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Calculation of the volume of nano- and microstructures formed on the surface of PbTe during ion-plasma treatment using machine learning

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Abstract. The formation of porous Pb nano- and microstructures on the surface of PbTe films during low-energy ion-plasma treatment has been studied using scanning electron microscopy (SEM) combined with machine learning-based image analysis. PbTe epitaxial films with (111) crystallographic orientation were exposed to argon plasma at an ion energy of approximately 25 eV for varying durations (60–240 s). SEM imaging at a tilt angle of 70° enabled three-dimensional size estimation of the formed structures, which, together with automated image processing using the DLgram01 deep learning service, allowed for precise calculation of particle number, area, height, and volume. In this paper, a comparative analysis of the parameters of the Pb structure on the surface of lead telluride films with the orientation (111) and PbTe single crystals with the orientation (100) is carried out. This study demonstrates the effectiveness of machine learning for quantitative analysis of surface nanostructures after low-energy argon plasma treatment of PbTe surface.

Keywords: argon plasma, lead telluride, nanostructuring, volume of material

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Материалы конференции
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Расчет объема нано- и микроструктур, образующихся на поверхности PbTe при ионно-плазменной обработке с использованием машинного обучения

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Аннотация. Формирование нано- и микроструктур на поверхности пленок PbTe при низкоэнергетической ионно-плазменной обработке изучалось с помощью сканирующей электронной микроскопии (СЭМ) в сочетании с анализом изображений на основе машинного обучения. Пленки PbTe с кристаллографической ориентацией (111) подвергались воздействию аргоновой плазмы при энергии ионов около 25 эВ при вариации времени (60–240 с). СЭМ-визуализация под углом наклона 70° позволила провести трехмерную оценку размеров сформированных структур, что вместе с автоматизированной обработкой изображений с использованием сервиса глубокого обучения DLgram01 позволило точно рассчитать количество частиц, площадь, высоту и объем. В данной работе проведен сравнительный анализ параметров структур Pb на поверхности пленок теллурида свинца с ориентацией (111) и монокристаллов PbTe с ориентацией (100). Данное исследование демонстрирует эффективность машинного обучения для количественного анализа поверхностных наноструктур после обработки поверхности PbTe низкоэнергетической аргоновой плазмой.

Ключевые слова: плазма аргона, теллурид свинца, наноструктурирование, объем материала

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Introduction

The study of processes forming nano- and microstructures on the surface of semiconductor materials is of significant interest both from a fundamental and applied perspective. Among promising materials for nanostructuring, lead telluride (PbTe) occupies a special place due to its unique physical properties and wide application in infrared optics and thermoelectric devices [1–2]. It is known that when PbTe is treated in argon plasma, surface sputtering occurs primarily due to the removal of lead telluride molecules, accompanied by subsequent redeposition of lead in the form of quasi-spherical nanostructures [1]. Previous studies have shown that at ion energies close to the sputtering threshold (~25 eV), the sizes of such structures increase with treatment duration. However, most studies were limited to analyzing morphological characteristics (shape, density, particle diameter), while a quantitative assessment of the deposited material volume—essential for understanding growth mechanisms and material redistribution—was not conducted. Such data are crucial for modeling self-organization processes and optimizing technologies for creating functional coatings.

The aim of this work was to develop a methodology for quantitatively assessing the volume of nano- and microstructures formed on the surface of PbTe films during ion-plasma treatment. To automate SEM image analysis and obtain accurate data on the number, size, and spatial distribution of structures, a machine learning approach was used with the DLgram01 service [3, 4].

Materials and Methods

(111) PbTe films, 2 μm thick, were grown by molecular beam epitaxy on (111) CaF_2/Si substrates. Samples were treated for durations of t = 60, 120, 180, and 240 seconds in a dense argon plasma reactor with high-frequency inductive discharge (13.56 MHz, 800 W, 0.15 Pa) at an ion energy of ~25 eV [5]. To investigate the role of PbTe crystallographic orientation in sputtering processes, similar treatments were performed on the surface of (100) PbTe crystals, as described in [1]. Surface morphology after ion-plasma treatment was analyzed using a Zeiss Supra-40 scanning electron microscope. Automated SEM image processing, including determination of the number, size, and area of 2D nanostructures, was performed using machine learning [4] with the DLgram01 service [3]. This network was trained on previously obtained images, which were manually marked as a test sample. After that, a model was created on the markup data in the service to determine this type of particles. Using SEM images of nanostructures obtained at a 70° tilt angle, their 3D dimensions were determined, allowing for the calculation of the deposited material volume and the study of quantitative parameters of surface structures depending on treatment time and crystallographic orientation.

Results and Discussion

Analysis of the (111) PbTe film surface after plasma treatment showed that hemispherical nanostructures formed after 60 and 120 seconds of exposure (insets in Fig. 1, *a*, *b*). At t = 180 s, both hemispherical nanostructures and larger quasi-spherical structures appeared (inset in Fig. 1, *c*). Analysis of SEM images using the DLgram01 service helped determine particle sizes and densities. With increasing treatment time, the average diameter of nanostructures increased, while their density decreased (Table 1). Based on SEM images of nanostructures obtained at a 70° tilt angle (insets in Fig. 1), the heights of the formed nanostructures were determined, and particle size distribution histograms were constructed. Using these data, considering particle shapes, the total volume of lead particles on the surface (equal to the sum of all particle volumes over an area of 9.35×10^5 nm^2) and the corresponding equivalent film thickness (ratio of particle volume to investigation area) were calculated (Table 1).

Based on SEM image analysis, it was found that with increasing treatment time of (111) PbTe films in argon plasma, not only did the average diameter of nanostructures increase, but their shape also changed from hemispherical to teardrop-like and partially merging. This may be related to coalescence and material redistribution processes under the influence of the ion flux. These changes became especially pronounced after 180 seconds of treatment, when the active formation of large aggregates began. Analysis of the dependence of particle volume on time showed linear growth up to 180 seconds. Changes in particle shape led to a stepwise increase in volume, possibly indicating pore formation [6] or changes in the redeposition mechanism, such as increased local concentration of Pb vapor on the surface or changes in adatom mobility due to sample heating caused by prolonged argon plasma exposure.

a) *b*) *c*)

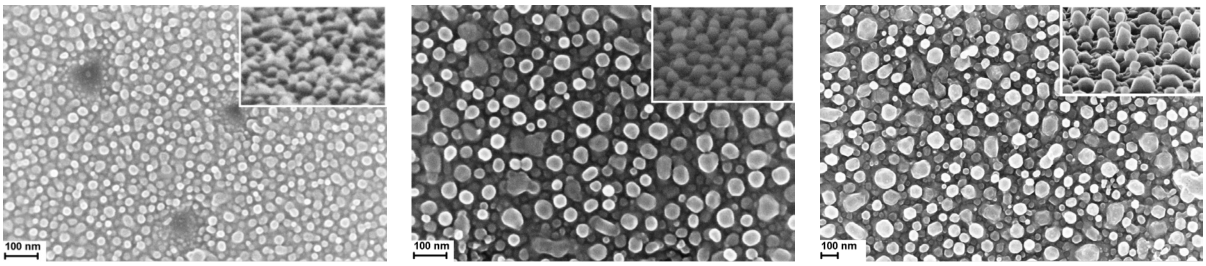

Fig. 1. Images of (111)PbTe films surface after treatment for 1-3 min (*a*–*c*, respectively)
The insets show SEM images of the structures obtained at 70°

Table 1

Characteristics of nano- and microstructures after plasma treatment with different times from area 9.35×10^5 nm^2

Orientation	(111) PbTe	(111) PbTe	(111) PbTe	(111) PbTe	(100) PbTe
Plasma treatment time, s	60	120	180	240	240
Particle density, cm^{-2}	$9.6 \cdot 10^{10}$	$4.4 \cdot 10^{10}$	$3.1 \cdot 10^{10}$	$1.1 \cdot 10^{10}$	$7 \cdot 10^9$
Diameter, nm	15-25	20-35	25-50	50-75	65-85
Average particle height, nm	20±2	25±3	40±4	60±6	78±8
Volume of particles, nm^3	$2.3 \pm 0.2 \cdot 10^6$	$7.0 \pm 0.6 \cdot 10^6$	$1.1 \pm 0.1 \cdot 10^7$	$2.1 \pm 0.2 \cdot 10^7$	$2.7 \pm 0.3 \cdot 10^7$
Equivalent Pb thickness, nm	2.5±0.4	7.5±1.1	12.2±1.7	22.5±3.2	28±3.9

a) *b*)

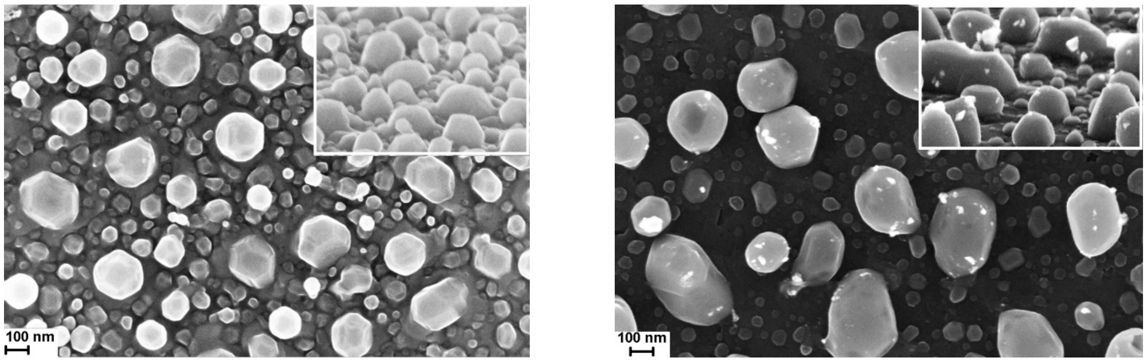

Fig. 2. Comparison of PbTe surface morphology after 240 s treatment for (111) (*a*) and (100) (*b*) orientations

Surface analysis of PbTe after 240 seconds of argon plasma treatment revealed significant differences in the nature of nano- and microstructure formation depending on the crystallographic orientation of the sample. Fig. 2 shows the surface morphology for single-crystal PbTe samples with (111) and (100) orientations. For (100) PbTe (Fig. 2, *b*), redeposition occurred more actively, and structure sizes increased. As seen from SEM images (Fig. 2), well-formed, predominantly hemispherical structures uniformly distributed across the surface were observed on the (111) film. In contrast, on the (100) film, larger and more heterogeneous formations appeared, including individual teardrop-like structures and their aggregates. At higher magnifications, pores were visible on the surface of such structures.

According to Table 1, the average structure diameter on the (111) film was 50–75 nm, while on the (100) film, it increased to 65–85 nm. A more significant difference was observed in particle height: the average height for the (100) orientation reached 78 nm compared to 60 nm for (111). The difference in volumes was minor since the density for (100) was 1.7 times lower: the particle volume on the (100) crystal was $\sim 2.7 \times 10^7$ nm^3 over an area of 9.35×10^5 nm^2, which was 20% higher than for the (111) film ($\sim 2.1 \times 10^7$ nm^3).

The equivalent lead layer thickness for the (100) orientation was 28 nm, compared to 22.5 nm for (111). This is because the sizes of large aggregates on (100) were much larger than on (111), although their density was 1.7 times lower.

The obtained data indicate that the crystallographic orientation of lead telluride plays an important role in the formation of morphology and structure volume during ion-plasma treatment, which should be considered when purposefully creating functional nanostructured surfaces.

Conclusion

The particle volume calculation method, based on automated SEM image processing using machine learning with the DLgram01 service, demonstrated high accuracy for analyzing structures formed on single-crystal PbTe surfaces during ion-plasma treatment. Changes in the size, shape, and structure of lead particles on the PbTe surface at constant ion energy, process duration, and crystallographic orientation are associated with varying sputtering rates, surface temperature, and the formation of lead particles on the treated surface.

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