

Phase Diagram of the System InBi-Sn

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The phase diagram of the system InBi-Sn has been determined by thermal analysis, metallographic, and x-ray procedures. A eutectic is formed at $81^\circ \pm 0.2^\circ \text{C}$., containing $16.3 \pm 0.1\%$ Sn and 83.7% InBi. The solid solubility of Sn in InBi is less than 0.1%. Sn dissolves 42% InBi at 81°C . and 20% at 71°C .

AN X-RAY and microscopic study of portions of the ternary system In-Bi-Sn showed that the InBi phase and Sn solid solutions coexisted over most of the compositional range of the InBi-Sn pair and that the two formed a quasi-binary section. The nature of this phase diagram was established by thermal analysis, x-ray, and microscopic examination.

EXPERIMENTAL

Materials. The indium (on loan from the Indium Corporation of America) had a guaranteed purity of 99.97 + % by chemical analysis. The bismuth (American Smelting and Refining Co.) contained 0.0004% Ag and less than 0.0001% each of Mg, Pb, Fe, and Cu. Baker's "analyzed reagent" tin was used; it had a lot analysis of 0.00001% As, 0.0002% Cu, 0.0005% Fe, 0.0003% Pb, and 0.0005% Zn.

Procedure. Alloys were made by melting the proper combinations of the three elements under a cover of mineral oil in a borosilicate glass tube and stirring with a glass rod.

Cooling and heating curves were taken with the alloys under a protective layer of mineral oil with mechanical stirring at a temperature change of 2° to 4°C . The temperature was measured with a calibrated chromel-alumel thermocouple protected from the molten liquid and centered in the crucible by a thin-walled quartz tube. Time-temperature curves were automatically traced on a Honeywell Range Recorder.

Conventional techniques proved satisfactory for the polishing of samples for microscopic examination. Hydral (a 2% solution of HCl in ethyl alcohol) and a mixture of Hydral and Vilella's reagent (1 gram picric acid: 5 ml. HCl: 100 ml. ethyl alcohol) were used as the etching solutions. The Hydral darkens InBi, but not the Sn; and the mixture (about 1:1) blackens Sn, but not InBi.

X-ray diffraction patterns were taken in a 114.6-mm. Debye-Scherrer camera with nickel-filtered copper radiation at 30 KVP and 10 ma. at exposure times of $1\frac{1}{2}$ to 3 hours.

The alpha solidus line was established by taking heating curves of alloys containing 75, 80, 85, and 95% Sn which had been heated for 30 days at 71°C .

The alpha solvus was partially determined by heat treating alloys for 30 days at 70°C . in the composition range 45 to 95% Sn. After being quenched in ice water, the samples were examined microscopically and with x-ray diffraction.

RESULTS

Table I lists the arrest points obtained by thermal analysis. The phase diagram of the system InBi-Sn is shown in Figure 1. The InBi-Sn pair forms a eutectic at $16.3 \pm 0.1\%$ Sn and 81°C . The eutectic composition was located at the intersection of the liquidus lines and by microscopic examination of a series of alloys in the eutectic area which varied by 0.1% Sn. These were ex-

Table J. Thermal Data for InBi-Sn System

Wt. %, Sn	Mole %, Sn	Liquidus, °C.	Solidus, °C.
2.00	5.25	105.8	78.0
4.00	10.18	103.3	80.0
6.00	14.82	100.5	81.0
8.00	19.15	96.5	81.0
10.00	23.25	91.5	81.0
12.00	27.09	87.0	81.0
15.00	32.49	84.3	81.6
16.50	35.04	82.3	81.4
20.00	40.52	87.3	81.2
25.00	47.62	100.3	80.8
30.00	53.90	112.5	80.8
35.00	59.49	122.8	79.8
40.00	64.52	133.8	78.5
45.00	69.06	144.5	77.0
50.00	73.17	153.5	75.6
55.00	76.93	162.5	75.2
60.00	80.36	170.8	74.0
65.00	83.52	179.3	...
70.00	86.42	186.8	...
75.00	89.11	194.3	139.5 ^a
80.00	91.61	201.5	157.0 ^a
85.00	93.92	207.5	173.2 ^a
90.00	96.09	217.5	...
95.00	98.11	224.8	213.0 ^a

^a Values from heating curves of homogenized specimens.

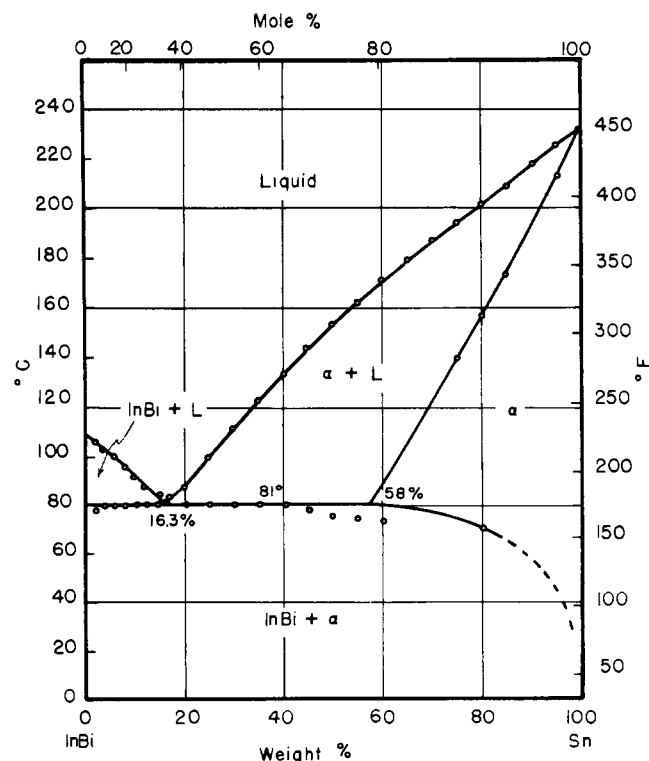


Figure 1. The InBi-Sn phase diagram

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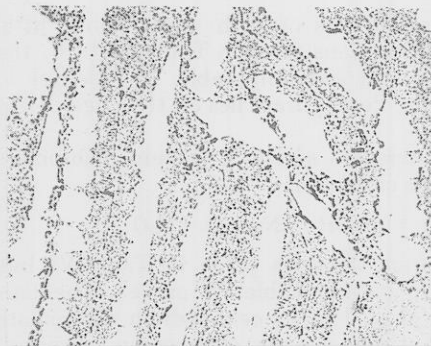


Figure 2. Microstructure of alloy containing 90% InBi-10% Sn, air-cooled. Primary InBi (white) and eutectic. Etching solution: Vilella-Hydal (X100)

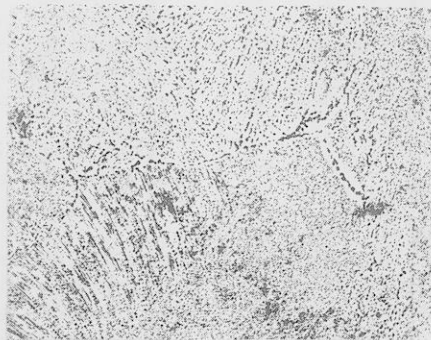


Figure 3. Microstructure of eutectic alloy: 83.7% InBi-16.3% Sn, air cooled. Etching solution: Vilella-Hydal (X 100)

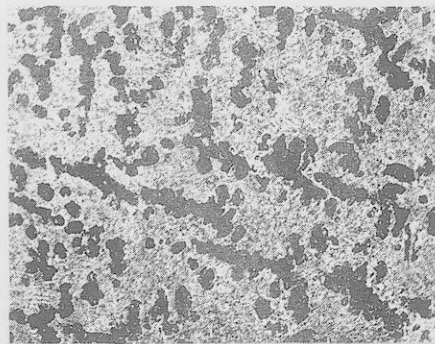


Figure 4. Photomicrograph of alloy containing 75% InBi-25% Sn, air cooled. Primary alpha (black) and eutectic. Etching solution: Vilella-Hydal (X150)

amined for the disappearance of the primary phase, approaching from both sides. Figures 2, 3, and 4 illustrate the different etching characteristics of the InBi and alpha phases. Figure 2 is a photomicrograph of an alloy which contains primary InBi and eutectic; Figure 3 is of the eutectic composition; and Figure 4 shows primary alpha and eutectic.

The solid solubility of Sn in InBi is so small that it could not be detected, but the solubility of InB is large—42% InBi at the eutectic temperature and 20% at 71° C.

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Phase Diagrams for the $\text{Na}_2\text{SO}_4\text{-Na}_2\text{Cr}_2\text{O}_7\text{-H}_2\text{O}$ System

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Solubility data have been obtained for the $\text{Na}_2\text{SO}_4\text{-Na}_2\text{Cr}_2\text{O}_7\text{-H}_2\text{O}$ system. Triangular phase diagrams have been plotted for a temperature range of 22.0 to 90° C. and a pH range of 2.0 to 10.0.

SOLUBILITY DATA have been obtained for sodium sulfate and sodium dichromate in water. This system was studied at temperatures from 22.0 to 90.0° C. and at pH's from 2.0 to 10.0. When the pH is increased above 7, sodium dichromate is converted to sodium chromate and the system is then one of chromate-sulfate and water.

EXPERIMENTAL PROCEDURES

Apparatus. Samples were allowed to reach equilibrium in a shaking, constant temperature bath. The bath was a Model 2156-1 temperature-controlled water bath shaker, manufactured by Research Specialties Co., Richmond, Calif.

Reagents. Mallinckrodt Analytical reagent grade sodium sulfate and sodium dichromate were used.

Sample Preparation and Sampling. Saturated solutions containing the salt used for the solid phase were placed in rubber-stoppered Erlenmeyer flasks and weighed quantities of the other salt and an excess of the solid phase salt were added. The flasks were placed in the bath. Mineral oil was used as the bath liquid, and the temperature was maintained at $\pm 0.5^\circ\text{C}$.

After 3 days, aliquots were analyzed. Subsequently, aliquots were analyzed every two days until equilibrium was attained.

Chemical Analyses. The sodium sulfate was determined gravimetrically by precipitation as barium sulfate (1). Chromium (VI), which interferes with this method by precipitating out as barium chromate, was removed by reduction to chromium (III) with hydroxylamine hydrochloride in dilute hydrochloric acid. Chromium (III) is soluble under the conditions used in precipitate barium sulfate.

The sodium dichromate was determined gravimetrically by precipitation as the chromium (III) oxide (2). The chromium (VI) was reduced to chromium (III) in acid solution with hydroxylamine hydrochloride. The hydrated oxide was precipitated on the addition of ammonium hydroxide and converted to the oxide by ignition at 850° C.

The degree of hydration of the solid phases was determined from the loss of weight incurred when the sample was heated to 110° C. for 2 hours. The samples were vacuum filtered and approximately 1 gram was spread on filter paper and blotted to remove adsorbed water. Sodium sulfate loses its water of hydration above 33° C. and sodium