

# Emission of ThO<sub>2</sub> Valence Electrons upon Excitation with Synchrotron Radiation Near the O<sub>4,5</sub>(Th) Resonance Absorption Threshold

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**Abstract**—The structure of the emission spectra of ThO<sub>2</sub> valence electrons, arising upon excitation with synchrotron radiation near the O<sub>4,5</sub>(Th) resonance absorption threshold in the photon energy range 70 <  $h\nu$  < 140 eV, was examined taking into account the structure of the photoelectron spectrum and X-ray O<sub>4,5</sub>(Th) absorption spectrum, and also the results of relativistic calculation of the electronic structure of ThO<sub>2</sub>. The line intensities in the emission spectrum of ThO<sub>2</sub> valence electrons in the energy range from 0 to 40 eV considerably increases near the O<sub>4,5</sub>(Th) resonance absorption thresholds at 90 and 102 eV. Appearance of this structure reflects the excitation and decay processes involving electrons of outer valence MOs (OVMOs, from 0 to 13 eV) and inner valence MOs (IVMOs, from 13 to 35 eV). These processes occur concurrently with common photoelectron emission processes. As the considered spectra at resonance are associated to a greater extent with giant Coster–Kronig transitions involving electrons of valence and inner shells, these spectra reflect the partial density of states of Th6*p* and Th5*f* electrons. The smearing of the structure in the range from ~13 to 35 eV is attributed to the IVMO formation in this oxide.

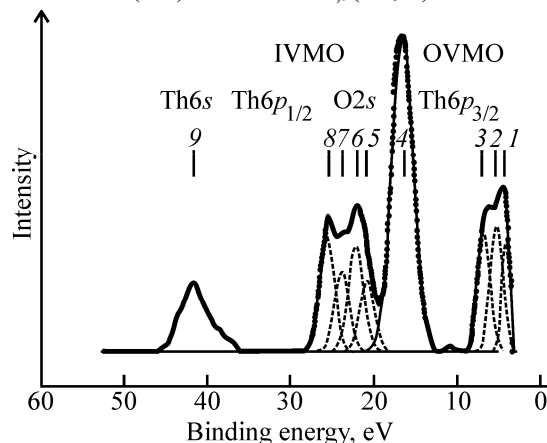
**Key words:** thorium dioxide, emission spectra, X-ray absorption spectra, X-ray photoelectron spectra, molecular orbitals

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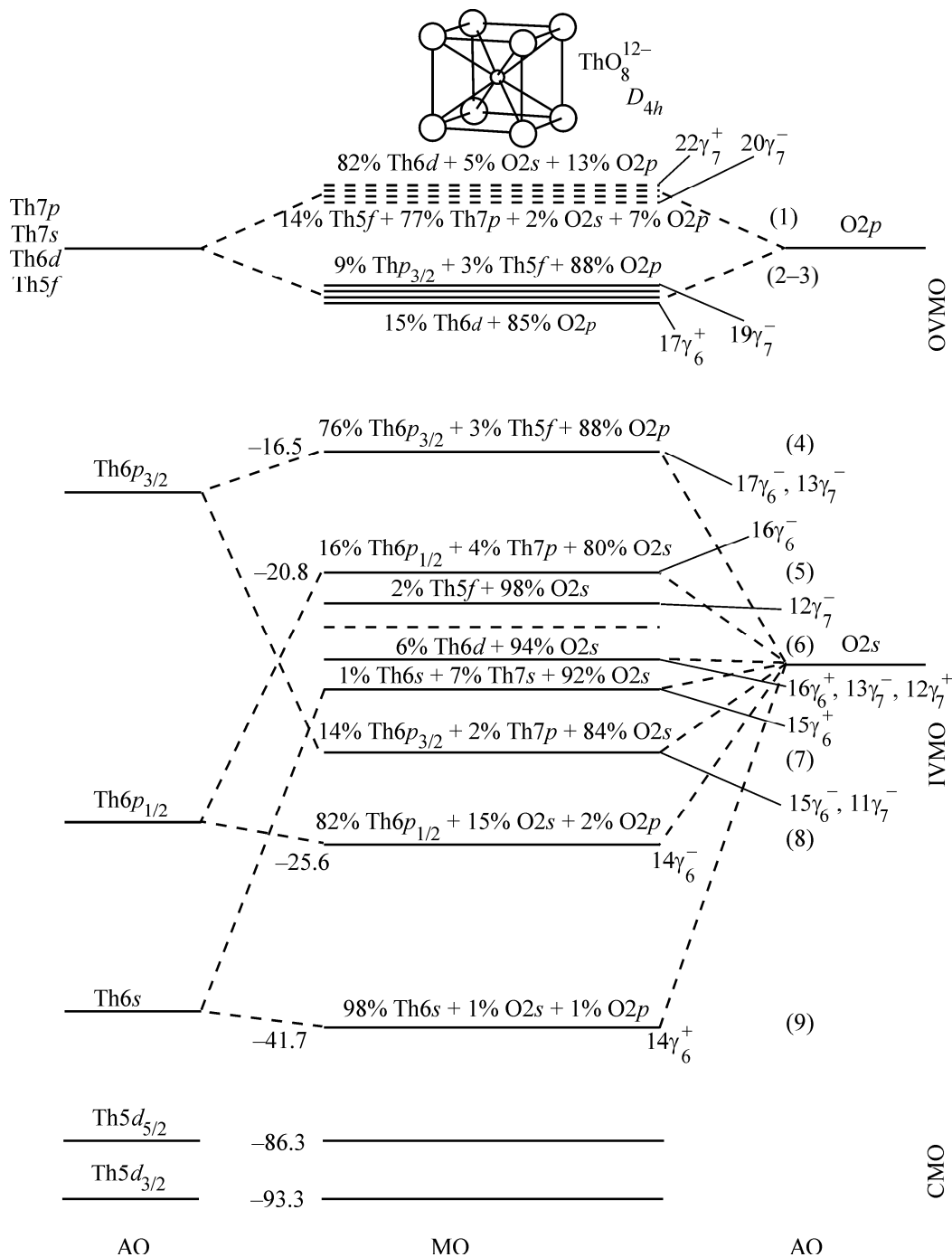
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Analysis of the fine structure of an X-ray photoelectron spectrum of ThO<sub>2</sub> valence electrons in the binding energy range from 0 to 35 eV showed that this structure is largely associated with the formation of outer valence (OVMO, from 0 to 13 eV) and inner valence (IVMO, from 13 to 35 eV) molecular orbitals [1]. It was shown that, along with Th6*d*,7*s* atomic orbitals (AOs), Th5*f*,6*p*,7*p* AOs also participate in OVMO formation, whereas IVMOs are largely formed by Th6*p* and O2*s* AOs of the adjacent Th and O atoms [2]. This spectrum reflects the structure of the valence band in the range from 0 to 35 eV and is observed as bands with a width of several electron-volts (Figs. 1, 2). Certain experimental [3, 4] and theoretical [2, 5] data suggest the presence in the ThO<sub>2</sub> valence band of filled states of Th5*f* electrons, despite the absence of Th5*f* electrons in the Th atom. Delocalized Th5*f* electron states can also be observed in metallic Th [6]. Participation of Th5*f* electrons in chemical bonding in ThO<sub>2</sub> should affect the structure of X-ray spectra if

formation of this structure involves these electrons. Emission of outer valence electrons with the binding energy from 0 to 10 eV upon excitation with synchrotron radiation (SR) near the O<sub>4,5</sub>(Th,U) resonance ab-



**Fig. 1.** X-ray photoelectron spectrum of ThO<sub>2</sub>. Dotted lines show resolution of the spectrum into separate components. Figures over the spectrum components correspond to the scheme (Fig. 2).



**Fig. 2.** Scheme of MO of the ThO<sub>8</sub><sup>12-</sup> cluster (D<sub>4h</sub>), constructed taking into account theoretical and experimental data. To the left are experimental values of electron binding energies (eV). The energy scale is not consistent. The MO nos. correspond to line nos. in XPE spectrum (Fig. 1).

sorption threshold was studied previously for metallic Th [7], U<sub>x</sub>Th<sub>1-x</sub>Sb [8], and UO<sub>2</sub> [9].

In this study we examined the fine structure of the emission spectra of ThO<sub>2</sub> valence electrons in the binding energy range from 0 to 40 eV, arising upon excita-

tion with SR near the O<sub>4,5</sub>(Th) resonance absorption threshold in the photon energy range 70 < hν < 140 eV, taking into account the structure of the X-ray photoelectron spectrum and O<sub>4,5</sub>(Th) X-ray absorption spectrum, and also the results of a relativistic calculation of the electronic structure of ThO<sub>2</sub>.

## EXPERIMENTAL

The X-ray photoelectron spectrum of ThO<sub>2</sub> was obtained with an HP5950A electrostatic spectrometer using monochromated AlK<sub>α1,2</sub> excitation X-ray radiation ( $h\nu = 1486.6$  eV) in a vacuum ( $1.3 \times 10^{-7}$  Pa) at room temperature. The binding energies  $E_b$  (eV) are given relative to the binding energy of C1s electrons of hydrocarbons on the sample surface, taken equal to 285.0 eV. The error in determination of electron binding energies and line widths does not exceed 0.1 eV, and that in determination of relative line intensities, 10%.

The ThO<sub>2</sub> sample for an X-ray photoelectron study was prepared from a finely dispersed powder ground in an agate mortar, in the form of a dense layer with a mirror-finished surface, pressed into In on a metallic support, and also in the form of a surface oxide on a Th plate. The characteristics of this spectrum (electron binding energies) within the measurement error did not differ from the corresponding quantities for the spectrum of ThO<sub>2</sub> formed on the surface of a metallic plate [2]. The spectrum background caused by elastically scattered electrons was subtracted from the X-ray photoelectron spectrum after Shirley [10].

The spectra of the total quantum yield and emission of ThO<sub>2</sub> valence electrons with the binding energy from 0 to 40 eV upon excitation near the O<sub>4,5</sub>(Th) resonance absorption threshold were recorded using SR (Russian-German Soft X-ray Beamline at BESSY II) and CLAMA4 electron energy analyzer (range from 0 to 2500 eV, resolution better than 0.009 eV [11, 12]).

The dioxide ThO<sub>2</sub> was prepared on a mechanically cleaned surface of Th metal foil ( $13 \times 10 \times 0.2$  mm) by oxidation in air [13]. Preliminarily this foil was glued to an aluminum plate ( $13 \times 10 \times 1.5$  mm) using a silver suspension (Leitsilber 204, Demetron). This specimen was glued to a copper holder with a vacuum adhesive (Bond Seal Repair, Huntington Mechanical Laboratories INC), and the electrical contact with it was provided by silver suspension. The specimen was heated in the specimen exchange chamber of the spectrometer at 80°C for 13 h. Mechanical cleaning of the specimen in the exchange chamber was not performed. The spectra were taken in a vacuum of  $5 \times 10^{-10}$  mm Hg using array no. 2 (400 lines per millimeter) at  $c_{ff} = 2.25$ ,  $PE = 5$  eV, and a step of 0.2 eV. The spectrum intensity was normalized against the SR beam current. The spectrum of the C1s electrons of hydrocarbons on the

sample surface was taken at an SR excitation energy of 320.0 eV. The energy of these electrons was taken equal to 285.0 eV and was used in calibration of the emission spectra of valence electrons. At this excitation energy (320.0 eV), the spectrum of valence electrons was not observed.

## RESULTS AND DISCUSSION

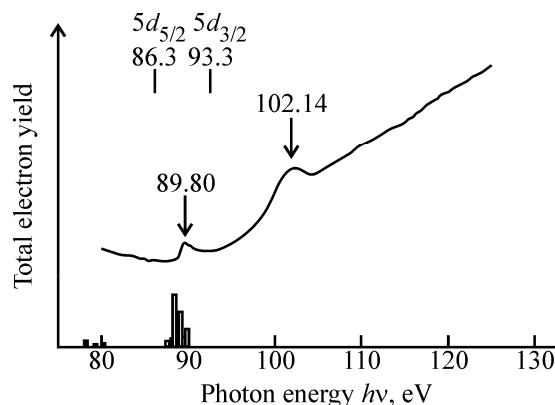
**The X-ray photoelectron spectrum of ThO<sub>2</sub>** in the binding energy range from 0 to 35 eV can be conventionally subdivided into two parts (Fig. 1). In the first part of the spectrum from 0 to 13 eV, there is the structure associated with OVMO electrons. The OVMOs are mainly formed by incompletely filled Th6*d*,7*s* and O2*p* and by unoccupied Th5*f*,7*p* AOs of the adjacent Th and O atoms (Fig. 2). In the second part of the spectrum, from 13 to 35 eV, a fine structure caused by IVMO electrons arises. The IVMOs are largely formed owing to strong overlap of the completely filled Th6*p* and O2*s* AOs. The structure of the XPE spectrum of OVMO electrons can be resolved into three components (1–3). The first component (top of the valence band) reflects the density of states of Th6*p*,5*f* and O2*p* electrons. The second component (middle of the band) is associated with the states of Th5*f* and O2*p* electrons, and the third component (bottom of the band), with the states of Th6*d* and O2*p* electrons (Figs. 1, 2). Owing to high photoionization cross section, electrons of the filled Th5*f* states can significantly enhance the intensity of the OVMO electron band [4].

In the region of the IVMO electron spectra, there are well-defined maxima, and this region of the spectrum can be resolved into five components (4–8) (Fig. 1). Despite formalism of this resolution of the spectrum into components, it allows qualitative and quantitative comparison of the characteristics of the XPE spectrum with the results of a relativistic calculation of the electronic structure of the ThO<sub>8</sub><sup>12-</sup> cluster ( $D_{4h}$ ).

The results of this calculation for the ground state of the cluster and the composition of molecular orbitals (MOs) are partially reflected in the scheme (Fig. 2). This scheme is constructed taking into account the results of these calculations and the experimental differences of the binding energies of the outer and inner electrons of Th [14] in the MO LCAO approximation. This scheme allows understanding of the real structure

of the XPE spectrum of ThO<sub>2</sub> and can be used in consideration of its various X-ray spectra.

**X-ray absorption spectroscopy near  $O_{4,5}(\text{Th})$  edge (XAS).** The spectrum of the total (quantum) yield of electrons near the  $O_{4,5}(\text{Th})$  absorption edge in the range of quantum energies from 80 to 120 eV has an unusual structure consisting of two resonance absorption bands of different intensities and widths (Fig. 3). It is known that for ThO<sub>2</sub> the electron quantum yield and absorption spectra are similar in the shape, number, energy position, and width of lines and differ only in the intensity ratio of these two bands [15, 16]. As the absorption energy threshold of the weak peak is approximately equal to 89.8 eV, which is close to the binding energy of Th5d<sub>5/2</sub> electrons in ThO<sub>2</sub> (86.3 eV), this peak can be tentatively assigned to the Th5d<sub>5/2</sub> → Th5f transition. The absorption peak due to the Th5d<sub>3/2</sub> → Th5f transition should be observed at an approximately 7 eV higher photon energy, because the difference between the binding energies of the Th5d<sub>3/2</sub> and Th5d<sub>5/2</sub> electrons is 7.0 eV (Fig. 3). The intensity of this peak should be lower than that of the peak at 89.8 eV because of different populations of the Th5d levels and of the selection rules ( $\Delta l = \pm 1$ ;  $\Delta j = 0, \pm 1$  if  $j \neq 0$ ). Indeed, at 96.8 eV there is a shoulder in the spectrum. By analogy with UO<sub>2</sub> [9, 17], it can be assumed that the main intensity of the high-energy peak is associated with the Th5d → Th7p,8p,6f transitions. Under the spectrum (Fig. 3), we present for qualitative comparison the results of calculating the density of the occupied and unoccupied states of Th5f electrons for the ground state of the ThO<sub>8</sub><sup>12-</sup> cluster ( $D_{4h}$ ). Note that some of these unoccupied MOs have a significant contribution of Th7p AOs [2]. For comparison, the calculated energies for the ground state of the ThO<sub>8</sub><sup>12-</sup> cluster ( $D_{4h}$ ) are shifted to the right by 5.96 eV, so as to make the energy of the 17γ<sub>6</sub> IVMO equal to the experimental value of the maximum at 16.5 eV (Figs. 1, 2). It should be noted that such a comparison is improper and the calculated absorption spectrum should differ even for the examined part of the absorption spectrum. This is due to the fact that the final state of the system upon absorption contains a hole on the inner Th5d level. It is well known, however, that formation of a hole on the inner level, because of its shielding, leads to an increase in the energy of the valence states by approximately 1–2 eV. Therefore, satisfactory qualitative agreement is observed in comparison of the absorption energy at 89.80 eV with the energies of the ground state (Fig. 3).

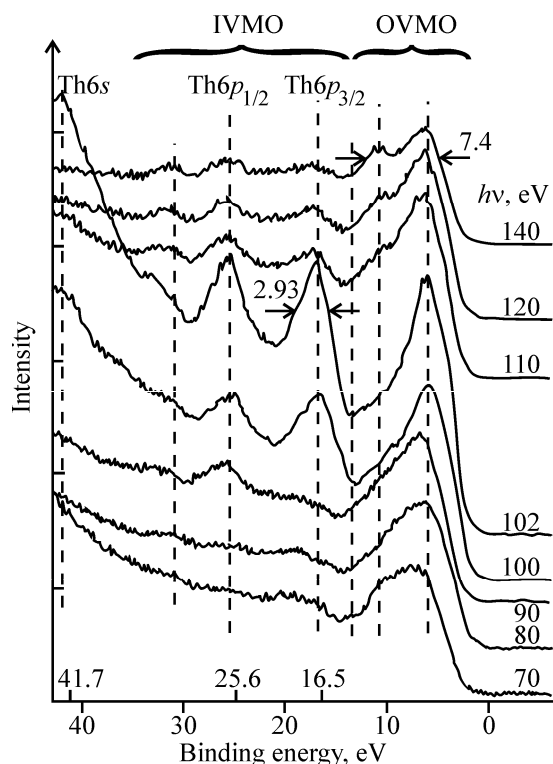


**Fig. 3.** Spectrum of the total quantum yield of electrons for ThO<sub>2</sub>. Over the spectrum are the binding energies of Th5d<sub>5/2,3/2</sub> electrons in ThO<sub>2</sub>. Bars below the spectrum denote the densities of occupied (black) and unoccupied (white) states of Th5f electrons for the ground state of the ThO<sub>8</sub><sup>12-</sup> cluster ( $D_{4h}$ ).

It follows from the results of this comparison that the low-energy peak is, apparently, indeed associated with the unfilled states of Th5f electrons. As already noted, free Th7p states onto which transitions of Th5d electrons are possible can occur at the same energies. However, transitions between shells with the same quantum number should be the most intense. These data do not contradict the assumption that Th5f electrons directly participate in chemical bonding. Indeed, if we assume that the structure of the absorption spectrum at 89.80 eV is associated with the formation of antibonding MOs, then the corresponding bonding states in OVMOs should contain filled electronic Th5f states.

Resonance electron emission (REE) for ThO<sub>2</sub> was observed upon excitation with synchrotron radiation (SR). The energy of this radiation  $h\nu$  (eV) was varied near the  $O_{4,5}(\text{Th})$  absorption edge in the energy range  $70 \text{ eV} \leq h\nu \leq 140 \text{ eV}$  and is given to the right of the REE spectra of ThO<sub>2</sub> (Fig. 4). The intensity of these spectra is normalized against the SR beam current. One graduation along the ordinate corresponds to 5000 counts in 50 scans (43 min). Under the spectra are the binding energies of electrons of certain OVMOs of ThO<sub>2</sub>. The binding energy of carbon C1s electrons on the sample surface was taken equal to 285.0 eV. In this case, the binding energies of the Th5d<sub>5/2</sub> and Th5d<sub>3/2</sub> electrons are 86.3 and 93.3 eV, respectively.

For the SR excitation energies of 70 and 80 eV (subresonance region), the structure characteristic of the photoelectron spectrum is observed (Fig. 4). The



**Fig. 4.** Resonance electron emission spectra of ThO<sub>2</sub> at various energies of excitation SR  $h\nu$  (eV). The spectrum intensity is normalized against the SR beam current. Scale division 5000 counts in 50 scans (43 min). Below the spectra are binding energies of certain IVMOs of ThO<sub>2</sub>. The binding energy of C1s electrons of carbon on the sample surface is taken equal to 285.0 eV.

most intense structure is observed in the binding energy range of OVMO electrons corresponding largely to O2p electrons of oxygen. Indeed, the photoionization cross section  $\sigma$  for O2p electrons at  $h\nu = 80$  eV is equal to 2.064 Mb and considerably exceeds the corresponding quantities for the other valence electrons (see table, [18]). In the range of binding energies of IVMO electrons of ThO<sub>2</sub>, there is weakly pronounced structure presumably associated with O2s and Th6p electrons. The surface can be contaminated with hydrocarbon molecules containing carbon and oxygen, which may significantly decrease the intensity of the spectrum of Th6p electrons in this range.

Because for Th  $E_b(\text{Th}6s) \approx E_b(\text{Th}6p_{1/2}) + E_b(\text{Th}6p_{3/2})$ , in photoemission of a Th6s electron an additional final state arises with a high probability along with the main final state, which should lead to a complex structure of the photoelectron spectrum of Th6s electrons [19]. This is associated with the giant Coster–Kronig transitions accompanying the photo-

emission of Th6s electrons. Because the transition occurs among shells with the same principal quantum number, the intensity of this transition can be higher than the intensities of the other transitions.

Because the transitions involve electrons localized on Th atoms, instead of IVMO the valence electronic configuration of the ground state of Th in ThO<sub>2</sub> can be conventionally given in atomic designations:

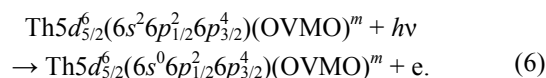
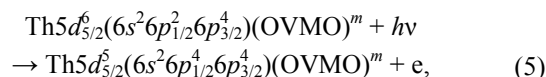
$$\text{Th}(\text{IVMO})^k(\text{OVMO})^m \approx \text{Th}(6s^2 6p_{1/2}^2 6p_{3/2}^4)(\text{OVMO})^m. \quad (1)$$

Several final states can be formed as a result of photoemission of a Th6s electron under the action of radiation  $h\nu$ : one-hole ground state (2) and two two-hole additional [(3), (4)] states



It is known [19] that formation of an additional final state (3) leads to a complex structure of the X-ray photoelectron spectrum of Th6s electrons. This fact may be responsible for the formation of a structure in the region of binding energies of Th6s electrons (Fig. 4). The maximum appearing at 30.6 eV can be attributed to an additional final state (4).

Also, for Th  $E_b(\text{Th}5d_{5/2}) \approx 2E_b(\text{Th}6s)$ . In photoemission of Th5d<sub>5/2</sub> electron, this leads to a two-hole final state (6), additional to the main final state (5):



Formation of this additional state in photoemission of Th5d<sub>5/2</sub> electron is associated with Coster–Kronig transitions and leads to a complex structure in the spectrum region corresponding to Th5d electrons. At resonance, the probability of this state (6) can increase, and state (6) itself can transform into (3) and enhance the intensity in the spectrum region corresponding to Th6s electrons.

Energies  $I_i$  and photoionization cross sections  $\sigma$  of atomic shells at various energies  $h\nu$  of excitation with photons [18]

Shell	$I_i$ , eV	$\sigma$ , Mb, at indicated $h\nu$ , eV				
		40.8	80.0	132.3	151.4	1486.6
C1s	290.9					$0.13 \times 10^{-1}$
C2s	17.5	1.170	0.5440	0.2252	0.1714	$0.66 \times 10^{-1}$
C2p	9.0	1.875	0.3262	$0.78 \times 10^{-1}$	$0.47 \times 10^{-1}$	$0.10 \times 10^{-4}$
O1s	536.8					$0.40 \times 10^{-1}$
O2s	29.2	0.8342	0.6901	0.3677	0.2984	$0.19 \times 10^{-2}$
O2p	14.2	6.816	2.064	0.5772	0.4037	$0.24 \times 10^{-3}$
Th5d	108.4			2.758	1.399	$0.76 \times 10^{-1}$
Th6s	40.5		0.2136	0.1309	0.1101	$0.29 \times 10^{-2}$
Th6p	26.7	2.911	0.4769	0.2200	0.1862	$0.81 \times 10^{-2}$
Th7s	5.0	$0.76 \times 10^{-1}$	$0.33 \times 10^{-1}$	$0.18 \times 10^{-1}$	$0.14 \times 10^{-1}$	$0.37 \times 10^{-3}$
Th6d	7.0	1.454	0.1495	$0.24 \times 10^{-1}$	$0.15 \times 10^{-1}$	$0.16 \times 10^{-2}$

At the photon energy  $h\nu = 90$  eV near the first resonance (Figs. 3, 4), certain increase in the intensity is observed in the region of OVMO electrons and of a maximum at 25.6 eV in the region of binding energies of Th6 $p_{1/2}$  electrons (Fig. 4). At the excitation energy  $h\nu = 100$  eV, close to the energy of the second resonance (102.14 eV, Fig. 3), an additional maximum at 16.5 eV is observed in the REE spectrum, and the intensity of the lines observed considerably increases except the maximum at 10.8 eV. Near the second resonance at the SR energy  $h\nu = 102$  eV, the intensity of the spectrum in the region of OVMO and IVMO binding energies, including Th6s electrons, considerably increases.

As already noted, the electronic configuration of IVMO of Th in ThO<sub>2</sub> in the ground state can be conventionally given in atomic designations (7):

$$\begin{aligned} & \text{Th}5d^{10}(\text{IVMO})^k(\text{OVMO})^m5f^0 \\ & \approx \text{Th}5d^{10}(6s^26p_{1/2}^26p_{3/2}^4)(\text{OVMO})^m5f^0, \end{aligned} \quad (7)$$

where Th5 $f^0$  denotes unfilled states of Th5f electrons. It should be noted that near these states there are unfilled states of Th7p electrons [2], which can also participate in the transitions under consideration, though with a lower probability.

At the excitation energy  $h\nu$  near the resonance absorption, a virtual excited state arises with a hole and one Th5f electron (8):

$$\begin{aligned} & \text{Th}5d^{10}(6s^26p_{1/2}^26p_{3/2}^4)(\text{OVMO})^m5f^0 + h\nu \\ & \rightarrow \text{Th}5d^9(6s^26p_{1/2}^26p_{3/2}^4)(\text{OVMO})^m5f^{0+1}, \end{aligned} \quad (8)$$

which, owing to Coulomb interaction ( $e^2/r$ ) of the hole with electrons, can decay along different pathways:

$$\begin{aligned} & \text{Th}5d^9(6s^26p_{1/2}^26p_{3/2}^4)(\text{OVMO})^m5f^{0+1} + (e^2/r) \\ & \rightarrow \text{Th}5d^{10}(6s^26p_{1/2}^26p_{3/2}^4)(\text{OVMO})^{m-1}5f^0 + e, \end{aligned} \quad (9)$$

$$\begin{aligned} & \text{Th}5d^9(6s^26p_{1/2}^26p_{3/2}^4)(\text{OVMO})^m5f^{0+1} + (e^2/r) \\ & \rightarrow \text{Th}5d^{10}(6s^26p_{1/2}^26p_{3/2}^4)(\text{OVMO})^m5f^0 + e, \end{aligned} \quad (10)$$

$$\begin{aligned} & \text{Th}5d^9(6s^26p_{1/2}^26p_{3/2}^4)(\text{OVMO})^m5f^{0+1} + (e^2/r) \\ & \rightarrow \text{Th}5d^{10}(6s^26p_{1/2}^26p_{3/2}^3)(\text{OVMO})^m5f^0 + e, \end{aligned} \quad (11)$$

$$\begin{aligned} & \text{Th}5d^9(6s^26p_{1/2}^26p_{3/2}^4)(\text{OVMO})^m5f^{0+1} + (e^2/r) \\ & \rightarrow \text{Th}5d^{10}(6s^16p_{1/2}^26p_{3/2}^4)(\text{OVMO})^m5f^0 + e. \end{aligned} \quad (12)$$

Formation of state (9) enhances the intensity of the OVMO electron band, and formation of state (10), the intensity of the Th6 $p_{1/2}$  electron line arising when the excitation energy approaches the first resonance at 89.80 eV ( $h\nu = 90$  eV).

Expressions (8)–(12) describe the resonance hole formation on the Th5d level (8), followed by the system relaxation via giant Coster–Kronig transitions [(9)–(12)]. These transitions can be considered as quasi-Auger transitions. Common photoelectron emission occurs concurrently. As the spectra under consideration at resonance are associated to a lesser extent with photoelectrons and to a greater extent with quasi-Auger electrons, these spectra should reflect the partial density of Thnp and Th5f states. The observed smearing of the structure in the region from ~15 to 35 eV is apparently associated with OVMO formation in these

compounds. Because at resonance the strongest changes in intensities of various parts of the spectrum should be expected for lines associated with Th5*f*,6*p* electrons, two qualitative conclusions follow from our results. First, Th5*f* electrons directly participate in chemical bonding and are delocalized within OVMO (in the middle of the band), which leads to enhancement of the spectrum of OVMO electrons at resonance. Second, Th6*p* electrons efficiently participate in the formation of IVMO and, probably, OVMO. As a result, at resonance an intense structure reflecting the partial density of states of Th6*p* electrons appears in the region of IVMO.

At excitation energies  $h\nu$  (110, 120, and 140 eV) exceeding the resonance energy, the intensity of the REE structure arising at resonance considerably decreases. The photoionization cross sections of the shells under consideration also significantly decrease (see table). Nevertheless, the structure of the spectrum under consideration in its general features approaches the structure of the photoelectron spectrum obtained at the excitation energy  $h\nu = 70$  eV (Fig. 4). The difference is that some features of the spectrum structure arising at resonance are preserved in the spectrum taken at  $h\nu = 140$  eV. This is a fundamental experimental result. It shows that calculations of photoionization cross sections of shells in atoms should take into account the effect of the resonance on these quantities.

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