GAMMA-IRRADIATION OF ALUMINUM OXIDE Al_2O_3 AND ITS FEATURES DETECTED BY POWDER DIFFRACTOMETRY

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Abstract. *X-ray diffraction analysis of materials is a standard method for phase identification and characterization of polycrystalline materials [1]. Experimentally, various peaks were detected in the X-ray phase analysis (XRD) spectra of aluminum oxide Al_2O_3 samples. Based on the data (XRD) of samples of aluminum oxide Al_2O_3 obtained for sample with different modes of powder production, Miller indices and lattice parameters were determined. The experimental data obtained are in good agreement with the data obtained by other methods.*

Keywords: *gamma irradiation, aluminum oxide, powder, wide-gap semiconductor, Miller indices, microstructure.*

Introduction

In this work, we used powder X-ray phase analysis to study the structure, composition, properties of raw materials and products of aluminum oxide Al_2O_3. It was used to study the mineralogical and phase compositions [2]. Aluminum oxide Al_2O_3 is a binary compound of aluminum and oxygen [3]. In nature, it is distributed in the form of alumina, which is a constituent of clays, a non-stoichiometric mixture of oxides of aluminum, potassium, sodium, magnesium, etc. [4–6]. In the modification of corundum, it has an atomic crystal lattice. Taking into account the above, in this work, we carried out a study by the method of powder diffractometer of the main features of materials used in semiconductor technology. And also an attempt was made to apply the analysis technique for materials of various stoichiometry.

Research methodology

Powder X-ray diffraction is a method for studying the structural characteristics of a material using X-ray diffraction (X-ray diffraction analysis) on a powder or polycrystalline sample of the material under study. Also called the powder method. The result of the study is the dependence of the scattered radiation intensity on the scattering angle. The corresponding instrument is called a powder diffractometer. The advantage of the method is that the debyegram for each substance is unique and allows you to determine the substance even when its structure is not known. The X-ray phase analysis method was used to study the structure, composition, properties of raw materials and calcined products. It was used to study the mineralogical and phase compositions [7]. A monochromatic X-ray beam is directed to a sample of the test material, ground into powder. On a photographic film rolled into a cylinder around the sample, the image (Debyegram) is obtained in the form of rings. The distance between the lines of the same ring on the debygram allows you to find the Bragg reflection angles. Then, using the Bragg-Wulf formula $2d \sin\Theta=n\lambda$, the ratio d/n of the distance between the reflecting planes to the order of reflection can be obtained.

X-ray analysis allows solving the following tasks:

Determination of the qualitative composition of the sample, semi-quantitative determination of the components of the sample, determination of the crystal structure of the

substance. As well as precision determination of unit cell parameters, determination of the arrangement of atoms in an elementary cell (full profile analysis - the Rietveld method), determination of the crystallite size (coherent scattering region) of a polycrystalline sample. Study of texture in polycrystalline materials. In addition, the study of the phase composition of the substance and the study of state diagrams, the assessment of the size of crystals in the sample, the exact determination of lattice constants, the thermal expansion coefficient, the analysis of minerals. In Fig.1. the device - powder diffractometer is given.

Fig.1.

Powder x-ray diffractometry. XRD-6100.

Main results and discussion

Al2O3 powder samples were studied by X-ray diffraction and elemental analysis. Samples were identified on the basis of diffraction patterns, which were recorded on a computer-controlled XRD-6100 (Shimadzu, Japan) apparatus. Cu-Kα radiation (β-filter, Ni, λ=1.54178 Å, tube current and voltage mode 30 mA, 40 kV) and a constant detector rotation speed of 4 deg/min with a step of 0.05 deg were used. (ω/2θ-coupling), and the scanning angle varied from 10 to 80°. The X-ray power was 2 kW. The results were analyzed using the database [8]. The penetration depth of Cu-Kα radiation is about 1 mm (980 μm) for light elements (carbon) and a few μm for heavy elements (Ag, W). For most inorganic substances, simple compounds, Cu-Kα- is tens of microns (μm). Figure 2 shows the spectral dependences of Al2O3 obtained by the powder diffractometer. In addition, the Miller indices are given, as well as the interplanar spacing dhkl for these samples. We used the Rietveld method [7, 9] to refine the structure from powder data obtained using X-rays. The principle of the method is to use independent intensity measurements at each point of the diffraction pattern, describing the line profile using analytical functions, instead of using the integral reflection intensity. Function parameters, including structural, device and other characteristics, are refined using the nonlinear least squares method. Using this refinement method, we determined the interplanar spacing dhkl and the Miller indices (hkl). In addition, using this method, we were able to accurately determine and designate the interplanar spacing dhkl and Miller indices (hkl) as can be seen from Figure 2. As mentioned above, powder X-ray diffractometry allows for quantitative elemental analysis. The elemental analysis carried out by us using the “Search and Match” software [8] of the samples shows that the Al2O3 samples have the

following composition (in weight percent): for Al2O3, the following weight ratios were obtained: Al - 68.56%, O - 31.44% . As is known from the literature data [7,10-11], Miller indices are applicable in all syngonies. As the Miller index increases, the interplanar spacing decreases [12–13]. For the Al2O3 sample measured by X-ray diffraction analysis using the “Search and Match” software [8,13–14], the degree of crystallinity and amorphism was assessed. For alumina, it looks like this: the amorphous phase for alumina is 56.84%, and the crystalline phase, respectively, is 43.16%. Indexing - determination of indices (HKL) for each line of the diffraction pattern and grating type. Indexing was carried out to identify impurities in the sample by isolating reflections that do not belong to the main substance. In this work, we determined the presence of hydrogen impurities for Al2O3. The most characteristic results are shown in Fig.2.

Fig.2.

Spectral dependence of Al2O3 obtained by the method of powder diffractometer. And also processed by the Rietveld refinement using the FullProf software [9]. Miller indices are given.

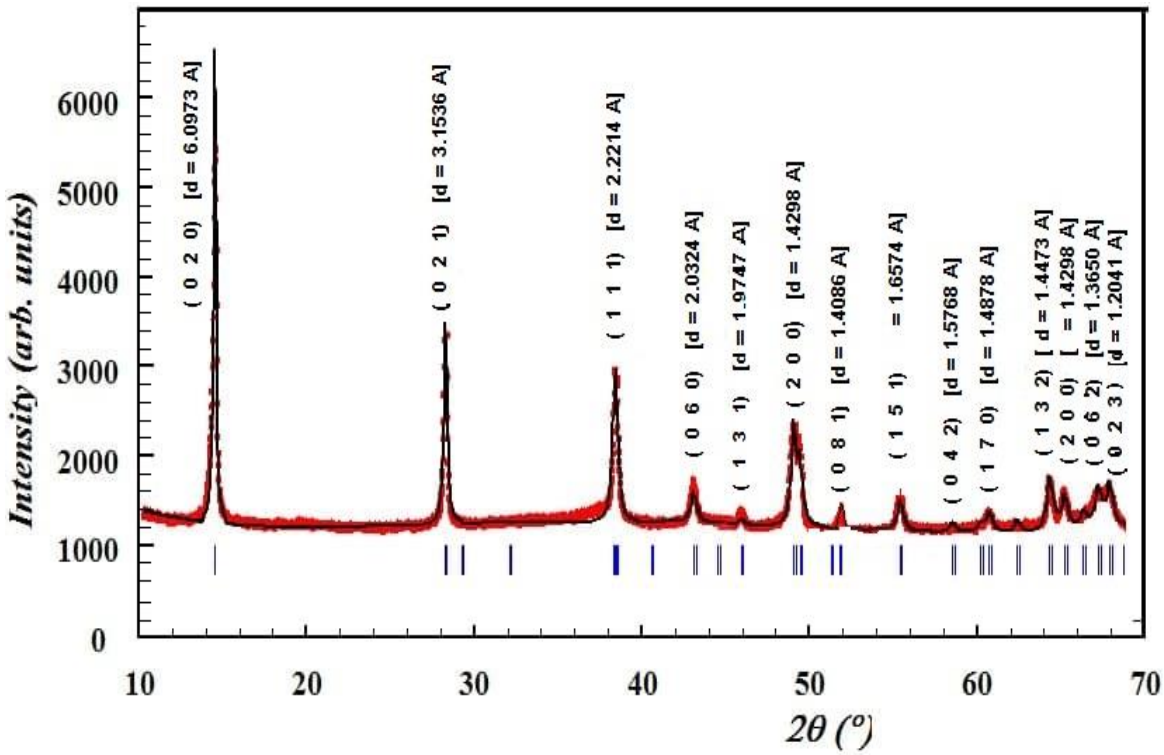


Figure 3 shows the spectral dependence of Al_2O_3, obtained by the method of powder diffractometer (XRD-6100) after gamma irradiation with a dose of D=107 ion/cm2. As can be seen from the spectral dependence, irradiation leads to a decrease in the peaks of the corresponding Al_2HO_2, and to the amorphization of this sample. We can determine this as follows: for an Al_2O_3 sample measured by X-ray diffraction analysis using the “Search and Match” software [8,12-14], the degree of crystallinity and amorphism was assessed. For an irradiated alumina sample, this is as follows: the amorphous phase for alumina is 66.42%, and the crystalline phase, respectively, is 33.58%. In addition, we carried out elemental analysis using the “Search and Match” software [8] of the samples. Samples of Al_2O_3 have the following composition (in weight percent): for Al_2O_3, the following weight ratios were obtained: Al - 45%, O - 53.30%, and hydrogen-H-1.7% was also found. Indexing was carried out to identify impurities in the sample by isolating reflections that do not belong to the main substance. In this work, we determined the presence of hydrogen impurities for Al_2O_3. This indicates that the hydrogen in the volume after gamma irradiation comes to the surface.

Fig.3.

Spectral dependence of Al_2O_3 obtained by the method of powder diffractometer after gamma irradiation with a dose of D=107 ion/cm2.

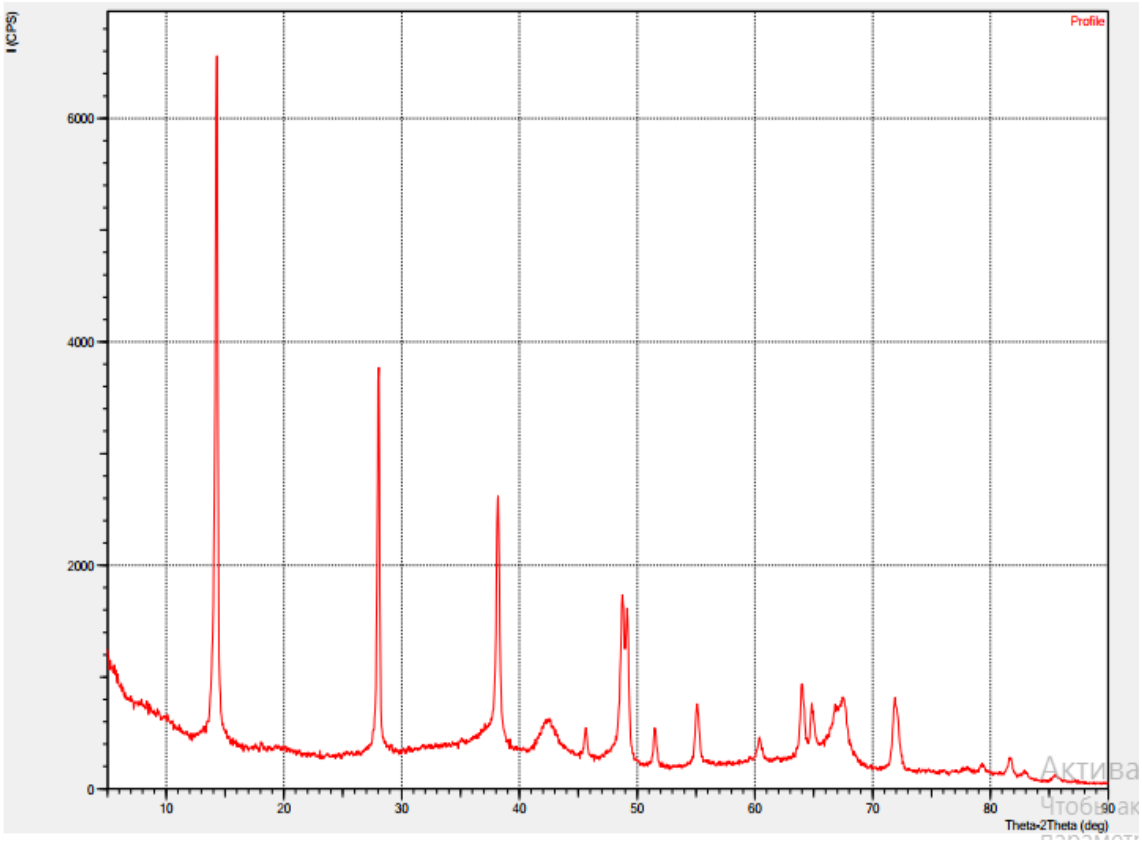
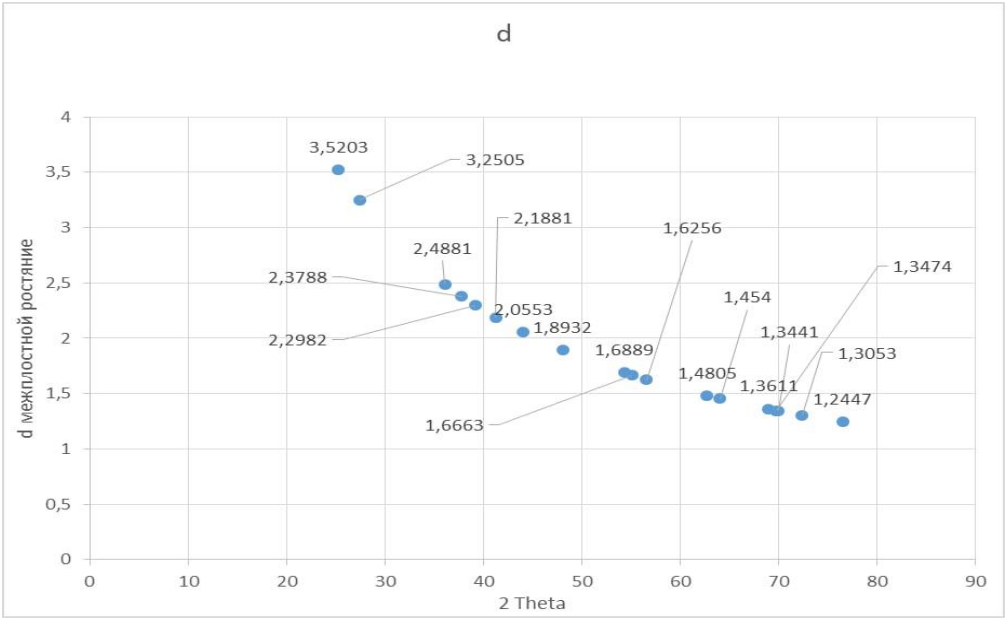


Fig.4.

Dependence of the interplanar distance on the angle of incidence of radiation.



As can be seen from Figure 4, with an increase in the angle of incidence of X-ray radiation on the sample, the interplanar distance decreases [10,12,13-14]. Thus, we can conclude from Figure 4 that the interplanar spacing strongly depends on the Miller index. This shows that using this method, based on analytical work, we can conduct a qualitative and quantitative analysis of

samples of various compositions, with different stoichiometry, and determine various crystallographic parameters.

Findings

The Rietveld method [7, 9] was used to refine the structure from powder data obtained using X-rays. A decrease in the interplanar distance with increasing Miller indices is determined. Indexing was used to identify impurities in the sample by isolating reflections that do not belong to the main substance. Elemental analysis was carried out in weight percent for Al_2O_3 Al - 68.56%, O - 31.44%. The degree of crystallinity and amorphism of the Al_2O_3 sample was determined. Also, gamma irradiation of the Al_2O_3 sample was carried out with a dose of D=107 ion/cm2. Surface amorphization after gamma irradiation was determined.

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