Table IV. Comparison of Solubilities at 25.0° C.

Source	Potassium Content, P.P.M.	Solubility, G./100 G. Solution
Swedish	700	19.8
Special preparation	7	19.9
(3)	Not known	19.89

Table V. Refractive Index Data

Solution Temp., ° C.	Saturation Temp., ° C.	R.I.	Concn., G./ 100 G. Soln.	R.I., Calcd.
20	20	1.3466	17.9	1.3466
25	20	1.3458	17.9	1.3458
25	25	1.3473	19.8	1.3473
25	26	1.3476	20.1	1.3476
30	25	1.3466	19.8	1.3465
30	30	1.3481	21.8	1.3481
30	31	1.3484	22.1	1.3484
35	30	1.3473	21.8	1.3473
35	35	1.3488	23.7	1.3488
40	35	1.3480	23.7	1.3480
40	40	1.3495	25.6	1.3495
45	40	1.3486	25.6	1.3487

of solution. Potassium ions, which are not removed by recrystallization because of a strong absorption effect, appear, therefore, to depress the solubility of ammonium perchlorate.

REFRACTIVE INDEX DETERMINATION

The refractive index of undersaturated, saturated, and supersaturated solutions of ammonium perchlorate was measured in the temperature range 20° to 50° C., with a Bellingham and Stanley dipping refractometer, using the makers' calibration checked at one point against distilled water at 20.0° C. A plot of the data obtained is given in Figure 2 which shows the refractive index at points on the saturation curve at a number of temperatures between 20° and 50° C. Values obtained above and below the saturation curves are also given and these values show no discontinuity in passing through the saturation point in either direction, and show a linear plot.

It is, therefore, possible to express the information in the form of an empirical equation applicable within the limits of 20° to 50° C. and supersaturation or undersaturations of at least 5° C. The equation is

$$\text{R.I.} = 1.3325 + 0.00079c - 0.00016\,(t - 20)$$

where c = solution concentration g./100 g. solution

t = solution temperature, ° C.

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The Ternary System Indium Telluride–Indium Antimonide–Antimony

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The liquidus and solidus surfaces of the system InTe-InSb-Sb have been determined by thermal analysis, metallographic, and x-ray procedures. InTe is the primary phase of precipitation over most of the system. An intermediate phase, beta, in the InTe-InSb system extends into this ternary and is stable between 511° and 420° C. At the lower temperature, the system decomposes into InTe and alpha, a solid solution of InTe in InSb. A ternary eutectic of antimony, alpha, and beta melts at 486° C. and contains 7 wt.% InTe, 59.5 wt.% InSb, and 33.5 wt.% Sb. Liquidus isotherms are presented in a figure, and a schematic space model is illustrated.

SOME ALLOYS in the InTe-InSb-Sb subternary system of the indium-tellurium-antimony system have been studied by Wooley, Gillet, and Evans (*6*). They measured the solid solubility along the InSb-In_2Te_3 section, a portion of which lies in the subternary under discussion. InSb and In_2Te_3 do not form a quasibinary section, but InTe and Sb do, as reported by Stegman and Peretti (*4*). Rosenberg and Strauss (*3*) measured the liquidus of the InSb-Te and InSb-In_2Te_3 isopleths, portions of which lie in the InTe-InSb-Sb ternary area. Goryunova, Radautson, and Kiosse (*1*) first reported the existence of a semiconducting compound on the InSb-InTe tie line; it has a sodium chloride structure and a lattice parameter of 6.128 A.

The InTe-Sb quasibinary diagram (*4*) is a simple eutectic type with very small solid solubility in either phase. The alloys form a eutectic at 571.5° C., containing 72.5 wt. % Sb and 27.5% InTe. Indium telluride melts at 696° C. and forms solid solutions with InSb, which melts at 525° C.

According to Stegman and Peretti (*5*), the intermediate phase formed by InTe and InSb has the composition 2InTe·InSb. This phase melts incongruently at 553.5° C. and decomposes below 420° C. into InTe and α(InSb). The solid solubility of InSb, in either the intermediate phase or InTe, is immeasurably small; on the other hand, InTe dissolves in solid InSb to the extent of 9.9 wt.% at 500° C.

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Table I. Thermal Data for the InTe-InSb-Sb System

Composition, Wt. %			Temperature, ° C.			
InTe	InSb	Sb	1st arrest	2nd arrest	3rd arrest	4th arrest
2.00	98.00	0.00	519.1	...	...	...
2.50	0.00	97.50	624.0	571.6	...	...
4.00	96.00	0.00	517.1	...	...	...
5.00	35.15	59.85	551.8	497.3	488.2	486.0
5.00	54.15	40.85	504.2	489.3	...	...
5.00	66.50	28.50	500.7	492.3	486.8	...
5.00	80.75	14.25	509.6	487.5	414.3	...
5.00	0.00	95.00	614.2	571.4	...	...
6.00	94.00	0.00	515.4	...	...	...
7.50	0.00	92.50	609.0	571.4	...	...
8.00	92.00	0.00	512.1	...	...	...
10.00	0.00	90.00	605.1	571.8	...	...
10.00	90.00	0.00	510.2	...	...	...
10.00	22.50	67.50	569.5	540.0	511.8	485.3
10.00	33.30	56.70	546.0	529.3	524.9	486.3
10.00	45.00	45.00	520.2	514.0	487.3	...
10.00	51.30	38.70	511.6	501.6	485.1	...
10.00	63.00	27.00	509.1	493.8	487.0	...
10.00	76.50	13.50	507.0	502.2	488.9	...
12.00	88.00	0.00	507.3	505.7	...	...
12.50	0.00	87.50	597.0	571.4	...	...
15.00	31.45	53.55	548.0	509.3	489.6	485.3
15.00	48.45	36.55	539.0	521.1	504.9	485.8
15.00	72.25	12.75	518.3	425.5	...	...
15.00	0.00	85.00	596.7	571.8	...	...
15.00	85.00	0.00	507.8	497.5	420.0	...
16.00	84.00	0.00	507.8	506.3	...	...
18.00	82.00	0.00	511.8	506.3	...	...
20.00	20.00	60.00	571.0	560.5	511.8	485.1
20.00	29.60	50.40	567.3	543.3	541.1	486.3
20.00	40.00	40.00	564.3	518.0	486.5	...
20.00	45.60	34.40	560.0	525.1	504.7	487.0
20.00	56.00	24.00	548.7	490.9	487.5	...
20.00	68.00	12.00	538.3	503.0	487.5	...
20.00	0.00	80.00	581.6	571.3	...	...
20.00	80.00	0.00	520.0	506.2	...	...
22.00	78.00	0.00	525.2	506.1	...	...
22.50	0.00	77.50	579.1	571.8	...	...
24.00	76.00	0.00	530.2	506.4	...	...
25.00	27.75	47.25	581.8	542.7	511.8	485.6
25.00	42.75	32.25	571.3	523.0	517.3	503.6
25.00	63.75	11.25	550.7	507.8	425.0	...
25.00	75.00	0.00	531.8	505.2	426.0	...
25.00	0.00	75.00	575.3	572.0	...	...
26.00	74.00	0.00	533.8	506.4	...	...
28.00	0.00	72.00	571.8	571.0	...	...
28.00	72.00	0.00	537.6	506.4	...	...
29.00	0.00	71.00	573.0	571.0	...	...
30.00	59.50	10.50	543.6	509.3	425.3	...
30.00	0.00	70.00	583.5	571.6	...	...
30.00	70.00	0.00	539.9	505.9	421.0	...
32.00	68.00	0.00	541.7	505.1	420.0	...
35.00	0.00	65.00	593.8	572.0	...	...
35.00	65.00	0.00	545.8	504.0	415.0	...
38.00	62.00	0.00	552.0	505.3	418.0	...
40.00	15.00	45.00	617.8	558.2	509.6	484.5
40.00	30.00	30.00	613.7	601.8	517.6	486.3
40.00	42.00	18.00	593.3	541.6	497.0	...
40.00	51.00	9.00	580.8	555.1	500.9	487
40.00	60.00	0.00	555.0	505.1	418.9	...
40.00	0.00	60.00	601.6	571.6	...	...
42.00	58.00	0.00	559.8	553.6	504.9	...
44.00	56.00	0.00	562.7	552.7	...	...
45.00	0.00	55.00	608.0	571.2	...	...
46.00	54.00	0.00	568.1	552.7	505.0	420.0
48.00	52.00	0.00	573.3	552.8	505.1	415.5
50.00	0.00	50.00	613.8	571.3	...	...
50.00	50.00	0.00	575.1	553.5	505.2	419.0
55.00	45.00	0.00	591.3	553.5	505.3	418.8
55.00	0.00	45.00	613.8	571.2	...	...
60.00	28.00	12.00	626.0	542.7	486.8	...
60.00	34.00	6.00	592.4	553.3	500.2	487.8
60.00	10.00	30.00	625.0	555.3	506.3	480.7
60.00	20.00	20.00	634.8	520.5	512.2	480.7
60.00	0.00	40.00	621.3	572.0	...	...
60.00	40.00	0.00	600.9	554.2	504.9	418.0
65.00	0.00	35.00	620.0	570.4	...	...
65.00	35.00	0.00	610.7	553.8	504.5	429.5
70.00	0.00	30.00	628.9	571.1	...	...
70.00	30.00	0.00	622.7	554.7	510.3	419.0
75.00	0.00	25.00	630.0	570.0	...	...
75.00	25.00	0.00	552.5	503.6	420.0	...
80.00	5.00	15.00	628.9	538.0	504.7	477.3
80.00	10.00	10.00	652.8	523.8	508.7	480.2
80.00	14.00	6.00	648.9	552.8	542.9	484.7
80.00	17.00	3.00	650.0	553.0	498.0	487.0
80.00	0.00	20.00	644.7	570.0	...	...
80.00	20.00	0.00	641.3	554.4	506.5	420.5
83.40	0.00	14.60	645.0	566.0	...	...
85.00	15.00	0.00	646.0	553.2	...	...
90.00	0.00	10.00	665.3	563.3	...	...
90.00	10.00	0.00	666.4	554.4	418.8	...
95.00	0.00	5.00	672.3	550.0	...	...
95.00	5.00	0.00	679.0	552.0	...	...

and 3 wt.% at 488° C. The intermediate phase and InSb form a eutectic at 506.4° C. and 86.5 wt.% InSb.

The portion of the indium-antimony diagram (2) of interest here exhibits a eutectic which consists of Sb and InSb, melts at 494° C., and contains 41.5% Sb and 58.5% InSb.

EXPERIMENTAL

The antimony (Bunker Hill Co.) had the following lot analysis: lead 0.0001%, copper 0.0001%, iron 0.0001%, arsenic 0.0001%, and remainder antimony. The indium was obtained on loan from the Indium Corporation of America and had a guaranteed purity of 99.97+% with the following impurities: copper 0.002%, lead 0.006%, tin 0.010%, and zinc 0.01%. The tellurium, purchased from the American Smelting and Refining Co., had the following spectrographic analysis: magnesium 0.0001%, silicon 0.0001%, iron 0.0001%, aluminum 0.0002%, and remainder tellurium.

All compositions studied were made by sealing into evacuated quartz tubes the required amount of each element which had been weighed on an analytical balance to produce an ingot of the desired size and composition. To ensure complete dissolution, each alloy was held at a temperature of 750° C. for 30 minutes with frequent agitation. This was followed by a water quench to give a solid ingot for analysis or heat treatment. For thermal analysis, the alloys

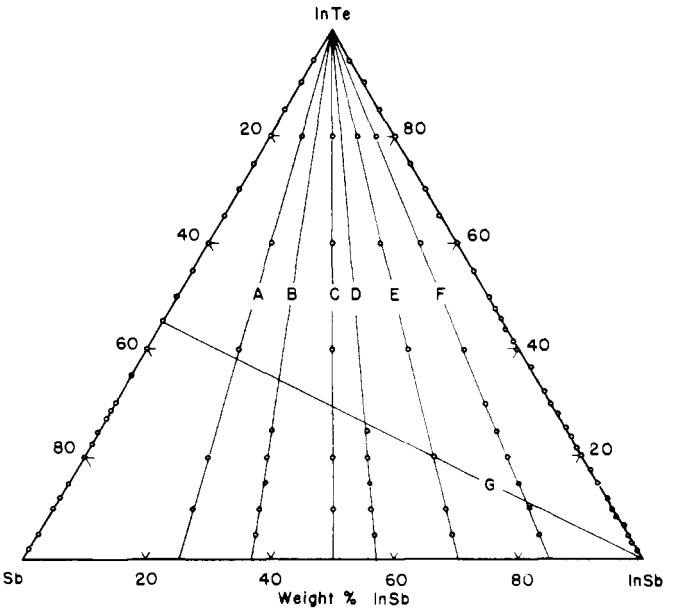

Figure 1. Location of alloys studied

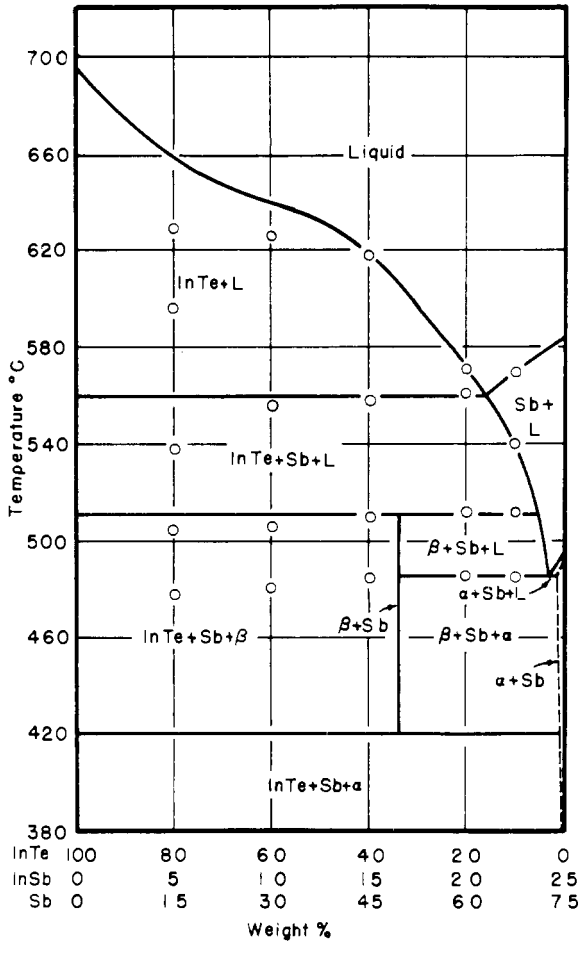

Figure 2. Isopleth A (see Figure 1)
Sb:InSb = 3:1

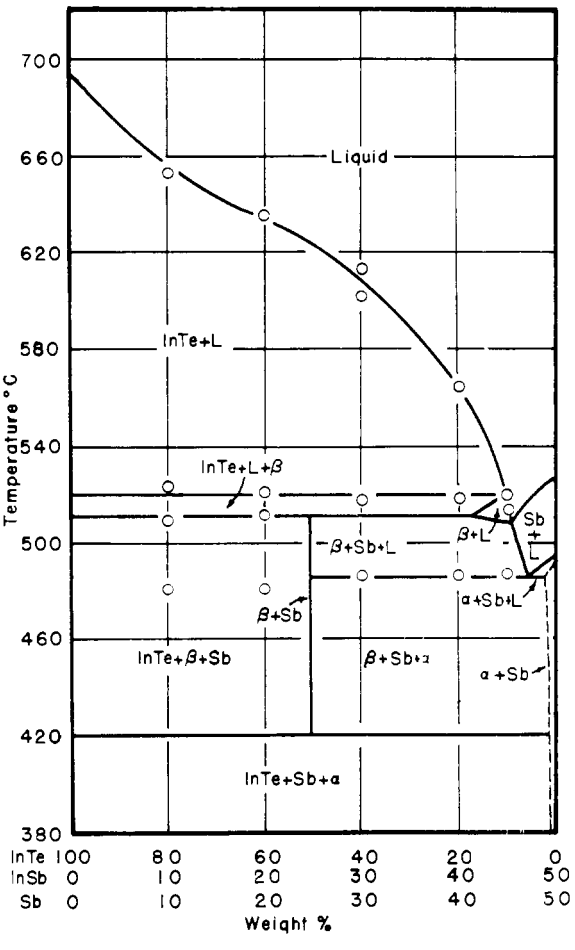

Figure 4. Isopleth C (see Figure 1)
Sb:InSb = 1:1

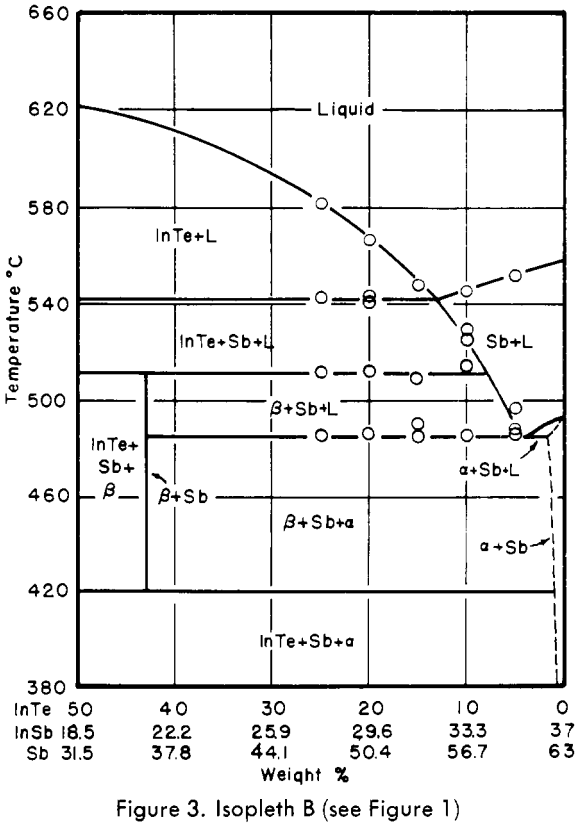

Figure 3. Isopleth B (see Figure 1)
Sb:InSb = 63:37

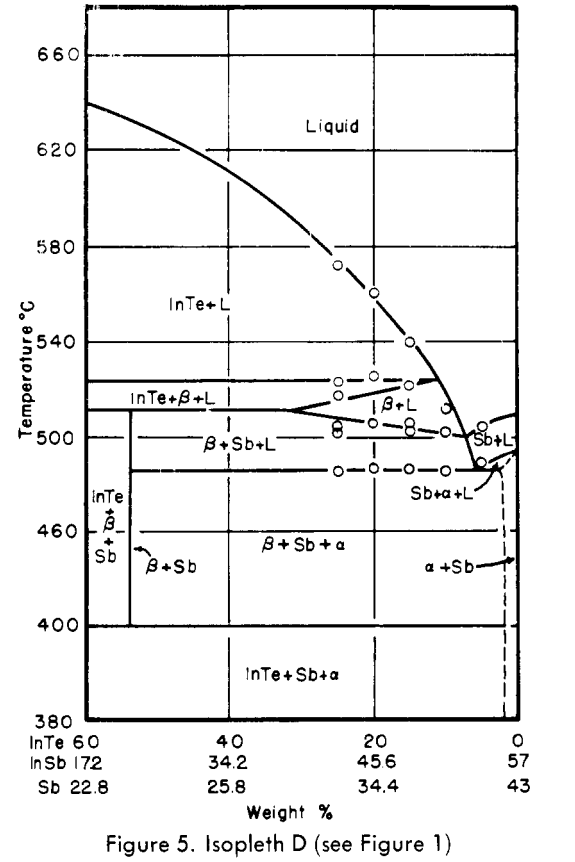

Figure 5. Isopleth D (see Figure 1)
Sb:InSb = 43:57

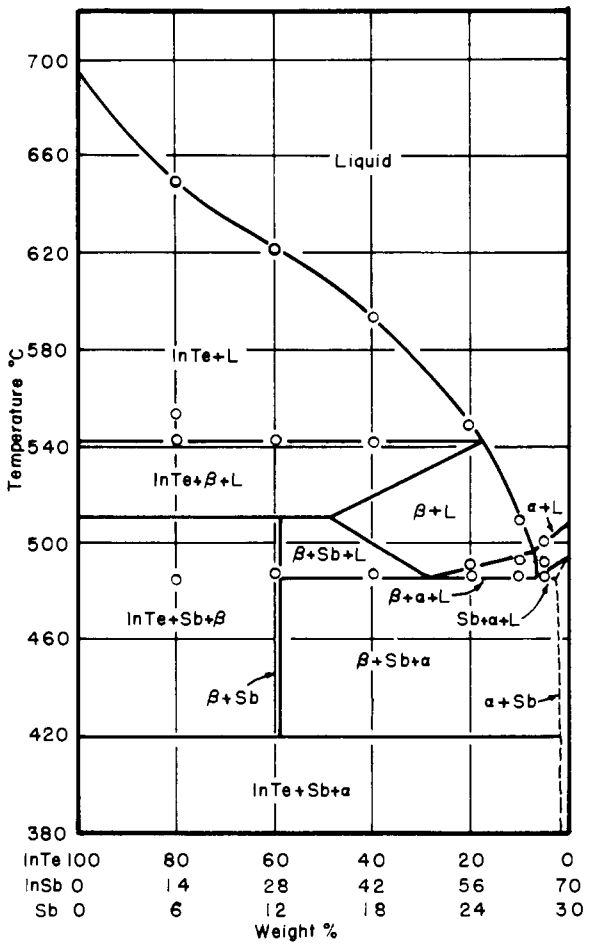

Figure 6. Isopleth E (see Figure 1)
Sb:InSb = 3:7

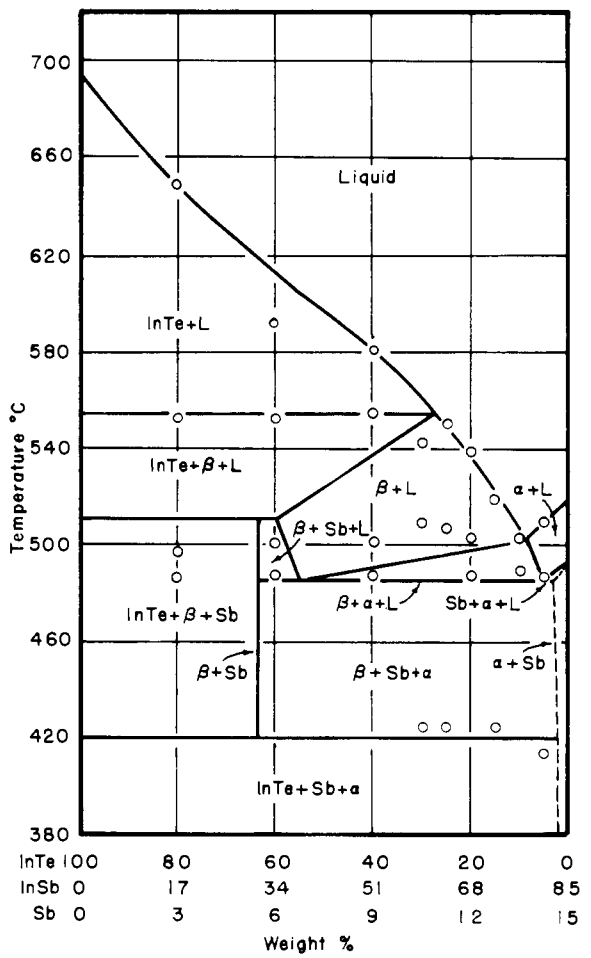

Figure 7. Isopleth F (see Figure 1)
Sb:InSb = 3:17

weighed from 70 to 110 grams, and the samples for microscopic and x-ray studies contained 5 grams of material.

For thermal analysis the alloy to be studied was placed either in a borosilicate tube, encased in a graphite crucible to prevent collapse at the higher temperatures, or in a mullite crucible encased in a mullite tube. A transite, graphite, or mullite cover was provided with holes for a chromel-alumel thermocouple, stirring rod, and gas-inlet tube. A small flow of nitrogen or argon was maintained to prevent oxidation during cooling. Each melt was stirred vigorously during the course of a cooling experiment by a motor-driven quartz or mullite rod. The thermocouple e.m.f. was measured and recorded by a Honeywell Elektronic extended range recorder in conjunction with a Leeds and Northrup precision potentiometer. The cooling was controlled by a variable transformer driven by a suitably geared motor to give a rate of 2° to 4° C. per minute. The thermocouples used were calibrated against the melting points of indium, tin, bismuth, lead, and antimony. The accuracy of temperature measurement is better than ±0.3° C.

Equilibration of specimens for metallographic and x-ray analysis was carried out in glass or quartz tubes for periods of time up to 3 months. The usual polishing techniques were employed; the final operation was performed on a vibratory polisher with a suspension of Linde "B" alumina. The etching properties of the alloys varied considerably, depending upon composition and heat treatment. The most satisfactory etchants were: a 2.5-to-1 mixture of 30% acetic acid and 10% ammonium persulfate, a 6% $FeCl_3$–22% HCl water solution, a 7% I_2–21% KI–water solution, and a 1% I_2–5% citric acid–ethanol solution. X-ray studies were made with a 114.6-mm. diameter Debye-Scherrer camera with nickel-filtered copper radiation. The metallographic

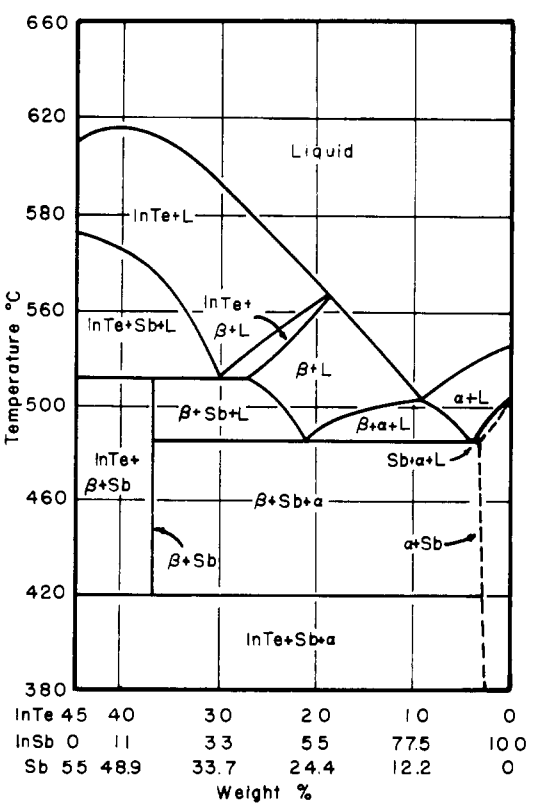

Figure 8. Isopleth G (see Figure 1)
Sb:InTe = 6.1:5

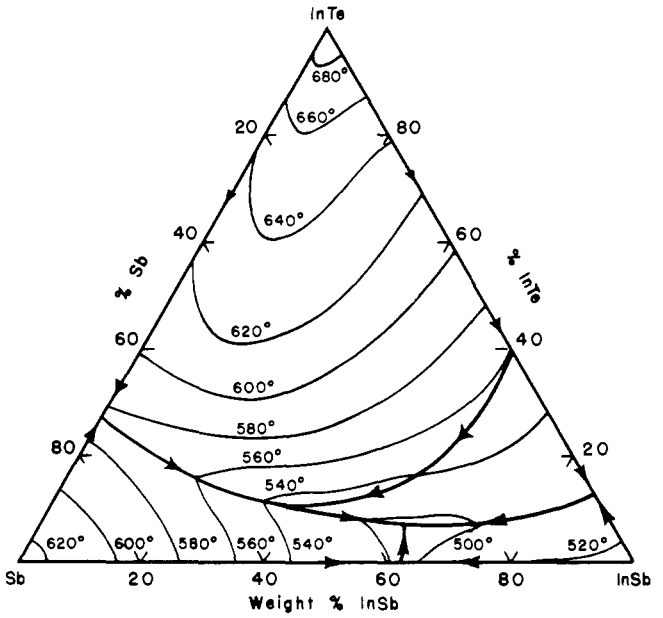

Figure 9. The liquidus surface of the InTe-InSb-Sb system

and x-ray studies were used to confirm the conclusions reached by thermal analysis regarding the number and composition of phases in the solid state.

RESULTS

Compositions were studied according to the plan shown in Figure 1; the results of thermal analysis are given in Table I and plotted graphically in Figures 2 to 9. Figures 2 to 7 show the isopleths with a constant ratio of InSb to Sb, and Figure 8 depicts a typical section with constant InTe to Sb ratio. Deviations of experimental data from the lines drawn in the figures are due to supercooling where the liquidus is involved and to a lack of attainment of equilibrium in the other areas. Peritectic reactions are normally too sluggish to achieve complete equilibrium while cooling at practical rates. The liquidus surfaces derived from the data and Figures 2 to 8 are shown in Figure 9. In Figure 10 a schematic space model of the subternary system is depicted.

InTe is the primary phase of crystallization over the major compositional area of the diagram. The peritectic reaction in the InTe-InSb binary system at 553.5° C., InTe + liquid $\rightleftarrows$ beta, in which the liquid contains 60 wt. % InSb and 40 wt. % InTe manifests itself, also, in the ternary. The loci of points of the liquid composition for this peritectic in the ternary intersects a binary InTe-Sb eutectic valley at a temperature of 511° C. and at a composition of 10 wt.% InTe, 43 wt.% InSb, and 47 wt.% Sb.

A ternary eutectic is formed at 7 wt.% InTe, 59.5 wt.% InSb, and 33.5 wt.% Sb; it melts at 486° C., and consists of the following phases: InTe, β(2InTe·InSb) and a solid solution, α, of InTe in InSb. The intermediate compound, β, decomposes below 420° C. into InTe and alpha.

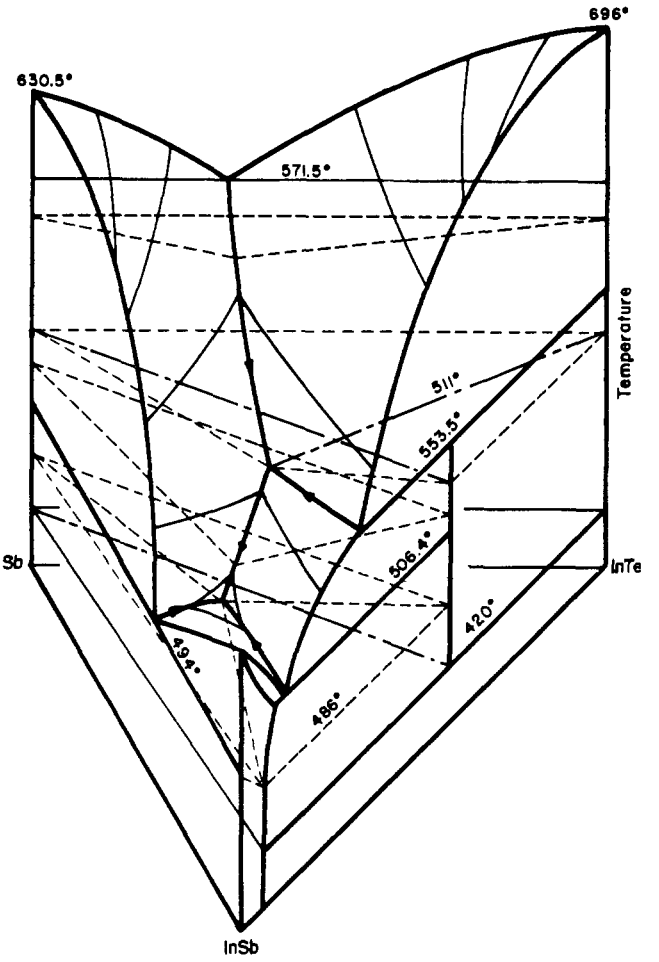

Figure 10. Space model of the InTe-InSb-Sb system

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