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CHEMISTRY

Yu. M. UKRAINSKII and Corresponding Member of the Academy
of Sciences of the USSR A. V. NOVOSELOVA

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Abstract

Full Text

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MOLYBDENUM AND RHENIUM DISLENIDES

MoSe₂ was first obtained by heating molybdic acid with selenium in a stream of hydrogen (¹), and ReSe₂ by decomposing Re₂Se₇ in vacuum at 325-330° (²). Judging from the literature, the crystallographic and electrical properties of these compounds have not been studied up to the present time. Since the disulfides of molybdenum and rhenium (³) have the same type of crystal structure, and MoS₂ has long been known as a semiconductor, it was of interest to study from this point of view the properties of the diselenides of molybdenum and rhenium.

In our work, the synthesis of MoSe₂ and ReSe₂ was carried out by sintering powdered molybdenum and rhenium (both of approximately 99% purity) with a stoichiometric amount of selenium (99.9% principal substance), in quartz ampoules sealed under vacuum, at 700° for 100 hours. The resulting preparations were gray powders with a metallic luster, quite stable in air. X-ray photographs of the samples were taken by the powder method in RKD-57 type cameras using copper radiation. Table 1 gives the results of indexing the powder pattern of MoSe₂. Molybdenum diselenide has a hexagonal unit cell with parameters: $a = 3.28_4$ kX; $c = 12.8_8$ kX.

Table 1

Indexing of the powder pattern of MoSe₂, Cu $K\alpha = 1.539$ kX radiation

Intensity	$\sin^2 \theta \cdot 10^4$, found	hkl	$\sin^2 \theta \cdot 10^4$, calculated	Intensity	$\sin^2 \theta \cdot 10^4$, found	hkl	$\sin^2 \theta \cdot 10^4$, calculated
8	140	002	143	5	3496	204	3499
2	565	004	571	3 sh	3833	205	3820
8	732	100	732	7	4486	118	4479
1	870	102	875	2	5131	120	5124
10 sh	1058	103	1053	2	5131	0012	5138
4	1284	006	1284	6	5445	123	5445
6	1628	105	1624	4	5757	1110	5764

	$\sin^2 \theta \cdot 10^4$, found	hkl	$\sin^2 \theta \cdot 10^4$, calculated	Intensity	$\sin^2 \theta \cdot 10^4$, found	hkl	$\sin^2 \theta \cdot 10^4$, calculated	
	10	2197	110	2196	3 sh	6014	125	6016
	5	2284	008	2283	4	6586	300	6588
	4	2342	112	2339	4	6718	302	6731
	2	2771	114	2767	3	7875	306	7872
	4	2934	200	2928	4 sh	8780	220	8784
	6	3249	203	3249	5	8878	308	8871

The pycnometric density for MoSe_2 was found to be $6.90 \pm 0.05 \text{ g/cm}^3$.

If it is assumed that in the unit cell of MoSe_2 , as in MoS_2 , there are two formula units, then the calculated density of MoSe_2 is 7.0 g/cm^3 , which agrees rather well with the experimentally found density. The majority of the lines of the MoSe_2 powder pattern

are indexed with the same indices hkl as in the powder pattern of MoS_2 . Moreover, in both cases the same extinction conditions hold for reflections of the types hhl and $00l$ for even l . All this makes it possible to regard molybdenum diselenide and disulfide as isostructural compounds.

The powder pattern of ReSe_2 has its own set of interplanar spacings, which indicates that the reaction between rhenium and selenium has gone to completion. It contains a considerably larger number of lines than MoSe_2 , and it is not possible to fit them into a unit cell of the MoS_2 type. The structure of rhenium diselenide is probably of lower symmetry.

The electrical conductivity and thermoelectric power of MoSe_2 and ReSe_2 were measured potentiometrically, by a compensation circuit.* The samples were cylinders 4 mm in diameter and 12–15 mm high, pressed from powders under a pressure of 8 T/cm^2 . Measurements of electrical conductivity were carried out in the temperature range 20–70°. At 22° the specific electrical conductivity of MoSe_2 proved to be $1.23 \cdot 10^{-4} \Omega^{-1} \cdot \text{cm}^{-1}$; with an increase in the temperature of the sample it rose noticeably and at 62° was $2.30 \cdot 10^{-4} \Omega^{-1} \cdot \text{cm}^{-1}$. For ReSe_2 at 24° it was $6.75 \cdot 10^{-5} \Omega^{-1} \cdot \text{cm}^{-1}$, and at 64° increased to $10.7 \cdot 10^{-5} \Omega^{-1} \cdot \text{cm}^{-1}$. The positive temperature coefficient of electrical conductivity and its magnitude point to the semiconducting properties of MoSe_2 and ReSe_2 , which is also confirmed by the high thermoelectric power that we found in these compounds. The thermoelectromotive force of MoSe_2 and ReSe_2 samples was measured by the integral method⁽⁵⁾, relative to alumel. The temperature difference between the ends of the sample was varied from 25 to 150°. For MoSe_2 , under these conditions the thermoelectric power correspondingly varied in the range 610–430 $\mu\text{V/deg}$, and for ReSe_2 from 1200 to 880 $\mu\text{V/deg}$. In both cases the hot probe (alumel) became negatively charged; on this basis it may be concluded

that the molybdenum and rhenium diselenides synthesized by us are *p*-type semiconductors ⁽⁵⁾.

In conclusion, we consider it our pleasant duty to express our gratitude to Yu. P. Simanov for discussion of the results of the X-ray investigation.

Moscow State University
named after M. V. Lomonosov

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* The measurement method was described by us in ⁽⁴⁾.

Note: Figure translations are in progress. See original paper for figures.

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