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Preparation of New Compound $AgTaS_3$

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A new ternary sulfide compound $AgTaS_3$ has been prepared by a sealed silica tube method at 500 °C. The crystal structure has been determined by X-ray powder and electron diffraction. It was found that $AgTaS_3$ crystallizes in the orthorhombic system, space group $Cmc2_1$ with unit cell dimensions of a=3.3755 Å, b=14.0608 Å, c=7.7486 Å.

Several ternary tantalum sulfides with formula of $MTaS_3$ (M=Pb, Sn, Bi, and Cu)[1-3] have been reported recently. All these compounds crystallize with orthorhombic symmetry and exhibit a variety of structures. $PbTaS_3$, $SnTaS_3$ and $BiTaS_3$ reveal composite layer structures[4] based on the combination of MS and TaS_2 sandwiches. $CuTaS_3$ possesses the honeycomb-like structure with large empty channels along b axis. A similar $MTaS_3$ compound could be expected for another metal systems. However, in the case of M=Ag there are no reports. Only $Ag_{1/3}TaS_2$ and $Ag_{2/3}TaS_2$ have been known so far in the Ag-Ta-S system.[5,6] We have succeeded recently in preparing the new compound $AgTaS_3$ whose X-ray diffractograms are completely different from those of other $MTaS_3$ series. In the present paper we report the results of X-ray and electron diffraction studies of $AgTaS_3$.

Tantalum (3N6), sulfur (6N) and Ag_2S (3N) powders were used as the starting materials. Initially TaS_2 was prepared from the elements at 600°C, being employed as source materials. Stoichiometric amounts of Ag_2S, TaS_2 and S were mixed in an agate mortar, pressed into pellets and sealed in an evacuated silica tube. The heat treatment was carried out at 500 °C for 4 days. $AgTaS_3$ thus obtained were identified using a Rigaku X-ray diffractometer (Geigerflex, RAD-B system) with graphite-monochromated Cu $K\alpha$ radiation. Electron diffraction patterns were taken from the crushed particles using a 100 kV electron microscope (Hitachi-500-type).

The X-ray diffraction patterns of $AgTaS_3$ were refined to give an orthorhombic unit cell with a=3.3755±0.0002 Å, b=14.0608±0.0011 Å, c=7.7486±0.0007 Å, and V=367.77±0.04 $Å^3$. The X-ray powder diffraction data

of $AgTaS_3$ are listed in Table 1. The electron diffraction patterns, taken with the incident electron beam parallel to the [010] direction and perpendicular to both [200] and [022] directions, are shown in Figs.1(a) and 1(b). From the comparison of electron diffraction patterns and X-ray powder data, the indices of observed reflections were found to obey the following conditions: hkl; h+k=2n, 0kl; k=2n, h0l; h=2n, l=2n, hk0; h+k=2n, h00; h=2n, 0k0; k=2n, 00l; l=2n. These reflection conditions clearly indicate the space group $Cmc2_1$ (No.36).

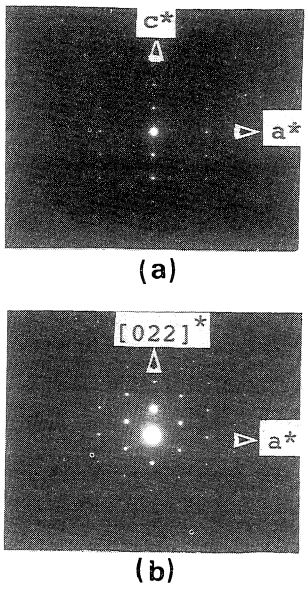

Fig. 1. Electron diffraction patterns from $AgTaS_3$. The incident beams are parallel to the [010] direction in (a) and perpendicular to both [200] and [022] directions in (b).

Table 1. X-ray powder diffraction data of $AgTaS_3$

h	k	l	d_{obs}/Å	d_{calc}/Å	I/I_o
0	2	0	7.041	7.030	37
0	2	1	5.208	5.207	8
0	0	2	3.874	3.874	5
0	4	0	3.516	3.515	53
0	2	2	3.393	3.393	100
1	1	0	3.283	3.282	10
1	1	1	3.023	3.022	27
1	3	0	2.739	2.739	30
1	3	1	2.583	2.583	44
1	1	2	2.504	2.504	7
0	2	3	2.4243	2.4244	11
1	3	2	2.2376	2.2366	14
0	4	3	2.0814	2.0814	27
1	1	3	2.0299	2.0298	29
0	6	2	2.0052	2.0052	45
0	0	4	1.9371	1.9371	15
1	5	2	1.8871	1.8870	16
1	3	3	1.8794	1.8792	24
0	8	0	1.7574	1.7576	8
1	7	0	1.7260	1.7262	7
0	8	1	1.7140	1.7141	7
0	4	4	1.6966	1.6966	15
2	0	0	1.6871	1.6878	13
1	1	4	1.6684	1.6683	7
1	5	3	1.6567	1.6572	5
2	2	0	1.6409	1.6412	3
0	8	2	1.6009	1.6006	4
1	3	4	1.5813	1.5816	5
2	0	2	1.5478	1.5473	3
2	4	0	1.5217	1.5215	5
2	2	2	1.5111	1.5111	11
1	9	1	1.3946	1.3947	6

It was found that $AgTaS_3$ is not stable on prolonged heating above 550 °C and decomposes to two-phase mixtures of the unknown cubic phase and $Ag_{1/3}TaS_2$. Experiments are now in progress to investigate the detailed structure and phase relations of silver tantalum sulfides.

References

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