

Short Communications

PHASE DIAGRAM OF THE SYSTEM $\text{PbTe}-\text{As}_2\text{Te}_3$

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The $T-x$ phase diagram of the pseudobinary system $\text{PbTe}-\text{As}_2\text{Te}_3$ was constructed from DTA data and results of X-ray diffraction analysis and electron-probe microanalysis. No new compound was found in the system $\text{PbTe}-\text{As}_2\text{Te}_3$. The phase diagram of this system is of an eutectic type with an eutectic temperature of $350 \pm 5^\circ$, the eutectic composition corresponding to 10 mole% PbTe . Two solid phases with compositions near to As_2Te_3 and PbTe , respectively, coexist in the system below the eutectic temperature. The solubility of PbTe in As_2Te_3 is smaller than 2 mole% PbTe , and that of As_2Te_3 in PbTe is smaller than 0.5 mole% As_2Te_3 at 290° .

The chemical and physical properties of a series of metal chalcogenides of elements of group IV and V of the periodic system have been intensively studied recently. Attention has also been paid to the tellurides of these groups, for example, to arsenic telluride [1, 2] and lead telluride [3]. However, the system $\text{PbTe}-\text{As}_2\text{Te}_3$ has not yet been studied.

Experimental

The samples were synthesized by the direct alloying of the parent components of 99.999% purity in evacuated ($p < 10^{-3}$ torr) quartz ampoules. The syntheses were performed for 3 hours in a furnace, with shaking to ensure homogeneity of the samples. After this the furnace was switched off and left to cool slowly to room temperature. Samples annealed for 500 hours at 290° were also investigated.

For differential thermal analysis (DTA), the samples were crushed, and batches of 1.5–2 g were put into thin-walled quartz ampoules which were then evacuated ($p < 10^{-2}$ torr) and sealed. The DTA measurements were carried out with a Paulik–Paulik–Erdey MOM Derivatograph [4]. The temperature was raised at a rate of $12^\circ/\text{min}$. Al_2O_3 was used as the reference material.

The X-ray phase analysis was made on a URS-50 IM (Burevestnik, Leningrad) apparatus by the Debye–Scherrer powder technique, using $\text{Cu}-K_\alpha$ radiation with a nickel filter. The values of the interplanar spacings were obtained from the diffractograms by using Giller's tables [5].

The homogeneity and the composition of the materials were studied by using

the JXA-5 electron-probe X-ray microanalyzer (JEOLCO, Japan). On polished samples the composition image and Pb-M_β and As-K_β X-ray images were obtained.

Results

The prepared samples were crystalline with a metallic lustre, and most of them had an electric conductivity in the range 10^{-2} – 10^{-3} ohm⁻¹ cm⁻¹.

The study of polished samples from the system PbTe–As₂Te₃ with the electron-probe microanalyzer showed the presence of two phases: α and β in a wide concentration range (3.0–99.5 mole % PbTe).

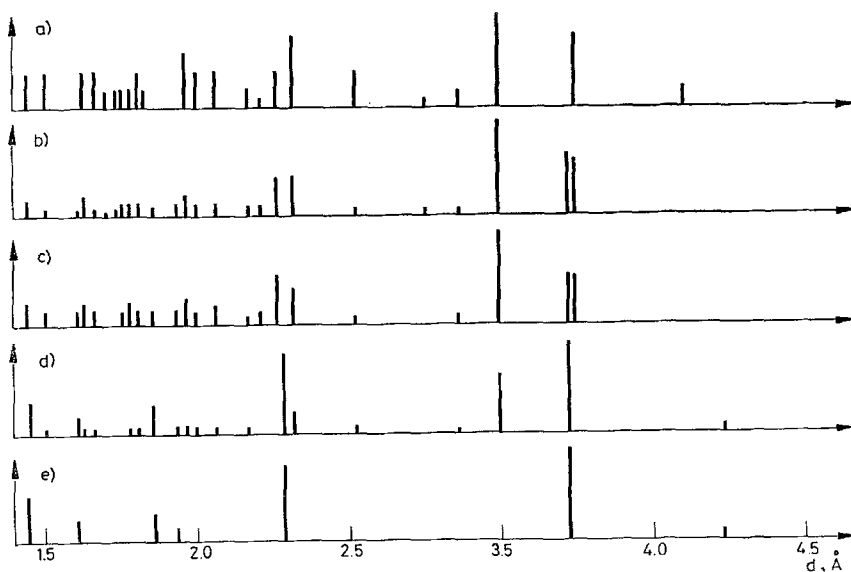


Fig. 1. Interplanar spacings of samples from the system PbTe–As₂Te₃: a) As₂Te₃; b) 20% PbTe–80% As₂Te₃; c) 40% PbTe–60% As₂Te₃; d) 70% PbTe–30% As₂Te₃; e) PbTe

X-ray diffraction measurements were made to detect the formation of possible new compounds in the system PbTe–As₂Te₃. The results, some of which are shown in Fig. 1, revealed the presence of X-ray patterns belonging to only two phases, with the spacings d identical with those of PbTe and As₂Te₃ in almost all the samples of the system. No new diffraction lines due to a new compound in the system PbTe–As₂Te₃ were found.

For most of the samples prepared, the DTA curves were characterized by two endothermic peaks. The first endothermic peak was at 350°, a temperature which, in the range 3–98 mole % PbTe in As₂Te₃, did not depend on the composition

of the samples. The area of this peak decreased with increasing PbTe content. From an analysis of the DTA results which was performed analogously to that described in the paper of Gäumann [6], the first peak corresponds to the melting of two solid-solution phases (α and β) at an eutectic temperature.

The second endothermic peak was at $350-930^\circ$, and varied with the composition of the samples. With increasing PbTe concentration, both the area of this peak and the temperature of the corresponding endothermic effect increased. From the results we have concluded the second peak belongs to the transition of a heterogeneous mixture of liquid + solid phase to the uniform liquid phase, i.e. to the liquidus curve.

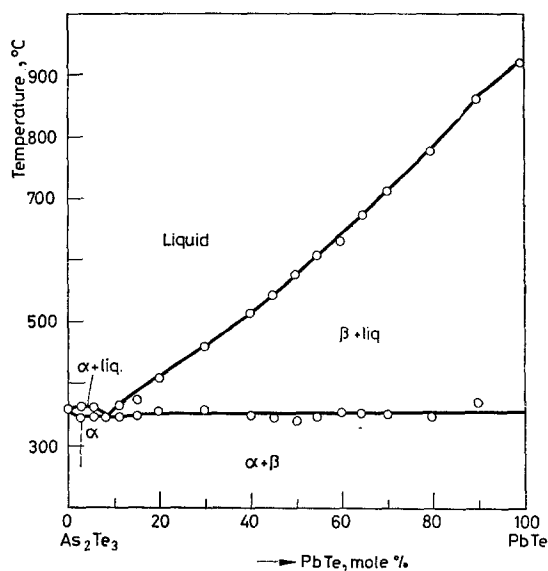


Fig. 2. Proposed phase diagram of the system PbTe- As_2Te_3

The thermal treatment of the samples at 290° for 500 hours affects neither the DTA results nor the X-ray diffraction patterns of samples from the system under study. No new compound was found in the PbTe- As_2Te_3 system and the $T-x$ diagram obtained is of an eutectic type (Fig. 2). An eutectic point occurs in the system at a PbTe concentration of 10 mole % and a temperature of $350 \pm 5^\circ$. Below the eutectic temperature, only two phases, α and β , coexist with compositions near to As_2Te_3 and PbTe, respectively. The mutual solubilities of the components were studied by using an electron-probe microanalyzer, and it was established that at 290° the solubility of PbTe in As_2Te_3 was smaller than 2 mole % PbTe, and that of As_2Te_3 in PbTe smaller than 0.5 mole % As_2Te_3 .

References

1. B. T. KOLOMIETS, L. A. ZHURAKOVSKII and A. KH. ZEINALY, *Fiz. Tekh. Poluprov.*, 5 (1971) 270.
2. S. A. DEMBOVSKII, A. I. KIRILENKO and A. S. CHVOSTORENKO, *Zh. Neorgan. Khim.*, 13 (1968) 1462.
3. MASATOMO FUJIMOTO and YASUO SATO, *J. Appl. Phys., Japan*, 5 (1966) 128.
4. F. PAULIK, J. PAULIK and L. ERDEY, *Z. Anal. Chem.*, 160 (1958) 241.
5. YA. L. GILLER, *Tablicy Mezhploskostnykh Rasstoianii*, Vol 2, Nedra, Moscow, 1966.
6. A. GÄUMANN, *Chimia Aarau*, 20 (1966) 82.