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Abstract

Full Text

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PHYSICS

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INVESTIGATION OF THE TEMPERATURE DEPENDENCE OF THE FINE STRUCTURE OF THE MAIN EDGE OF THE X-RAY ABSORPTION SPECTRA OF TITANIUM IN BARIUM TITANATES NEAR THE CURIE POINT FOR $BaTiO_3$

(Presented by Academician A. P. Vinogradov, 7 XII 1959)

Investigations of the fine structure of the x-ray titanium K-absorption edge in barium titanates ([1,2]) showed that the structure of the main absorption edge in monotitanate at room temperature differs substantially from that for tetratitanate and is connected with the crystal structure of the compound and with the state of polarization of the atoms in it. For a more detailed study of this phenomenon and for testing certain assumptions put forward ([2]) in the course of theoretical discussion, an experimental investigation of the temperature dependence of the fine structure of the main titanium absorption edge in barium titanates in the temperature region near the point of the ferroelectric transition for monotitanate was of great interest.

The present article sets forth the results of such an investigation. The absorption spectra were obtained with a focusing tube-spectrograph of the design described earlier ([3]). The analyzer was a quartz crystal with lattice constant $d = 3.336$ Å, bent to a radius of 250 cm.

The linear dispersion of the instrument in the wavelength region under consideration was 2.5 X/mm. The anode of the x-ray tube was gold-plated. The comparison lines used were the lines $AuL_{\alpha 1}$ and $AsK_{\alpha 1}$ in the second order of reflection. The operating conditions of the tube were 11 kV and 50 mA; exposures were 3–5 hours. The spectra were recorded photographically under conditions analogous to those described in ([1,2]).

Fig. 1. Furnace for heating absorbers.

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Fig. 1

Figure 1: Fig. 1

Fig. 2. Microphotograms of the titanium K absorption edge in barium titanates at different temperatures

Figure 2: Fig. 2. Microphotograms of the titanium K absorption edge in barium titanates at different temperatures

1 —ceramic tube; 2 —metal flange; 3 —celluloid covering the window; 4 —water-cooling tube; 5 —absorber.

For heating the specimens, a special furnace was designed and constructed (Fig. 1), consisting of a ceramic tube heated by a spiral wound around it. The temperature of the heater could be regulated within the range 18–400° and maintained constant to an accuracy of 2.5°. Temperature measurement was carried out with an iron–constantan thermocouple. Copper flanges were placed at the ends of the heater tube; these were cooled by flowing water and were provided with special windows. The windows were covered with a thin layer of celluloid. On the outside, the heating spiral was coated with a layer of kaolin mixed with asbestos. In the middle part of the heater a slot was made, through which the absorber and the thermocouple adjoining it were inserted into the heater. From the outside the slot was tightly—

was closed with asbestos. As control measurements of the temperature inside the heater showed, it remained unchanged to within 3–4° along the diameter of the selected cross section. The heater was placed in a horizontal position between the X-ray tube and the crystal, so that the X-rays, directed along the axis of the heater tube, passed through the absorber. The distance from the anode of the X-ray tube to the crystal was 10 cm. Under these conditions, as a check showed, the crystal was not heated.

In contrast to the generally accepted method of preparing absorbers, described, for example, in [4], the absorbers used in the present work, in order to give them the necessary heat resistance, were prepared on the basis of BF-2 glue. The finely dispersed powder of the compound under study was thoroughly shaken in a solution of BF-2 glue in alcohol and poured onto the smooth surface of aluminum foil 0.01 mm thick. Thus, in its finished form the absorber was a sheet of foil with a layer of the investigated substance glued onto it. The surface density of the absorbers was chosen close to 7 mg/cm^2. To give the absorber greater mechanical strength, the aluminum foil with the absorbing substance was glued onto a thin but rigid aluminum ring, whose diameter was equal to the inner diameter of the heater tube. In this form the absorber was inserted into the heater.

Fig. 2. Microphotograms of the titanium *K*-edge absorption in barium titanates at different temperatures

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The X-ray absorption spectra of titanium in barium mono- and tetratitanate were investigated on both sides of the Curie temperature for $BaTiO_3$ (120°), in the interval from 20 to 200°. For each of the titanates at a given temperature, 7 to 10 independent spectrograms were obtained; these were photometered in three sections over the height of the spectrum and, after averaging and recalculation on the electron-volt scale, were compared. The titanium absorption spectra thus obtained in barium mono- and tetratitanate at temperatures of 20, 50, 100, 120, 150, and 200° are shown in Fig. 2.

Consideration of the results obtained makes it possible to draw the following conclusions about the fine structure of the spectra:

1. The conclusion drawn earlier concerning the substantial difference in the fine structure of the X-ray titanium absorption edge in barium titanates with different ratios of BaO and TiO_2 is fully confirmed. As the results of the present work show, this is the case at all temperatures investigated.
2. The temperature dependence of the fine structure of the principal titanium absorption edge in monotitanate reveals the following regularities.

As the temperature is raised from 20 to 100°, a noticeable and systematic increase is observed in the degree of blurring of the structure, which becomes maximal at the Curie point. Above this point, the intensity of the fluctuations and the sharpness of the fine structure in the titanium absorption edge in the compound are much smaller than in the ferroelectric region, and depend very little on temperature.

3. In nonferroelectric barium tetratitanate, practically no dependence of the fine structure on temperature is observed.

In conclusion, I consider it my pleasant duty to express my sincere gratitude to E. E. Vainshtein and I. B. Staryi for suggesting the topic of the present work, for their assistance in carrying it out, and for participating in the discussion of the results obtained.

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