

## USE OF X-RAY ANALYSIS FOR CONDUCTING INPUT AND PROCESS CONTROL OF ELECTRONICS MATERIALS

Aleksandra N. Matvienko<sup>\*</sup>, Sergey P. Teslenko, Svetlana V. Shtelmah

Giricond, JSC; Kurchatov Str. 10, St. Petersburg, 194223, Russia

<sup>\*</sup>e-mail: matvienko\_an@spbstu.ru

**Abstract.** The paper presents examples of using X-ray diffraction and X-ray phase analysis for monitoring and researching materials for electronic equipment.

**Keywords:** crystal lattice parameter, diffraction line, materials for electronic equipment, phase composition, technological control, X-ray phase analysis

### 1. Introduction

X-ray structural and X-ray phase analysis allows determining the phase composition and crystallographic modification of analyzed material with the sensitivity of at least 5% of the mass. Analysis of results is based on the use of the ASTM X-ray data base. The phase composition of a sample is defined from the analysis of the diffraction pattern taken in a wide range of angles. To obtain information about the crystallographic structure (composition of the solid solution, the degree of formation of the crystal lattice), a survey is carried out in the region of large diffraction angles, which makes it possible to determine the lattice parameter and the broadening of diffraction lines with the required accuracy. In complex cases, additional information about the chemical composition of the sample is involved [1].

Based on this approach, we have developed a number of techniques for conducting input and current process control of materials for electronic equipment [2].

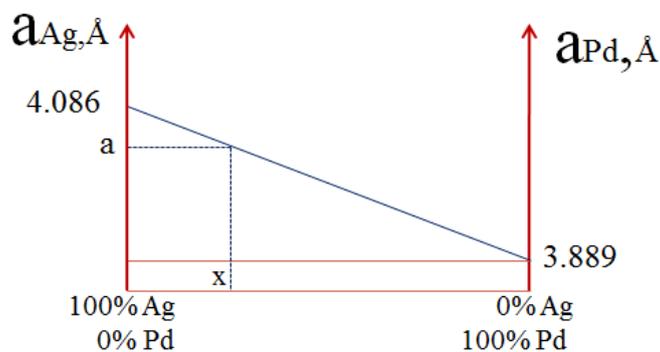
### 2. Experimental

**Input control. Silver-palladium alloys.** Ag-Pd alloys are used as materials for internal electrodes of multilayer ceramic capacitors and varistors [3]. In the process of work, it turned out that for the production technology of the above-mentioned products, information not being stipulated by the alloy specification may be significant. The technique developed allows determining a more wide range of phases that can be present in the alloy, including X-ray amorphous phase, by recording the X-ray pattern of the sample in a wide range of diffraction angles; for example, Ag<sub>2</sub>O, PdO, Ag<sub>2</sub>S phases. It includes also the determination of crystal lattice parameter that is necessary for defining alloy composition. Silver and palladium form a continuous series of solid solutions [4] for which Vegard's law is fulfilled. This enables the composition to be found from the dependence of crystal lattice parameter of a solid solution on component concentration (Fig. 1),

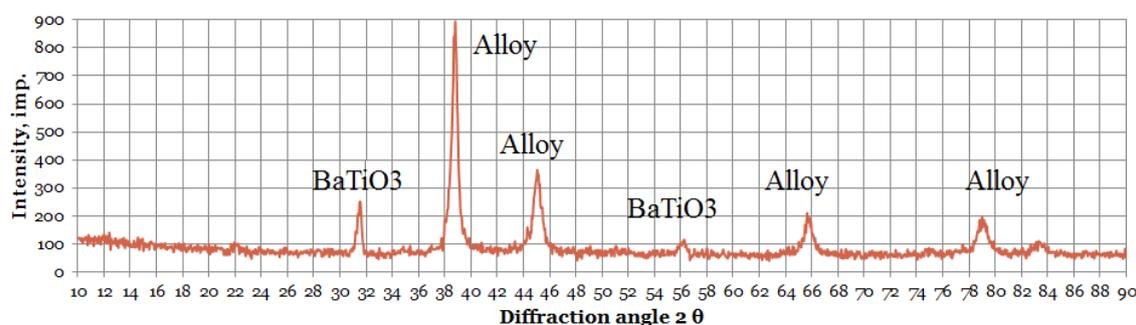
In addition to the phase composition and the lattice parameter, the half-width of the diffraction lines is fixed that indicates the size of coherent scattering domain of and, thus, the degree of structure formation.

Consider also an alloy with the addition of cladding elements. To determine the concentration of incoming components, a diffraction line is taken in the region of large diffraction angles. Recording and decoding the radiogram (Fig. 2) shows that, in addition to the alloy, there is a cladding element BaTiO<sub>3</sub>. Since the crystal lattice of the alloy is cubic, it

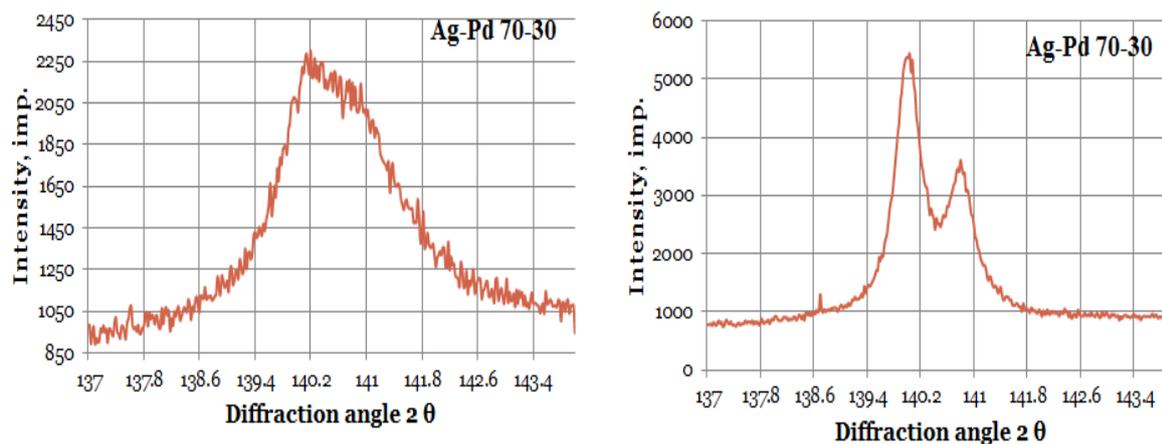
is enough to record only one diffraction line. We use diffraction line (422), since no other diffraction lines are superimposed on it. The structure formation is estimated by the separation degree of doublet  $\alpha_1$  и  $\alpha_2$  (Fig. 3): if the structure is well formed, the doublet is separated (Fig. 3, at the right).



**Fig. 1.** Vegard's law for system Pd-Ag



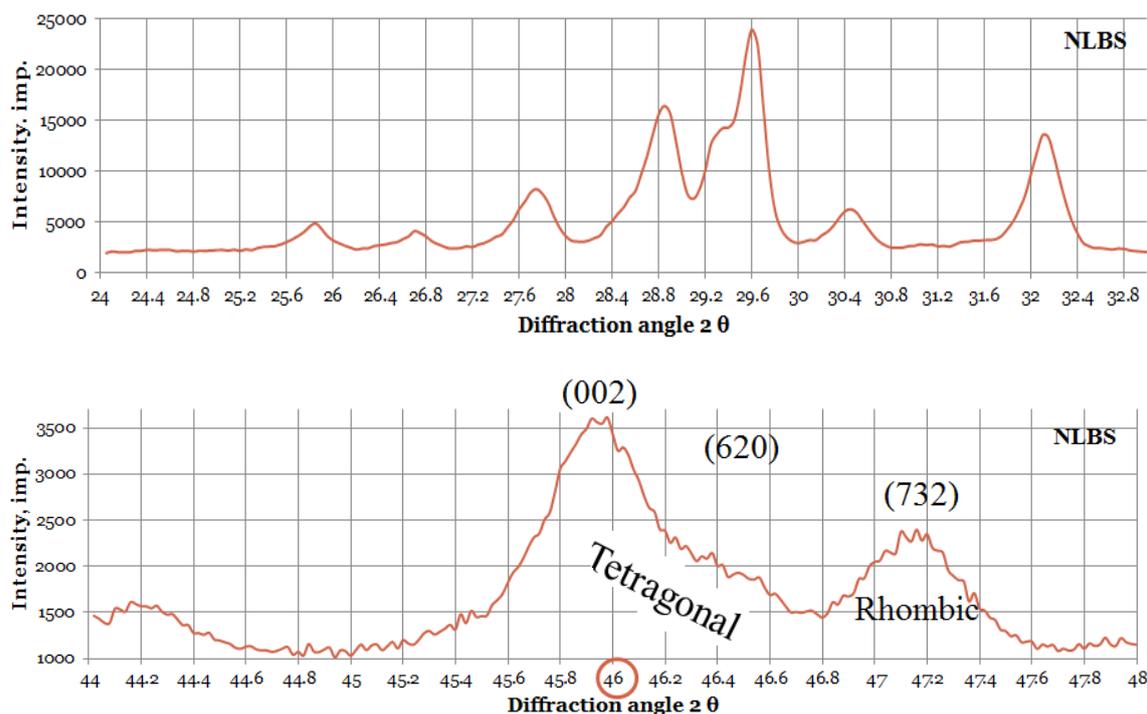
**Fig.2.** X-ray pattern of the alloy Ag-Pd with the addition of cladding elements



**Fig. 3.** Diffraction line (422)

**Technological control of the preliminary synthesis of NLBS material.** NLBS is ferroelectric material with the structure of potassium-tungsten bronze based metaniobate lead:(Pb, Ba, Sr)  $\text{Nb}_2\text{O}_6$  that is used in capacitor manufacturing [3, 5]. The synthesis of this ceramics takes place in two stages: preliminary synthesis and final one during high-temperature sintering. We have found correlation between the crystallographic structure of the material formed during the primary synthesis and the electrophysical properties of the subsequent high-temperature firing of ceramics. We have deciphered the phase composition

of the material obtained by the preliminary synthesis. There are two phases in its foundation: tetragonal structure  $\text{Pb}_{0.3}\text{Ba}_{0.7}\text{Nb}_2\text{O}_6$  and orthorhombic structure  $\text{Pb}_{0.7}\text{Ba}_{0.3}\text{Nb}_2\text{O}_6$ . The crystal lattice parameters of these phases are changing depending on the introduced additives, their quantity and synthesis conditions. Under normal conditions of synthesis, a material with a quantitative advantage of the tetragonal phase should be obtained. The complexity of decoding and evaluating the phase ratio is connected with the fact that the diffraction lines of these phases superimpose on each other. However, having a "good" reference sample, which was preliminarily synthesized in a tunnel kiln, we were able to isolate diffraction lines of each of the indicated phases (not overlapping on each other) and to determine the ratio of their intensities and diffraction angles (Fig. 4).



**Fig. 4.** Radiogram of a reference sample

These lines are (002), (620) for the tetragonal phase, and (732) for the orthorhombic phase. The diffraction lines under consideration are located in the region of diffraction angles  $2\theta = 44^\circ\text{--}48^\circ$ . The recording of these lines and the determination of their intensity ratio and diffraction angles allow us to compare them with the standard and make conclusion about the quality of synthesis.

### 3. Conclusion

The developed technique of input control of the phase composition and crystallographic parameters of the silver-palladium alloy allows controlling the presence of foreign phases, the composition of solid solution and the formation degree of crystal lattice of the alloy.

The proposed method of controlling the phase composition of NSBS material after preliminary synthesis allows predicting the quality of ceramics after high-temperature firing.

**Acknowledgements.** No external funding was received for this study.

**References**

- [1] Gorelik SS, Skakov YA, Rastorguev LN. *Radiographic and Electron-Optical Analysis*. Moscow: MISIS; 2002.
- [2] Matvienko AN, Shtelmah SV. Control and study of materials by X-ray analysis. In: *Materials of II Scientific and Technical Conference of Young Scientists and Specialists: Materials science, technological problems and development directions of passive electronic components and photoelectric and optoelectronic devices of the infrared range, St. Petersburg; May 15-16; 2018*. St. Petersburg; 2018. p.24-25.
- [3] Rotenberg BA. *Ceramic Capacitor Dielectrics*. St. Petersburg: Girikond; 2000.
- [4] Masterov VA, Saksonov UV. *Silver, alloys and bimetal based on it*. Moscow: Metallurgy; 1979.
- [5] Sidorov AO, Rubinshtein OV. Ceramics based on solid solutions of the pseudo-binary system BaNb<sub>2</sub>O<sub>6</sub> - SrNb<sub>2</sub>O<sub>6</sub> for multilayer capacitors. In: *Materials of II Scientific and Technical Conference of Young Scientists and Specialists: Materials science, technological problems and development directions of passive electronic components and photoelectric and optoelectronic devices of the infrared range, St. Petersburg; May 15-16, 2018*. St. Petersburg; 2018. p.35-37.