adsorption capacity, and be compatible with other materials used for manufacturing microstrip lines, capacitors, resistors and contact pads [7]. The quality of the substrate surface is one of the crucial factors in integrated circuit manufacturing. Therefore, increased demands are placed on the surface quality of such substrates.

The final stage in substrate manufacturing is mechanical grinding and polishing. This type of treatment of the substrate surface is very expensive and can have a destructive effect; moreover, polishing should be performed in several stages to achieve the required surface quality, which increases the cost of the final product.

An alternative and effective technique to improve the quality of the surface of materials is exposure to low energy high current pulsed electron (LEHCPEB) [9–12] and ion beams [13–16], and laser processing [17, 18]. These types of radiation treatment were developed mainly for metals and alloys [14, 19–21], however treatment with concentrated energy of accelerated particle fluxes can be successfully used for oxide ceramics. This type of treatment allows high-precision processing of ceramic parts of complex shapes within short time of surface exposure; it does not destruct ceramics, improves its condition and changes its properties. Alternative treatment techniques can be employed to control the surface properties and significantly change the catalytic and adsorption capacity of ceramic substrates.

When choosing the type of radiation treatment it is important to know the benefits of each of the types. Therefore a comparative analysis of the results of electronic and ion treatment for a specific type of ceramics becomes relevant. This comparison was performed for zirconia ceramics [22]. The results of this study cannot be extrapolated to corundum ceramics due to significant differences in the properties of corundum and zirconia ceramics. Therefore great practical relevance of corundum ceramics necessitates a study of its treatment with powerful electron and ion beams.

Thus, the aim of the study is to compare the effect of high-current pulsed electron and ion beams on modification of the microstructure of the alumina ceramic surface.

Experimental

The test samples were commercial VK-94-1 grade substrates made of vacuum-tight alumina ceramics (α-Al_2O_3) produced in Russia in standard sizes of 60×48 mm and 1 mm thick and intended for hybrid integrated circuits. The alumina ceramic substrate was unilaterally polished. For the experiment bulk ceramic substrates were cut into samples not larger than 1 cm^2 in size using a diamond-tipped tool. The polished surface of the prepared samples was exposed to electron and ion beams.

The samples were exposed to LEHCPEB using the SOLO accelerator developed at the Institute of High Current Electronics (IHCE SB RAS, Tomsk) [23]. Irradiation was performed with accelerating voltage U = 15 kV, beam current J = 70 and J = 100 A, pulse duration t = 100 µs, pulse repetition rate f = 0.3 Hz, and the number of pulses N = 5 and 10.

Ion irradiation was carried out using the TEMP-4M accelerator that allows irradiation in a pulsed mode [24]. Irradiation was performed with C ions at accelerating voltage U = 180 keV. The pulse duration (at half maximum of the accelerating voltage) was 100 ns. The pulse repetition rate was 8 s^{-1}. Three irradiation modes were employed: mode A, j = 15 A/cm^2, W = 0.3 J/cm^2, N = 100; mode B, j = 50 A/cm^2, W = 1 J/cm^2, N = 100; mode C, j = 85 A/cm^2, W = 1.5 J/cm^2, N = 25. The amount of energy W acting on the test sample surface depended on the number of pulses N. The number of pulses was chosen to make the ceramic samples resistant to the energy impact and to prevent their complete melting and failure.

The effect of electron and ion irradiation on the microstructure of alumina ceramics was studied with regard to the irradiation mode.

The microstructure of the test samples before and after ion irradiation was investigated by scanning electron microscopy using a Hitachi TM-3000 microscope equipped with a backscattered electron detector. Metal or graphite coatings with a thickness of not more than 100 nm were applied to the sample surface to remove the electric charge from the ceramics and improve the quality of the resulting image. The microstructure of the subsurface layers by depth was studied on ceramic sample cleavages prepared after irradiation.

Results and Discussion

Fig. 1 shows SEM images of the surface of unirradiated alumina ceramic samples. The SEM image *a* of the sample surface was taken before etching, which was carried out to visualize the grain structure. The SEM image *b* of the sample surface was taken after thermal etching with simultaneous argon ion etching. The sample was etched using an ILM-1 ion implanter (Yekaterinburg, Russia) equipped with a Pulsar-1M ion source based on a low-pressure glow discharge with a cold hollow cathode [25]. Etching was carried out in vacuum with argon ions with energy of U = 30 keV and ion current density j = 300 µA/cm^2. Under ion irradiation grains can be visualized due to different rates of ion etching of the grain volume and grain boundaries. The etching rate at the grain boundaries is higher due to their greatest defectiveness. The surface exhibits a granular ceramic structure with distinct grain boundaries (Fig. 1 b). The grain sizes vary from 10 to 60 µm. SEM images (Fig. 1) show pores and defects randomly distributed over the surface.

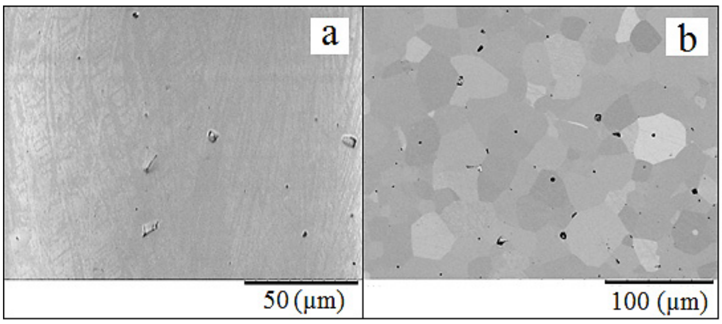

Figure 1. Surface of alumina ceramics before irradiation (top view):
a) before etching; b) after etching with argon ions.

A powerful local effect of LEHCPEB as a result of rapid heating and cooling causes modification of the subsurface ceramic layers (Fig. 2). Fig. 2 a shows that exposure to LEHCPEB at J = 70 and N = 10 causes melting of the subsurface ceramic layers. As a result, the surface becomes developed and cracked at the sites exposed to LEHCPEB. In this case, no clear grain structure can be observed. Exposure to LEHCPEB at J = 100 and N = 5 leads to structuring of the subsurface ceramic layers (Fig. 2 b). In these irradiation conditions a cellular structure made of 5–8 μm individual blocks separated by cracks is formed on the surface. Smaller formations caused by recrystallization are found inside the blocks. As can be seen in Fig. 2 b after recrystallization grains acquire the shape of elongated rectangles.

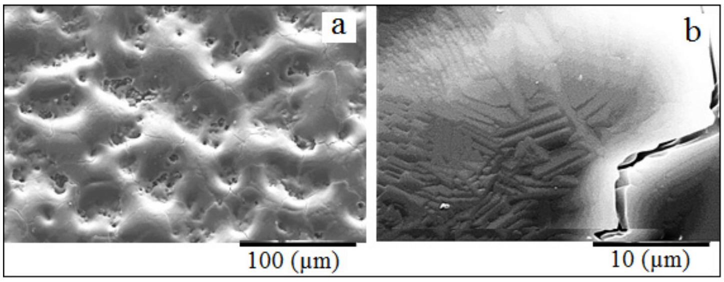

Figure 2. Surface of ceramics after exposure to LEHCPEB (top view):
a) beam current J = 70, N = 10; b) beam current J = 100, N = 5.

Fig. 3 shows the SEM image of a transverse cleavage of ceramic samples exposed to ion irradiation. When the surface of the alumina ceramics is irradiated in mode A (j = 15 A/cm^2, W = 0.3 J/cm^2, N = 100), no melting or recrystallization occurs. As can be seen (Fig. 3 a), irradiation in mode B (j = 50 A/cm^2, W = 1 J/cm^2, N = 100) causes not only surface melting, but also formation of a finer microstructure through the depth of the irradiated layer elongated in the direction of the ion beam exposure. Irradiation in mode C (j = 85 A/cm^2, W = 1.5 J/cm^2, N = 25) leads to a continuous melted layer not more than 15 μm thick. This irradiation mode causes the formation of cracks through the entire thickness of the melted layer. It should be noted that an increase in the ion current density and power density does not increase the thickness of the melted layer, the surface erosion is likely to occur simultaneously with the fusion.

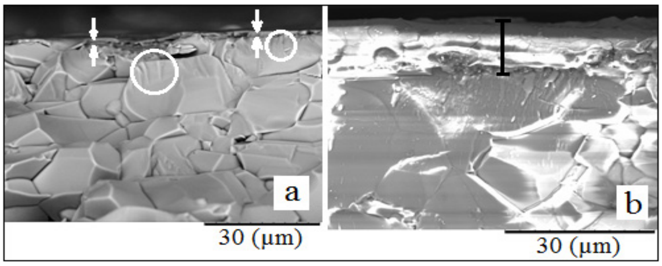

Figure 3. Transverse cleavage of ceramics after ion irradiation (side view): a) mode B; b) mode C.

In general, the nature of the effect of electron and ion beams on corundum ceramics is similar to that reported for zirconia ceramics [22]. Some differences observed are due to the structure and properties of corundum ceramics. This is primarily the nature of crystallization during melt cooling after pulsed radiation exposure. As can be seen in Fig. 2 a, crystalline blocks not more than 10 μm in size are formed in corundum during crystallization. These blocks are oriented in the direction of the ion beam exposure. In zirconia ceramics [22] recrystallization was observed strictly in a thin subsurface layer, and the newly formed blocks were elongated from the surface deep into the sample. In our opinion, these differences are due to the lower melting point of corundum compared with zirconia ceramics.

Conclusion

The comparative analysis of the effect of low energy high current pulsed electron and ion beams on alumina ceramics revealed the following:

– The nature of changes in the microstructure of the analyzed ceramics is almost similar for both electron and ion irradiation. The key factor is the irradiation power density. It is obvious that the rate of melting and cracking can be reduced using an appropriate irradiation mode.

– Irradiation changes the microstructure of the subsurface ceramic layers. In this case, the subsurface layers will be either molten or structured depending on the irradiation type and mode. The nature of crystallization in the volume of the fused layer strictly depends on the melting temperature of the oxide material. The subsurface layer structure becomes denser due to the decreased porosity in the molten layer and recrystallization into finer grains.

The results obtained confirm that low energy high current pulsed electron and ion beams can be effectively employed to modify the surface of ceramic materials, including corundum.

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С.А. Гынгазов, В.А. Костенко, С.В. Матренин, А.И. Купчишин

Алюмооксидті керамиканы қарқынды электронды және ионды шоқтармен өңдеу

Электронды және иондық шоқтардың әсерінен алюминий оксидті керамикасының беткі қабаттарының микроқұрылымының модификациясы зерттелді. Электрондық сәулелермен сәулелендіру U=15 кВ үдетілген кернеуде, шоқ тогы J 70 және 100 A кезінде жүргізілді. Ионды сәулелендіру U=180 кэВ үдетілген кернеуінде көміртек иондарымен іске асырылады. Ток тығыздығы мен энергия тығыздығы сәйкесінше 15-85 А/см^2 және 0,3-1,5 Дж/см^2 аралығында болды. Керамика бетіне әсер ететін энергия мөлшері N импульстарының санымен өзгерді. Электронды және иондық сәулелердің әсері сәулеленген керамика қабатының микроқұрылымында өзгерістер тудыратыны көрсетілді. Жалпы алғанда әсер ету нәтижесі электронды және иондық сәулелену үшін бірдей, әрі бетінің балқуымен ғана емес, сонымен қатар электронды және иондық сәулелердің әсер ету бағытына бағытталған кіші микроқұрылымның сәулеленген қабатының тереңдігімен сипатталады. Керамиканың қыздырылған қабаттарындағы кристалдану процестері оның түріне және балқу температурасына байланысты екендігі көрсетілген.

Кілт сөздер: электронды және иондық шоқтар, алюмооксидті керамика, микроқұрылым, керамика қабаты.

С.А. Гынгазов, В.А. Костенко, С.В. Матренин, А.И. Купчишин

Обработка алюмооксидной керамики интенсивными электронными и ионными пучками

Исследована модификация микроструктуры приповерхностных слоев алюмооксидной керамики под действием электронных и ионных пучков. Облучение электронными пучками проводилось при ускоряющемся напряжении U=15 кВ и токе пучка J 70 и 100 А. Ионное облучение осуществлялось ионами углерода при ускоряющемся напряжении U=180 кэВ. Плотность тока и энергии варьировались в диапазоне 15–85 А/см^2 и 0,3–1,5 Дж/см^2 соответственно. Количество энергии, воздействующей на поверхность керамики, изменялось числом импульсов N. Показано, что воздействие электронными и ионными пучками вызывает изменения микроструктуры облученного слоя керамики. В целом, результат воздействия одинаков для электронного и ионного облучений и характеризуется не только оплавлением поверхности, но и формированием по глубине облученного слоя более мелкой микроструктуры, которая ориентирована по направлению воздействия электронных и ионных пучков. Показано, что кристаллизационные процессы в перегретых слоях керамики зависят от ее типа и температуры плавления.

Ключевые слова: электронные и ионные пучки, алюмооксидная керамика, микроструктура, слои керамики.

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