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OBTAINING AL NANOLAYERS AND CRYSTAL STRUCTURE

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Abstract- Thin aluminum layers were obtained on a glass substrate. The thermal spraying method under vacuum was used to obtain thin layers. Nanopowder with a particle size of d = 40-60 nm was taken as the starting material. Depending on the operating mode, thin Al layers with h = 15, 20 and 40 nm thickness were obtained. The crystal structure of thin layers of various thicknesses has been studied. The crystal structure of the thin layer was compared with the crystal structure of powdered Al nanoparticles. The studies were carried out by X-ray diffraction at room temperature. It was found that as the layer thickness increases, a crystal structure begins to form.

Keywords: Crystal Structure, XRD Analysis, Thin Films.

1. INTRODUCTION

The development of nanotechnology has led to the development of electronics and spintronics. Purchasing functional materials in small sizes is essential for purchasing new transducers. Therefore, research continues on the synthesis of known materials at the nanoscale. It has been determined that nanomaterials exhibit different physicochemical properties [1-5]. Obtaining and examining thin metal layers opens up possibilities for their applications in aerospace technology. During the study of the thin layer of Fe obtained on the GaAs substrate, it was found that the magnetic properties change depending on the thickness of the Fe layer and the influence of the substrate and temperature. As the thickness of the Fe layer increases, the magnetic properties approach the magnetic properties of iron [6].

In the case of thin layers, changes are observed in the electrical and magnetic properties of the materials, as well as in their optical properties. In the thin layers of $Au/Au/Cu_2O$ and Cu/Cu_2O, the optical properties change depending on the thickness of the metal layer, and this is observed with the increase of the photocurrent [7].

Among nanomaterials, Al nanoparticles have a special importance. It was determined that atoms on the surface of aluminum nanoparticles combine with oxygen and water to form aluminum oxide and aluminum hydroxide [8, 9]. It is known that an oxide layer, which acts as a coating, forms on the surface of many materials [10, 11]. Therefore, examining thin layers of material gives a realistic picture of them.

It is known that under the influence of external factors (temperature, pressure, radiation, etc.) there are changes in the properties of materials. Therefore, the effects of these influence must be studied. It has been found that a single-phase system is formed in Al nanoparticles under the influence of high-intensity electron beam. Thus, under normal conditions, the compounds of aluminum oxide and aluminum hydroxide formed by Al nanoparticles decompose under the influence of electron beams. As a result, a single-phase system consisting of pure Al nanoparticles is obtained [8,9]. It is known from radiation materials science that under the influence of ionizing radiation, various structural changes are observed in the system, such as amorphization and the formation of defects. However, the results of the irradiation of Al nanoparticles with electron beams show that the ionizing rays do not cause structural defects in these particles, but rather the process of chemical purification. This effect was observed during irradiation up to $\Phi = 1.03\times10^{18}$ cm^{-2}.

The above studies have shown that the size of materials affects their physical properties. There are two methods for studying the effects of nano-dimensions. One of them is the study of powdered samples of materials. Another method is to study thin layers of material. Each of these methods has different characteristics. The study of thin layers is of greater scientific and practical importance. Because the possibilities of applying thin layers are wide. These layers are used in modern electronics to obtain various converters.

The structure and thermophysical properties of Al nanoparticles are well studied. However, the structure and physical properties of thin layers are still insufficiently studied. In this work, thin aluminum layers were obtained on a glass substrate by vacuum thermal spraying. The crystal structure of thin layers of various thicknesses was studied by X-ray diffraction at room temperature.

2. EXPERIMENTAL PART

2.1. Obtaining Thin Layers

Thin layers are obtained from Al nanoparticles d = 40-60 nm in size (Nanoparticles were ordered from SkySpring Nanomaterials, through the website: www.ssnano.com. A thin Al layer of three different thicknesses was obtained by thermal spraying on a 25×19 mm glass substrate. The experiments were carried out on a Leybold-Herause L-560 vacuum setup (P = 2×10^{-5} mbar). Before thermal spraying, the surface of the glass substrates was ionized with a power of 800 W. To improve the pollination process, the glass substrate was heated to 100°C in a vacuum chamber. The pollination process was carried out for 25 seconds.

2.2. XRD Study of Samples

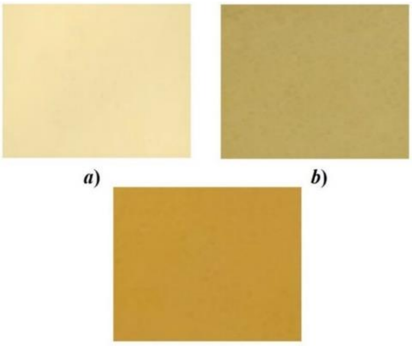
The crystal structures and crystallographic parameters of the samples were studied by the X-ray diffraction method. Spectra were obtained on a D8 Advance (Bruker, Germany) XRD diffractometer at room temperature. During the experiments, the parameters of the diffractometer corresponded to the following values: 40 kV, 40 mA, CuKα - radiation, λ = 1.5406 Å. The obtained spectra were analyzed in the Fullprof and Origin programs and the crystallographic parameters were determined. The Ritveld method was used to analyze the spectra. This method studies the crystal structure more accurately by determining the atomic coordinates as well as the parameters of the elementary lattice. By determining the coordinates of the atoms, it is also possible to calculate the values of the distances between the atoms and the angles between the bonds.

3. RESULTS AND DISCUSSIONS

In these studies, aluminum nanomaterials were studied in the form of thin layers. Al nanoparticles in powder form with particle size d = 40-60 nm were used to obtain thin layers.

Thin aluminum layers of were obtained by thermal spraying on glass substrates. The base is made of a chemically cleaned glass substrate measuring 25×19 mm. Before thermal spraying, the surface of the glass substrate was ionized with a power of 800 W. To improve the pollination process, the glass substrate was heated to 100°C in a vacuum chamber. Thermal spraying was carried out for 25 seconds. The most suitable modes were selected to obtain layers of different thicknesses. Images of thin Al layers (a, b and c) with thickness h = 15, 20 and 40 nm are shown in Figure 1.

As can be seen from Figure 1, the color of the samples gets darker as the thickness of the aluminum layer increases. The reason for this is that as the number of atomic planes in the layers increases, the optical properties change, and accordingly, the proportion of light reflected and absorbed from the samples also changes [12-15]. These properties are related to the structural properties of the material. Therefore, it is important to study the structural aspects of the formation of the resulting thin layers.



***a*)** ***b*)** ***c*)**

Figure 1. Image of thin layers of Al h = 15, 20 and 40 nm (a, b and c) thickness taken on a glass substrate

It can be seen from the thin layers of aluminum that they change color as their thickness increases. This is due to the fact that the optical properties also change depending on the thickness of the Al nanoparticles. As the thickness decreases, the transparency of the layer increases. As the thickness of the layer increases, the absorption of light rays in the system increases. Therefore, in the samples shown in Figure 1, the colors of the sample surface differ as the thickness changes.

When studying nanomaterials, it was found that their physical and chemical properties are related to their structure at the atomic level. As the dimensions of the structure change, so do the physical properties. The structural properties of thin layers are mainly the surface structure. However, the study of the crystal structure is also important in the formation of these layers. Because, when studying the crystal structure, it is possible to determine the mechanism of the phase formation process depending on the thickness of the layer. It is known that the X-ray diffraction method (XRD) for the study of crystal structure is a unique method for studying the crystal structure of solids [16-19]. The crystal structures of thin layers of aluminum have also been studied by the XRD method at room temperature and under normal conditions. In order to determine the process of phase formation in these layers, the obtained X-ray diffraction spectra were compared with the X-ray diffraction spectra of aluminum.

The crystal structure of Al nanopowders and Al thin layers with h = 15, 20 and 40 nm (a, b and c) thickness obtained on glass substrates was investigated by XRD. The spectra were obtained in the interval $10° \leq 2\theta \leq 70°$. XRD diffraction spectra obtained at room temperature are shown in Figure 2.

During the analysis of spectra in the Fullprof program, it was determined that the crystal structure of Al nanopowders corresponds to the cubic symmetry (Fm-3m singony). The lattice parameters were determined: $a = b = c$ = 4.0518 Å, V = 66.52 Å^3.

X-ray diffraction spectra obtained at room temperature were analyzed by the Ritveld method and crystallographic parameters were determined. The crystal structure was determined by determining the atomic coordinates. It has been found that Al atoms form a highly symmetrical crystal structure by forming metal bonds.

Four aluminum atoms stand at the nodes of the crystal lattice. Six aluminum atoms are centered on the faces of the crystal lattice. Positively charged aluminum ions form an elemental lattice that forms a crystalline structure. There is electronic gas in the volume of the lattice. Based on the specified parameters, the elementary lattice of Al nanoparticles was constructed in the Diamond 3.2 program. The crystal structure is shown in figure 3. As can be seen in the figure, this crystal structure has a fairly high symmetry. Al atoms stand at a minimum distance from each other. These distances are calculated as: $d_{\text{Al-Al}} = 2.8524$ Å.

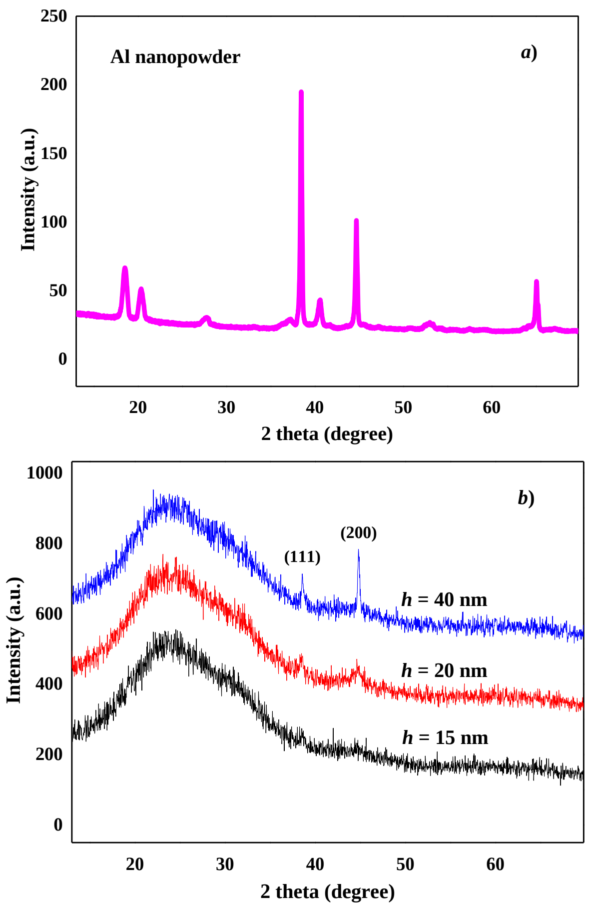


Figure 2. X-ray diffraction spectra of Al nanopowders (*a*) and Al thin layers (*b*) with thickness h = 15, 20 and 40 nm obtained on a glass substrate

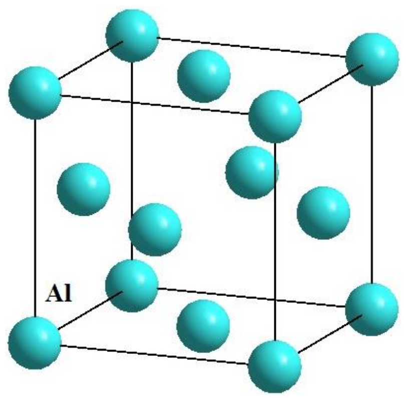


Figure 3. Crystal structure of Al nanoparticles

The X-ray diffraction spectra of thin films were compared with the X-ray diffraction spectra of nanopowders. An additional background is formed in thin layers due to amorphization. Two main diffraction maxima were observed in the spectrum at diffraction angles $2\theta = 38.57°$ and 44.79°. The analysis showed that these atomic planes correspond to the (111) and (200) atomic planes in the cubic symmetric crystal structure of the space group Fm-3m. As can be seen from figure 2, as the thickness of the nanolayers increases, the intensities of the peaks corresponding to the (111) and (200) atomic planes also increase. It turned out that as the thickness of the thin layer increases, the process of phase formation occurs and the crystal structure is formed.

As can be seen from structural studies, nano-sized structural properties can be formed when thin layers of aluminum are obtained on a glass substrate. The formation of the structure is of great physical importance. It is known that the crystal lattice of metallic materials consists of ionic bonds. Electron gas is present throughout the volume of the crystal. Therefore, conductivity is observed in metals. One of the main conditions is the presence of conductivity in thin layers. These studies have shown that crystalline structures can also form in aluminum nanoparticles obtained on glass. The main result is that aluminum nanoparticles of size h ~ 40 nm can be used as conductive boards.

The results of the study of the crystal structures of aluminum nano-layers show that highly symmetrical crystal structures can be formed in these layers. However, these layers also have a number of structural features. It is known that Al nanoparticles combine with oxygen and water in the air under normal conditions to form aluminum oxide and aluminum hydroxide. This process is inevitable even on the surface of thin layers. However, this effect was not observed during the study. This is because such small effects (processes at the atomic level) cannot be observed by X-ray diffraction. Additional physicochemical analysis is needed to observe these effects.

4. CONCLUSIONS

The nanometer thick Al thin layers were obtained on glass substrates and their surface structures were investigated. The X-ray diffraction spectra of the thin layers were compared with the spectra obtained for aluminum nanopowder. A model of the crystal structure of Al nanoparticles was constructed, and the location characteristics of atoms in a highly symmetrical crystal structure were shown. The Fm-3m space group model consisting of ionic bonds has been constructed. The phase formation process during the formation of a crystal structure in aluminum was investigated. It has been determined that structural aspects are felt in h = 15 nm thick layers. In h = 20 and 40 nm thick layers, a crystal structure has already been formed. This process took place in the X-ray diffraction spectra and was identified by analyzing the peaks corresponding to the (111) and (200) atomic planes.

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Khuraman Nusret Ahmadova was born in Gabala, Azerbaijan, on March 20, 1985. She received the M.Sc. degree in Quantum Electronics specialty in Physics from Baku State University (Baku, Azerbaijan) in 2008. She received Ph.D. degree in Semiconductor Physics titled "Dielectric Function and Optical Models of Multilayer Structures" in 2015. Her scientific interests are dielectric response of electron and phonon subsystems of solids to light irradiation, contribution of interband optical transitions and IR active phonons into dielectric response, spectroscopic ellipsometry of anisotropic bulk crystals, nanostructured materials, single- and multi-layer thin film structures.

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