



Figure 4 66 Nylon fractograph. Draw ratio $\times 5.8(\times 18030)$.

References

1. "Fracture of Solids", edited by D. C. Drucker and J. J. Gilman (Interscience, New York, 1962).
2. E. A. TIPPETTS and J. ZIMMERMAN, *J. Appl. Polymer Sci.* **8** (1964) 2465.
3. W. L. PHILLIPS JR, *Trans. Amer. Soc. Metals* **56** (1963) 778.
4. N. J. PETCH, *J. and Steel Inst.* **173** (1953) 25.
5. A. CRACKNELL and N. J. PETCH, *Acta Met.* **3** (1955) 186.
6. J. HESLOP and N. J. PETCH, *Phil. Mag.* **1** (1956) 986.
7. G. S. YEH and P. H. GEIL, *J. Macromol. Sci. (Phy)* **B(2)** (1967) 235.
8. P. F. DISMORE and W. O. STATTON, *J. Polymer Sci.* **13** (1966) 133.

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W. L. PHILLIPS JR
 Engineering Materials Laboratory
 W. O. STATTON
 Carothers Research Laboratory
 E.I. du Pont de Nemours and Co Inc
 Wilmington, Delaware, USA

The Quasibinary System $\text{SnTe}/\text{As}_2\text{Te}_3$

A search of the technical literature failed to reveal any reports of work on the Sn/As/Te ternary system. A preliminary X-ray and metallographic survey of the ternary system, conducted in our laboratories, indicated the probable existence of several isopleths which could be quasibinary in nature. These are the following pairs: SnTe/As, SnTe/SnAs, SnTe/Sn₃As₂, and SnTe/As₂Te₃. This report presents the results of an investigation of the latter system by thermal analysis, X-ray methods, and metallographic examination.

The As₂Te₃ structure was reported to be monoclinic by Singer and Spencer [1]. According to Carron [2] the lattice parameters are: $a = 14.339 \pm 0.001$, $b = 4.006 \pm 0.005$, $c = 9.873 \pm 0.005$ Å and $\beta = 95.0^\circ$. Eifert and Peretti [3] measured the melting point of As₂Te₃ at $381 \pm 0.5^\circ$ C. The crystal-structure of SnTe is the NaCl type with $a = 6.327$ Å [4]. The melting point is 806° C.

The tellurium used in this investigation was obtained from the American Smelting and

Refining Company, South Plainfield, New Jersey, and had the following spectrographic analysis: Mg, 0.0001%; Si, 0.0001%; Fe, 0.0002%; Cu, 0.0001%; Hg, 0.0002%; and 99.999+ % Te (by difference). Mallinckrodt's (St Louis, Missouri) "analytical reagent" Sn was used; it had a lot analysis of 0.005% Zn, 0.005% Pb, 0.005% Fe, 0.001% Cu, and 0.0001% As. Arsenic was supplied in lump form by the United Mineral and Chemical Corporation of New York, New York. It had a purity of 99.99+ % and was resublimed and stored under vacuum until ready for use.

The compositions As₂Te₃ and SnTe were prepared from the elements by weighing the proper amount of each element on an analytical balance and transferring the material to a quartz tube. After evacuating and sealing off the tube, it was placed in a muffle furnace controlled at a temperature which was 50° C above the expected melting temperature and held for 30 min, during which time it was vigorously shaken. The alloy was then allowed to air-cool before use. Combinations of As₂Te₃ and SnTe were prepared in a similar manner, using the

compounds as starting material rather than the elements. Alloys used for thermal analysis weighed between 40 and 80 g.

Cooling curves were recorded with the alloys in mullite (aluminium silicate) crucibles under a protective atmosphere of dried argon. A constant rate of temperature change was controlled at one to three degrees per minute by a motorised, variable transformer in series with the furnace. A calibrated kanthal thermocouple, protected by a thin-walled mullite tube, was centred in each melt to measure the temperature while the alloy was being vigorously stirred. The emf generated by the thermocouple was recorded graphically on a Minneapolis-Honeywell extended range recorder in conjunction with a precision potentiometer. Critical points were checked with a Leeds and Northrup type K-2 potentiometer.

Satisfactory results were achieved in preparing specimens for microscopic examination by the use of conventional polishing techniques. Etching was carried out with several reagents: 1 part

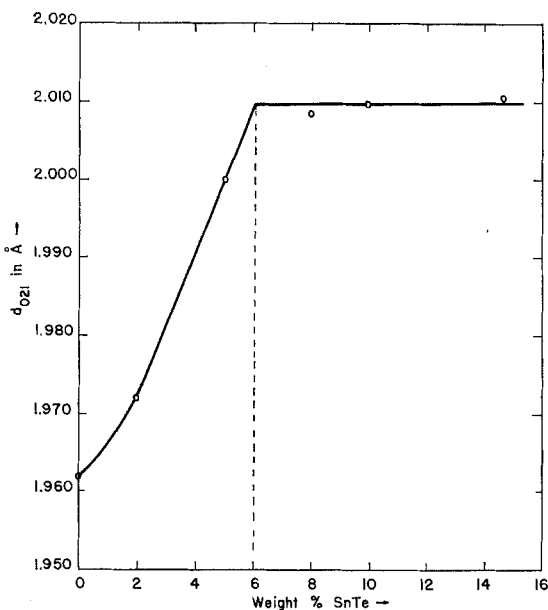


Figure 2 Variation of lattice spacing of the (021) planes in the alpha phase as a function of composition at 375°C.

FeCl₃: 1 part CrO₃: 1 part H₂O; 1 part CrO₃: 1 part H₂O: 1 part K₂Cr₂O₇; and 1 part CrO₃: 1 part Eden's reagent.

To check the results of thermal and microscopic studies Debye-Scherrer diffraction patterns were taken with characteristic chromium K_α radiation in a 57.3 mm camera employing the Straumanis technique [5].

Forty-five alloys were prepared for study. From the results of thermal analysis the resulting phase diagram shown in fig. 1 was constructed*.

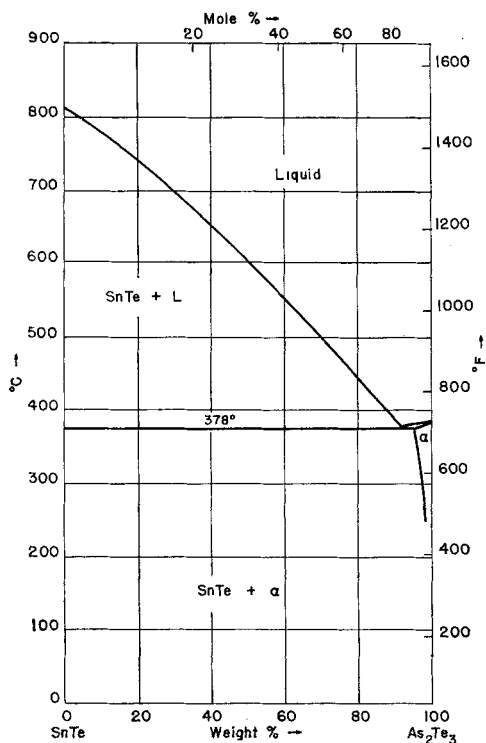


Figure 1 Phase diagram of the system SnTe/As₂Te₃.

*A table listing details of thermal arrests can be obtained from Professor Peretti on request.

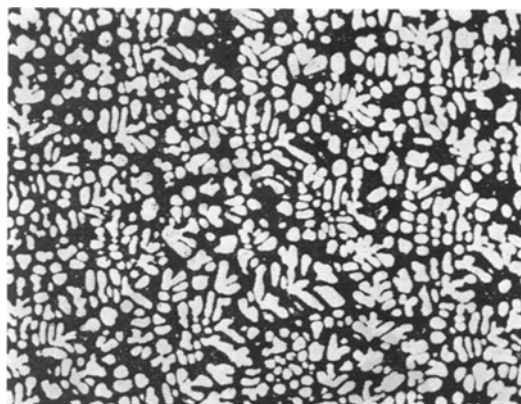


Figure 3 Microstructure of air-cooled hypoeutectic alloy containing 40 wt % As₂Te₃/60% SnTe. Primary crystals of SnTe (white) plus eutectic (black). Etchant: 1 part CrO₃: 1 part dichromate: 1 part H₂O (× 90).



Figure 4 Microstructure of furnace-cooled hypoeutectic alloy containing 50 wt % As_2Te_3 /50% SnTe. Primary crystals of SnTe (black) plus eutectic. Etchant: 1 part CrO_3 : 1 part Eden's reagent ($\times 90$).

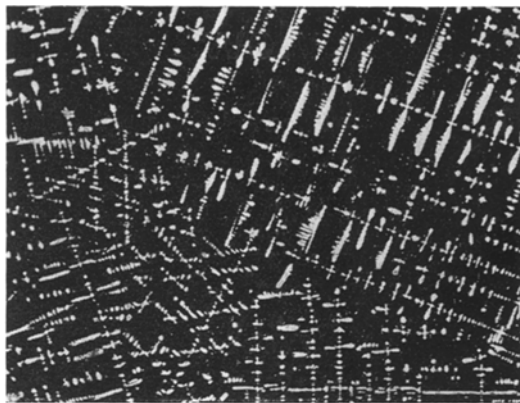


Figure 5 Microstructure of hypoeutectic alloy with 80 wt % As_2Te_3 /20% SnTe. White dendrites of SnTe plus black eutectic. Etchant: 1 part CrO_3 : 1 part dichromate: 1 part H_2O ($\times 90$).

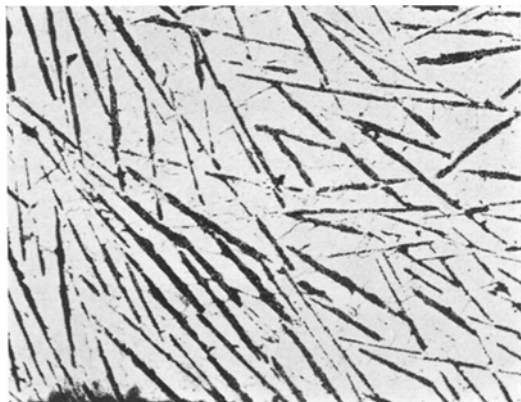


Figure 6 Microstructure of eutectic alloy with 92.2 wt % As_2Te_3 /7.8% SnTe. Etchant: 1 part CrO_3 : 1 part $FeCl_3$: 1 part H_2O ($\times 90$).

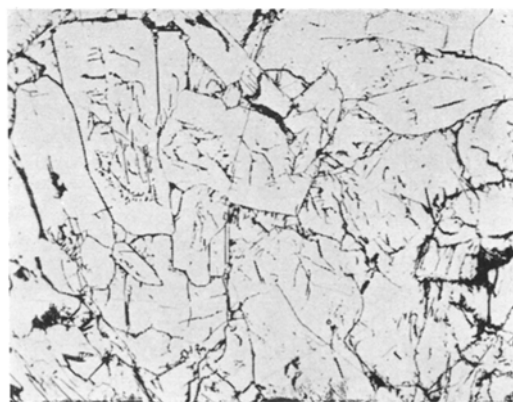


Figure 7 Microstructure of hypereutectic alloy containing 94 wt % As_2Te_3 /6% SnTe. Alpha solid solution plus eutectic. Etchant: 1 part CrO_3 : 1 part Eden's reagent ($\times 90$).

The solid solubility of As_2Te_3 in SnTe is less than 0.5% at the eutectic temperature, but As_2Te_3 dissolves 6 wt % As_2Te_3 . (See fig. 2.) Figs. 3, 4, and 5 show microstructures of hypoeutectic alloys of different compositions, which exhibit primary SnTe and eutectic. The latter is formed at 92.2 wt % As_2Te_3 /7.8 wt % SnTe. It melts at 378° C and consists of SnTe and As_2Te_3 -rich solid solution. Fig. 6 is a photomicrograph of the as-cast eutectic composition, and fig. 7 is that of a hypereutectic alloy.

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References

1. J. SINGER and C. W. SPENCER, *Trans. AIME* **203** (1955) 144.
2. G. J. CARRON, *Acta Cryst.* **16** (1963) 338.
3. J. R. EIFERT and E. A. PERETTI, *J. Materials Sci.* **3** (1968) 293.
4. A. M. RETI, A. K. JENA, and M. B. BEVER, *Trans. Met. Soc. AIME* **242** (1968) 371.
5. B. D. CULLITY, "Elements of X-ray Diffraction" (Addison-Wesley, New York, 1959).

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J. K. PAULSEN
E. A. PERETTI

Department of Metallurgical Engineering and
Materials Science, University of Notre Dame
Notre Dame, Indiana, USA