The Ternary System Indium–Lead–Indium Antimonide

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The liquidus and solidus surfaces of the system In–Pb–InSb have been determined by thermal analysis, metallographic, and x-ray procedures, InSb is the primary phase of precipitation over most of the system. A binary eutectic valley drops from the InSb–Pb eutectic at 298.5° C. to the InSb–In eutectic at 155° C. Solid alloys at equilibrium consist of either InSb plus alpha, InSb plus alpha and beta, InSb plus beta, InSb plus beta and gamma or InSb plus gamma, depending upon composition. Alpha and gamma are the In-rich and Pb-rich terminal, solid solutions, respectively, and gamma is an intermediate solid solution in the In–Pb system. Liquidus isotherms for the In–Pb–Sb system also are given in a figure.

PRELIMINARY WORK in the indium-antimony-lead system performed in our laboratories revealed that indium antimonide and lead form a true quasi-binary section, and therefore, the system can be conveniently divided into two subternaries for study: lead-antimony-indium antimonide and indium-lead-indium antimonide. Phase relationship determinations in the subternary Pb-Sb-InSb (*3*) have been published recently. This article presents the results of an investigation, by thermal analysis, x-ray methods, and metallographic examination of the indium-lead-indium antimonide subternary system.

Figure 1 shows the three bordering binary systems involved and the location of alloys studied. The InSb-indium binary diagram, according to Liu and Peretti (*4*), shows the existence of a eutectic reaction occurring at 154.8° C. and 0.7 weight % antimony. The compound InSb melts at 525° C. and has no appreciable solid solubility for indium; and there is very little solid solubility of InSb in indium.

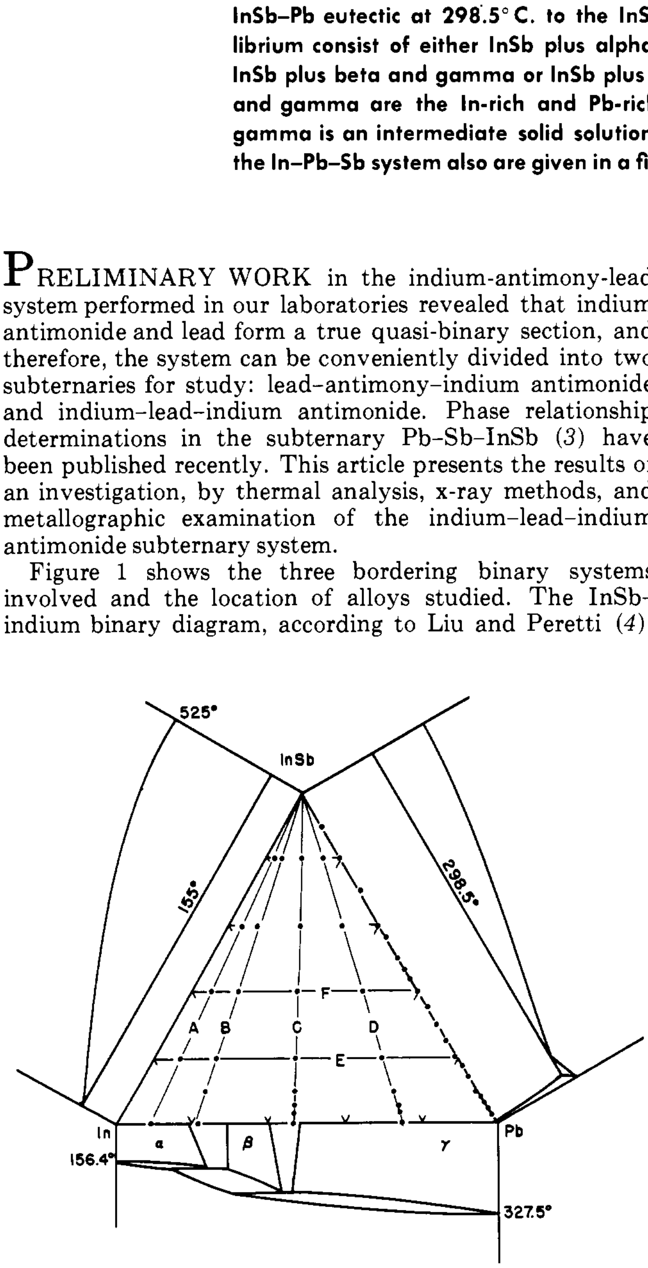

Figure 1. Bordering binary systems and location of alloys studied

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The indium-lead binary system (*1*) exhibits two peritectic horizontals occurring at 173.6° C. and 159.4° C. The existence of an intermediate phase beta, which would be predicted by the occurrence of the two peritectic horizontals, was confirmed by x-ray analysis. The compositional range of this phase as well as the extent of the primary solid solubilities has not been accurately determined. From x-ray analysis, the intermediate phase has been found to be face-centered tetragonal.

The InSb-lead diagram is from the work of the Geis and Peretti (*2*) and shows the existence of a eutectic reaction at 93.5 weight % lead at 298.5° C. X-ray, microscopic analysis show that there is no appreciable solid solubility on the InSb side and only very slight solubility on the lead side of the diagram.

EXPERIMENTAL

The indium obtained on a loan from the Indium Corporation of America had a guaranteed purity of 99.97+ % with the following chemical analysis: copper 0.002%, lead 0.006%, tin 0.010%, and zinc 0.010%.

The lead (American Smelting and Refining Co.) had the following spectrographic analysis: bismuth 0.0008%, copper 0.0003%, iron 0.0002%, and lead 99.99+ % by difference.

The antimony was obtained from two sources, the American Smelting and Refining Co. and the Bunker Hill Co. By spectrographic analysis, the metal from the first source contained the following: magnesium 0.0001%, lead 0.001%, silicon 0.0001%, iron 0.0001%, nickel 0.0040%, bismuth 0.008%, copper 0.0010%, arsenic 0.0010% and antimony 99.99+ % by difference. The Bunker Hill antimony had the following analysis: lead 0.0001%, copper 0.0001%, iron 0.0001%, and arsenic 0.0001%.

All alloys were prepared by weighing the desired amount of each element on an analytical balance in a borosilicate glass tube. The tube was then evacuated, sealed off, and placed in a muffle furnace controlled at a temperature well above the melting point of the alloy. While in the molten state, the alloy was vigorously shaken to ensure homogeneity. After about 30 minutes, the alloy was removed from the furnace, air cooled, reweighed, and placed in a borosilicate test tube for the taking of the cooling curves.

The temperatures of the alloys were measured by a calibrated Chromel-Alumel thermocouple which was protected from the molten alloy and centered in the liquid by a thin-walled quartz tube. Since the EMF produced by the Chromel-Alumel thermocouple was often greater than the range (10.2 mv.) of the Honeywell Extended Range Recorder used for recording the EMF, an external potentiometer was used as an auxiliary.

All cooling curves were taken with mechanical stirring of the liquid under a protective atmosphere of purified and preheated nitrogen or argon gas at cooling rates of from two to four degrees per minute.

Samples for microscopic analysis were rough-polished in the conventional manner, but the final polish was carried out on a Fisher Vibratory Polisher. The best etchant for InSb-rich specimens was found to be a solution of 5ml. of 30% acetic acid and 2ml. of a solution of 10% ammonium persulfate. For samples high in lead content many different etchants were tried, and a solution of acetic acid and hydrogen peroxide was the most successful. For alloys high in indium concentration, a solution of chromic anhydride, dichromate, and water in varying proportions was used. Debye–Scherrer x-ray patterns were taken in a 114.6-mm. diameter camera with copper K-alpha radiation.

RESULTS

The results of the thermal analyses carried out according to the plan shown in Figure 1 appear in Table I and are plotted graphically in Figures 2–7. These diagrams show that the precipitation of InSb as the primary phase predominates over most of the system. In Section *A* (Figure 2) and *B* (Figure 3), the point where the eutectic valley is crossed occurred at somewhat less than 1% InSb and could not be accurately determined. Therefore, the inserts on these two sections are ideally drawn rather than experimentally measured. Figures 6 and 7 show typical sections at constant InSb content, and Figure 8 shows some liquidus isotherms for the system.

Table I. Thermal Data For the In–Pb–InSb System

Alloy, Wt. %			Temperature, ° C.		
Pb	In	InSb	1st arrest	2nd arrest	3rd arrest
75.0	25.0	0.0	295.8	288.0	...
47.0	53.0	0.0	190.0	175.0	...
21.0	79.0	0.0	173.3	159.5	...
10.0	90.0	0.0	291.3	157.8	...
2.5	0.0	97.5	321.6	285.5	...
45.6	51.4	3.0	202.8	...	173.5
93.7	1.3	5.0	258.3	...	214.8
95.0	0.0	5.0	309.6	294.3	...
94.0	0.0	6.0	300.8	297.3	...
44.2	49.8	6.0	294.8	...	178.8
93.5	0.0	6.5	298.6	298.1	...
92.5	0.0	7.5	310.5	298.6	...
90.0	0.0	10.0	336.9	298.6	...
67.5	22.5	10.0	364.0	247.0	...
42.3	47.7	10.0	320.3	...	183.8
18.9	71.1	10.0	298.8	160.8	157.5
86.0	0.0	14.0	354.6	298.0	...
82.0	0.0	8.0	371.5	299.0	...
60.0	20.0	20.0	413.3	255.8	198.0
37.6	47.4	20.0	386.3	...	178.8
16.8	63.2	20.0	353.5	159.5	155.5
8.0	72.0	20.0	344.0	156.0	154.8
75.0	0.0	25.0	393.1	...	...
70.0	0.0	30.0	411.8	298.3	...
65.0	0.0	35.0	426.1	298.8	...
60.0	0.0	40.0	435.5	299.0	...
45.0	15.0	40.0	457.5	258.5	205.0
28.2	31.2	40.0	447.3	202.3	...
12.6	47.4	40.0	428.5	161.3	155.0
6.0	54.0	40.0	431.3	158.8	153.3
55.0	0.0	45.0	447.3	298.5	...
52.5	0.0	47.5	450.0	298.6	...
50.0	0.0	50.0	472.1	298.3	...
40.0	0.0	60.0	484.2	299.4	...
10.0	30.0	60.0	484.0	258.8	212.0
18.8	21.2	60.0	486.0	201.0	...
8.4	51.6	60.0	488.0	163.8	...
4.0	36.0	60.0	482.5	156.0	...
30.0	0.0	70.0	491.5	299.0	...
20.0	0.0	80.0	508.8	303.3	...
5.0	15.0	80.0	508.0	256.8	203.0
9.4	10.6	80.0	506.5	202.0	...
4.2	15.8	80.0	518.0	161.0	...
2.0	18.0	80.0	514.0	155.8	...
10.0	0.0	90.0	521.8	298.0	...

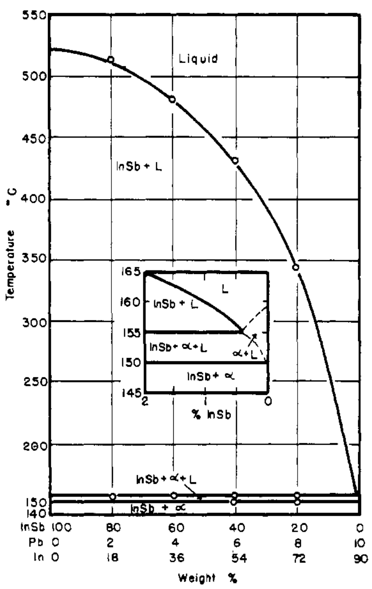

Figure 2. Isopleth A (See Figure 1), In:Pb = 9:1

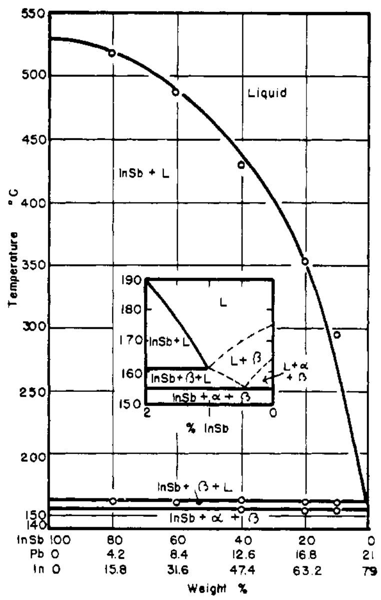

Figure 3. Isopleth B (See Figure 1), In:Pb = 79:21

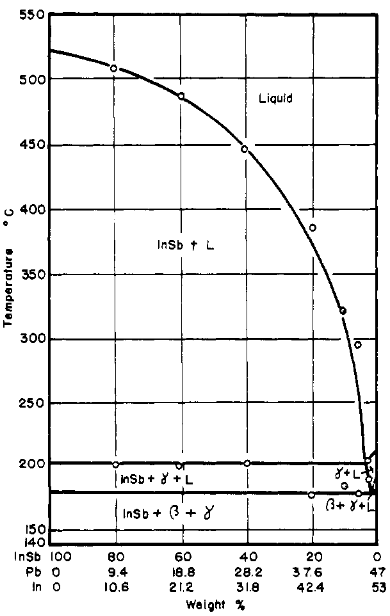

Figure 4. Isopleth C (See Figure 1), In:Pb = 53:47

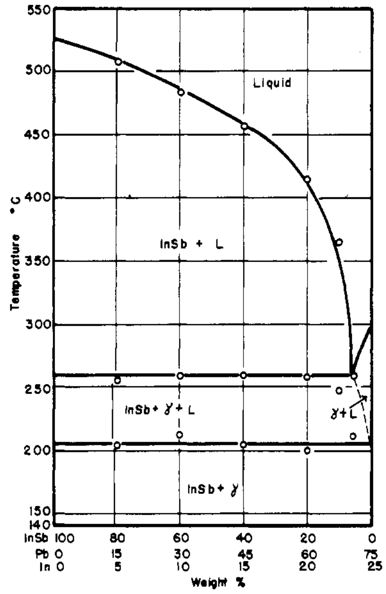

Figure 5. Isopleth D (See Figure 1), In:Pb = 1:3

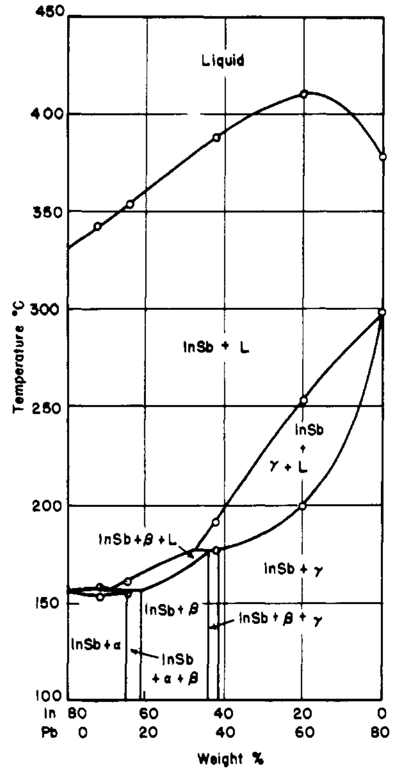

Figure 6. Isopleth E (See Figure 1), % InSb = 20

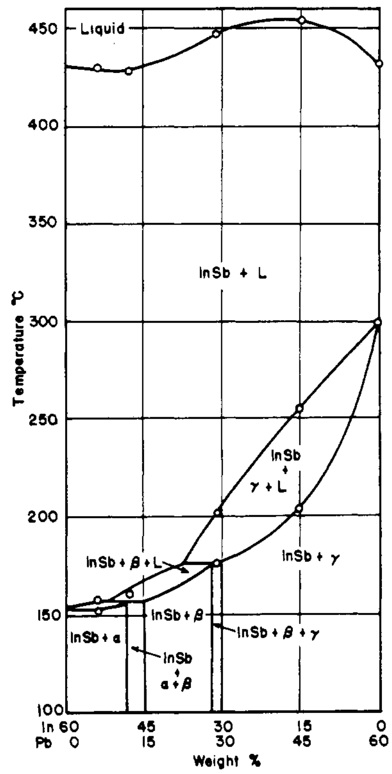

Figure 7. Isopleth F (See Figure 1), % InSb = 40

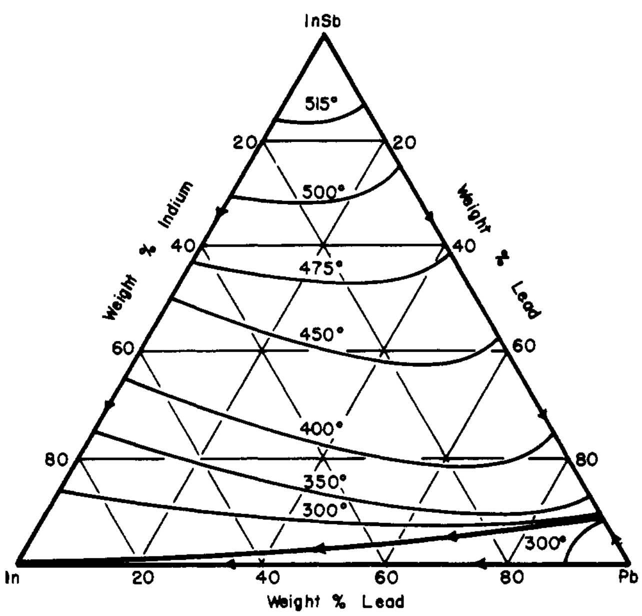

Figure 8. Liquidus surface of the In–Pb–InSb system

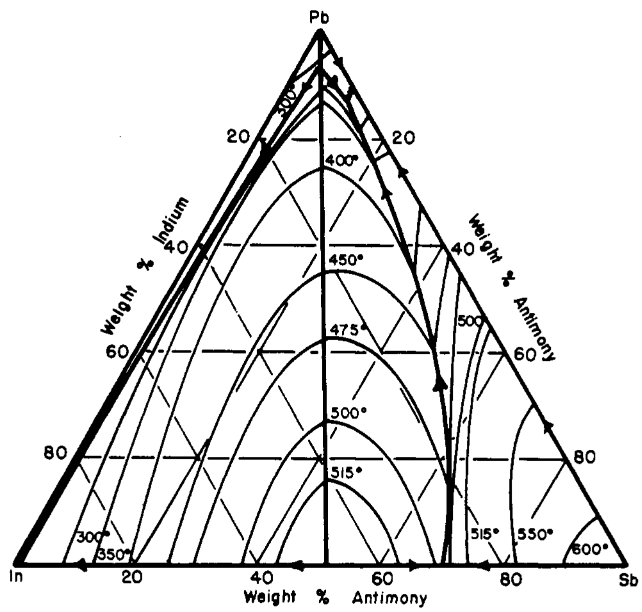

Figure 9. Liquidus surface of the In–Pb–Sb system

Solid alloys at equilibrium consist of either InSb plus alpha, InSb plus alpha and beta, InSb plus beta, InSb plus beta and gamma, or InSb plus gamma, depending upon composition.

Combining the data previously reported for the InSb–Pb–Sb system with that of the In-Pb-InSb subternary results in Figure 9 for the In–Pb–Sb ternary system.

ACKNOWLEDGMENT

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