

Copper – Antimony – Tin

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Introduction

A summary of experimental studies of phase equilibria is given in Table 1. Early studies [1898Cha, 1913Cam, 1919Tho, 1920Hud] reported the microstructures of as-cast alloys, except that [1913Cam] also presented a tentative phase diagram of the Sn rich corner. The first systematic study of phase equilibria was reported by [1927Bon]. They investigated 28 ternary alloys using metallography and thermal analysis, and determined a partial liquidus surface and an isopleth. [1928Ell] investigated 25 Sn rich alloys using metallography and thermal analysis, and reported liquidus isotherms in the temperature range of 240 to 400°C. [1929Tas] investigated phase equilibria of the entire system, and reported the liquidus surface and twelve isopleths. There is a good agreement between the results of [1928Ell] and [1929Tas], in the composition range investigated. [1938Sla] studied solidification process of a Sn-5.5 mass% Cu-11.5 mass% Sb alloy, and their results are consistent with those reported by [1927Bon].

[1948Har] investigated the phase equilibria of the Sn rich corner, and reported the liquidus and solidus surfaces, thirteen isopleths, and six isothermal sections between 20 and 240°C. They prepared 57 ternary alloys using 99.99% Sn, 99.9% Cu, 99.7% Sn, and determined the phase equilibria using thermal analysis and metallography. [1955Sol] correlated the hardness and electrical resistivity properties with the solid-state phase boundaries in the isopleths of 1 mass% Cu, 2 mass% Sb and 10 mass% Sb reported by [1948Har].

Phase relations in the Cu-Sb-Sn system have been reviewed by [1969Gue, 1973Bla, 1979Cha, 1979Dri].

Binary Systems

The Cu-Sb binary system is accepted from [1994Sub, 2006Rok]. The Cu-Sn binary system is accepted from the assessment of [1990Sau] which was later supplemented by thermodynamic modelling [1996Shi, 2000Moo, 2004Liu] within CALPHAD formalism. In the assessment [1990Sau] and in thermodynamic modeling [1996Shi, 2000Moo] of Cu-Sn system, the β_1/γ_1 phase relation is treated as a first-order phase transformation. In contrast, [2004Liu] reported that a two-stage ordering transition $\beta_1 \rightleftharpoons B2$ (CsCl type) $\rightleftharpoons \gamma_1$ takes place in Cu rich alloys. They used diffusion couples in conjunction with high-temperature electron and X-ray diffraction techniques to study the two-stage ordering transition. However, in the thermodynamic modeling of Cu-Sn system, [2004Liu] did not consider the γ_1 phase. Since the two-stage ordering transition is yet to be verified by others, in this assessment the β_1/γ_1 phase relation is treated as a first-order phase transformation. The η phase of Cu-Sn system is believed to be isotypic with $B8_1$ -NiAs phase [1990Sau]; however, X-ray and electron diffraction studies have shown that both η and η' phases have monoclinic symmetry [1994Lar, 1995Lar]. The Sb-Sn binary system is accepted from [Mas2].

Solid Phases

At high temperature, the γ_1 phase of Cu-Sb and Cu-Sn systems form a continuous solid solution [1929Tas]. The η phase dissolves about 18.5 mass% Sb just below the solidus, but it decreases with decreasing temperature [1929Tas].

[1979Kam] characterized the microstructures of slowly cooled and rapidly solidified Sn-1 mass% Cu-10.4 mass% Sb alloy, and found that the Cu content is negligible in SbSn phase formed under both conditions. [1995Sha] studied the microstructure of rapidly solidified Sn-0.5 mass% Cu-8 mass% Sb alloy using both X-ray and γ -ray diffraction methods, and found both γ_1 and η phases. The presence of former implies a metastable state of rapidly solidified microstructure.

[1929Tas] reported that the ternary phase (τ_1), $\text{Cu}_{12}\text{Sb}_3\text{Sn}_7$, forms by a reaction between the ε phase of Cu-Sb and (Sn), but the crystallographic details are presently unknown.

The crystallographic data of all solid phases is listed in Table 2.

Invariant Equilibria

Figure 1 shows the reaction scheme for the solidification of Cu–Sb–Sn alloys [1929Tas], and puts into evidence seven invariant reactions. The invariant reaction U_6 in the Sn corner was first reported by [1927Bon] and subsequently confirmed by [1929Tas, 1948Har].

The compositions of liquid phase in invariant reactions, taken from [1929Tas], are listed in Table 3.

Liquidus, Solidus and Solvus Surfaces

Figure 2 shows the entire liquidus surface [1929Tas], depicting the melting grooves separating ten different areas of primary crystallization. A few minor adjustments are made in Fig. 2 to comply with the accepted binary phase diagrams. In particular, the participation of phase equilibria involving Sb_2Sn_3 is shown in Fig. 2, as this phase was not known when the liquidus surface was experimentally determined [1929Tas]. Also, approximate isotherms at 100°C interval are superimposed in Fig. 2. [1948Har] made a detailed investigation of the liquidus surface of Sn corner, and their results are shown in Fig. 3. Figure 4 shows the phase relations of Cu–Sb–Sn system just below the solidus surface [1929Tas]. The phase fields involving β and Sb_2Sn_3 are shown dashed as they were not considered by [1929Tas]. In addition, a few minor adjustments are made to comply with the accepted binary phase diagrams.

Isothermal Sections

Figures 5, 6 and 7 show the isothermal section of Sn corner at 239, 230 and 200°C, respectively, based on a very detailed investigation by [1948Har]. According to the reaction scheme presented in Fig. 1, a three-phase field $(Sn)+Sb_2Sn_3+\eta$ is expected below 240°C; however, the stability of Sb_2Sn_3 below 240°C is unknown and it may transform to $SbSn$ via an yet unknown invariant solid-state reaction just below 240°C. Due to this reason, the three-phase field $(Sn)+SbSn+\eta$ is accepted in three isothermal sections as originally reported [1948Har].

Temperature – Composition Sections

[1929Tas] reported isopleths at 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 60, 70 mass% Sb, and [1948Har] reported isopleths at 0.5, 0.75, 1, 1.25, 2, 3 mass% Cu and also at 2, 4, 6, 8, 10, 12, 14 mass% Sb. Figures 8, 9, 10 and 11 show the isopleths at 0.5, 0.75, 1.25 and 3 mass% Cu, respectively. [1948Har] reported a liquidus minima in the isopleth at 1.25 mass% Cu. However, this is judged to be unlikely considering the trend of isopleths as a function of Cu content. Therefore, in Fig. 10, the liquidus in the composition range of less than 10 mass% Sb is shown dashed. Figures 12, 13, 14, 15, 16 and 17 show the isopleths at 2, 4, 6, 8, 10 and 14 mass% Sb, respectively. Once again, a few minor adjustments are made in the isopleths to comply with the accepted binary phase diagrams.

Thermodynamics

[1956Kle] measured the heat of mixing of three Sn rich liquid alloys at 450°C by mixing appropriate amounts of Cu–Sb and Cu–Sn liquids. Later, [1993Lee] measured the heat of mixing of liquid alloys over a wide composition range at 727°C using a high-temperature calorimeter. Figure 18 shows the isoenthalpy (mixing) contours in ternary alloys [1993Lee]. The experimental data show that the enthalpy of mixing of ternary alloys is positive in the Sb corner, and it gradually becomes negative in the Sn corner with increasing Cu concentration. A negative heat of mixing may imply the tendency for chemical short range ordering in the liquid phase. [1993Lee] calculated the composition dependence of heat of mixing, using an associate solution model [1982Som1, 1982Som2], with the assumption of the presence of five binary associates, Cu_3Sb_1 , Cu_1Sb_1 , Cu_3Sn_1 , Sb_1Sn_2 and Sb_1Sn_1 in liquid alloy. They obtained a good agreement between calculated and experimental results.

[1973Gri] measured the activity of Sn at 389°C in liquid alloys containing less than 12 at.% solute ($x_{Cu}+x_{Sb}$). Their data indicate a slight positive deviation from Raoult's law in the ternary alloys. [1984Sze] determined the activity of Sb in liquid alloys in the temperature range of 788 to 1163°C. [1989Cho]

determined the activity of Sb at 1150°C in liquid alloys in the entire composition range by isothermal-isopiestic method. Both measurements show a negative deviation from Rault's law; however, the data of [1984Sze] exhibit a large scatter.

Notes on Materials Properties and Applications

Hardness of ternary alloys have been measured by several authors [1919Tho, 1928Ell, 1929Tas, 1995Sha, 2003Elb]. In addition, [1928Ell] measured hardness at high temperature (up to 125°C), and yield strength and ultimate strength under compression. They also established several cross-property relationships in Sn rich alloys, such as hardness vs yield strength, hardness vs ultimate strength and hardness vs Young's modulus. [1929Tas] also reported mechanical properties of Sn rich alloys under compression. [1955Sol] determined the hardness and electrical resistivity of alloys along the isopleths of 1 mass% Cu, 2 mass% Sb and 10 mass% Sb. [2003Elb] measured the isotropic elastic moduli (bulk, shear and Young's), internal friction and electrical resistivity of rapidly solidified Sn-1Cu-10Sb (mass%) alloy. The addition of 1 mass% Cu in Sn-10 mass% Sb alloy increases the elastic moduli, but decreases hardness and internal friction while the resistivity remains unchanged.

The η phase alloyed with Sb is age-hardenable, because the solubility of Sb varies with temperature [1929Tas].

Miscellaneous

[1995Sha] demonstrated the advantage of using γ -ray diffraction technique over X-ray diffraction for structural studies of Cu-Sb-Sn alloys.

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Table 1: Investigations of the Cu-Sb-Sn Phase Relations, Structures and Thermodynamics

Reference	Method/Experimental Technique	Temperature/Composition/Phase Range Studied
[1898Cha]	Metallography	
[1913Cam]	Metallography, thermal analysis	$0.0 \leq w_{\text{Cu}} \leq 0.3$, $0.0 \leq w_{\text{Sb}} \leq 0.3$, $w_{\text{Sn}} = \text{balance}$
[1919Tho]	Metallography, thermal analysis, hardness	$w_{\text{Cu}} = 0.0451$, $x_{\text{Sb}} = 0.0876$, $w_{\text{Sn}} = \text{balance}$
[1920Hud]	Metallography, thermal analysis	$0.023 \leq w_{\text{Cu}} \leq 0.034$, $0.084 \leq w_{\text{Sb}} \leq 0.087$, $w_{\text{Sn}} = \text{balance}$
[1927Bon]	Metallography, thermal analysis	$0.0 \leq w_{\text{Cu}} \leq 0.3$, $0.0 \leq w_{\text{Sb}} \leq 0.58$, $w_{\text{Sn}} = \text{balance}$
[1928Eil]	Metallography, thermal analysis, hardness	$0.005 \leq w_{\text{Cu}} \leq 0.0842$, $0.021 \leq w_{\text{Sb}} \leq 0.11$, $w_{\text{Sn}} = \text{balance}$
[1929Tas]	Metallography, thermal analysis, hardness	$0.025 \leq w_{\text{Cu}} \leq 0.95$, $0.05 \leq w_{\text{Sb}} \leq 0.79$, $w_{\text{Sn}} = \text{balance}$
[1938Sla]	Infiltration, XRD	$w_{\text{Cu}} = 0.055$, $w_{\text{Sb}} = 0.115$, $w_{\text{Sn}} = \text{balance}$
[1948Har]	Metallography, thermal analysis	$0.0025 \leq w_{\text{Cu}} \leq 0.03$, $0.01 \leq w_{\text{Sb}} \leq 0.14$, $w_{\text{Sn}} = \text{balance}$
[1955Sol]	Hardness, electrical resistivity	$0.0025 \leq w_{\text{Cu}} \leq 0.03$, $0.01 \leq w_{\text{Sb}} \leq 0.14$, $w_{\text{Sn}} = \text{balance}$
[1956Kle]	Calorimetry	450°C ; $0.1012 \leq x_{\text{Cu}} \leq 0.1073$ and $0.0588 \leq x_{\text{Sb}} \leq 0.0688$; liquid
[1973Gri]	EMF	$389 \pm 2^\circ\text{C}$; $0.01 \leq x_{\text{Cu}} + x_{\text{Sb}} \leq 0.125$; liquid
[1979Kam]	Rapid solidification, SEM, TEM	$w_{\text{Cu}} = 0.01$, $0.118 \leq w_{\text{Sb}} \leq 0.124$, $w_{\text{Sn}} = \text{balance}$
[1981Ros]	XRD	$\text{Cu}_{10}\text{Sb}_1\text{Sn}_9$
[1984Sze]	Secondary ion mass spectroscopy	$788 - 1163^\circ\text{C}$; $0.193 \leq x_{\text{Cu}} \leq 0.762$ and $0.048 \leq x_{\text{Sb}} \leq 0.4$
[1989Cho]	Isothermal-isopiestic	1150°C ; $x_{\text{Cu}} < 0.9$ and $x_{\text{Sb}} > 0.1$
[1993Lee]	High-temperature calorimetry	723°C ; $0.05 \leq x_{\text{Cu}} \leq 0.62$ and $0.05 \leq x_{\text{Sb}} \leq 0.665$; liquid
[1995Sha]	Rapid solidification, Dynamic resonance, XRD, γ -ray diffraction	25°C ; $w_{\text{Cu}} = 0.005$, $w_{\text{Sb}} = 0.08$, $w_{\text{Sn}} = \text{balance}$
[2003Elb]	Dynamic resonance, resistivity, hardness, XRD	25°C ; $w_{\text{Cu}} = 0.01$, $w_{\text{Sb}} = 0.10$, $w_{\text{Sn}} = \text{balance}$

Table 2: Crystallographic Data of Solid Phases

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
γ , (Cu) ≤ 1084.62	<i>cF4</i> <i>Fm$\bar{3}m$</i> Cu	$a = 361.46$	pure Cu at 25°C [V-C2]
(Sb) < 630.755	<i>hR6</i> <i>R$\bar{3}m$</i> α As	$a = 450.67$ $\alpha = 57.11^\circ$	pure Sb at 25°C [V-C2]
(β Sn) 231.5 - 13	<i>tI4</i> <i>I4$_1$/amd</i> Sn	$a = 583.18$ $c = 318.18$	pure Sn at 25°C [V-C2]
(α Sn) < 13	<i>cF8</i> <i>Fd$\bar{3}m$</i> C (diamond)	$a = 648.92$	pure Sn [V-C2]
β_1 , Cu ₁₇ Sn ₃ 586 - 798	<i>cI2</i> <i>Im$\bar{3}m$</i> W	$a = 297.81$ to 298.71	$0.131 \leq x_{\text{Sn}} \leq 0.158$ [V-C2]
γ , Cu ₁₁ Sb ₂ 488 - 400	<i>hP2</i> <i>P6$_3$/mcm</i> Mg	$a = 270$ to 271.7 $c = 435$ to 437.4	$0.155 \leq x_{\text{Sb}} \leq 0.16$ [Mas2] [1994Sub]
γ_1 , Cu ₃ Sb 683 - 440	<i>cF16</i> <i>Fm$\bar{3}m$</i> BiF ₃	$a = 592.6$ to 601	$0.194 \leq x_{\text{Sb}} \leq 0.308$ [Mas2] [1994Sub]
Cu ₃ Sn(h) 755 - 520		$a = 606.05$ to 611.76	$0.165 \leq x_{\text{Sn}} \leq 0.279$ [V-C2]
δ , Cu ₉ Sb ₂ < 462	<i>hP2</i> <i>P6$_3$/mcm</i> Mg	$a = 272.1$ to 272.9 $c = 431.9$ to 433.1	$0.185 \leq x_{\text{Sb}} \leq 0.20$ [Mas2] [1994Sub]
ϵ , Cu ₃ Sb 445 - 360	<i>oP8</i> <i>Pmmn</i> β Cu ₃ Ti	$a = 548.6$ to 557.6 $b = 476.8$ to 480.5 $c = 435.2$ to 439.8	$0.245 \leq x_{\text{Sb}} \leq 0.259$ [Mas2] [1994Sub]
ζ , Cu ₁₀ Sb ₃ 390 - 260	<i>hP26</i> <i>P$\bar{3}$</i> Cu ₁₀ Sb ₃	$a = 992.0$ $c = 432.0$	$0.213 \leq x_{\text{Sb}} \leq 0.216$ [V-C2], [1994Sub]
ρ , Cu ₂ Sb < 526	<i>tP6</i> <i>P4/nmn</i> Cu ₂ Sb	$a = 398.8$ to 403.7 $c = 608.7$ to 615.2	$0.32 \leq x_{\text{Sb}} \leq 0.33$ [V-C2], [1994Sub]
ϵ_1 , Cu ₃ Sn(r) < 676	<i>oC80</i> <i>Cmcm</i> Cu ₃ Sn	$a = 552.9$ $b = 4775.0$ $c = 432.3$	$0.245 \leq x_{\text{Sn}} \leq 0.259$ [V-C2] at 25 at.% Sn [V-C2]
δ , Cu ₄₁ Sn ₁₁ 590 - 350	<i>cF416</i> <i>F$\bar{4}3m$</i> Cu ₄₁ Sn ₁₁	$a = 1798$ $a = 1801$	$0.203 \leq x_{\text{Sn}} \leq 0.208$ [V-C2], at 20.5 at.% Sn

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
ζ_1 , Cu ₁₀ Sn ₃ 640 - 582	<i>hP</i> 26 <i>P</i> 6 ₃	$a = 733.0$ $c = 784.0$	$0.209 \leq x_{\text{Sn}} \leq 0.225$ [V-C2], at 23.1 at.% Sn [V-C2]
η , Cu ₆ Sn ₅ (h) 415 - 186	<i>mP</i> 36 <i>P</i> 2 ₁ / <i>c</i> Cu ₅ Sn ₄	$a = 983.0$ $b = 727.0$ $c = 983.0$ $\beta = 62.5^\circ$	$0.435 \leq x_{\text{Sn}} \leq 0.445$ [Mas2], superstructure of <i>B</i> 8 ₁ -NiAs type, at 44.44 at.% Sn [1995Lar]
	<i>mC</i> 54 <i>C</i> 2 Cu ₅ Sn ₄	$a = 1260.0$ $b = 727.0$ $c = 1020.0$ $\beta = 90^\circ$	superstructure of <i>B</i> 8 ₁ -NiAs type, at 44.44 at.% Sn [1995Lar]
η' , Cu ₆ Sn ₅ (r) < 186	<i>mP</i> 44 <i>C</i> 2/ <i>c</i> Cu ₆ Sn ₅	$a = 1103.6$ $b = 728.8$ $c = 984.0$ $\beta = 98.81^\circ$	$0.435 \leq x_{\text{Sn}} \leq 0.445$ [Mas2], superstructure of <i>B</i> 8 ₁ -NiAs type, at 44.44 at.% Sn [1994Lar]
SbSn < 425	<i>cF</i> 8 <i>Fm</i> $\bar{3}$ <i>m</i> NaCl	$a = 588.0$	$0.348 \leq x_{\text{Sn}} \leq 0.57$ [V-C2], at 50 at.% Sn [V-C2]
	<i>hR</i> 8	$a = 864.8$ $c = 1067.5$	mineral at 56.2 at.% Sn [1981Ros]
Sb ₂ Sn ₃ 324 - 242	-	-	[V-C2]
τ , Cu ₁₂ Sb ₃ Sn ₇	-	-	[1929Tas]

Table 3: Invariant Equilibria

Reaction	T [°C]	Type	Phase	Composition (at.%)		
				Cu	Sb	Sn
$L \rightleftharpoons (\text{Cu}) + \beta + \gamma_1$	643	E ₁	L	79.5	18.0	2.5
$L + \gamma_1 \rightleftharpoons \rho + \varepsilon$	470	U ₁	L	34.8	29.7	35.5
$L + \varepsilon \rightleftharpoons \rho + \eta$	405	U ₂	L	18.9	30.5	50.6
$L + (\text{Sb}) \rightleftharpoons \text{SbSn} + \rho$	386	U ₃	L	14.1	42.4	43.5
$L + \rho \rightleftharpoons \text{SbSn} + \eta$	372	U ₄	L	10.8	37.4	51.8
$L + \text{SbSn} \rightleftharpoons \text{Sb}_2\text{Sn}_3 + \eta$	319	U ₅	L	3.7	20.2	76.1
$L + \text{Sb}_2\text{Sn}_3 \rightleftharpoons (\text{Sn}) + \eta$	319	U ₆	L	1.8	7.4	90.8

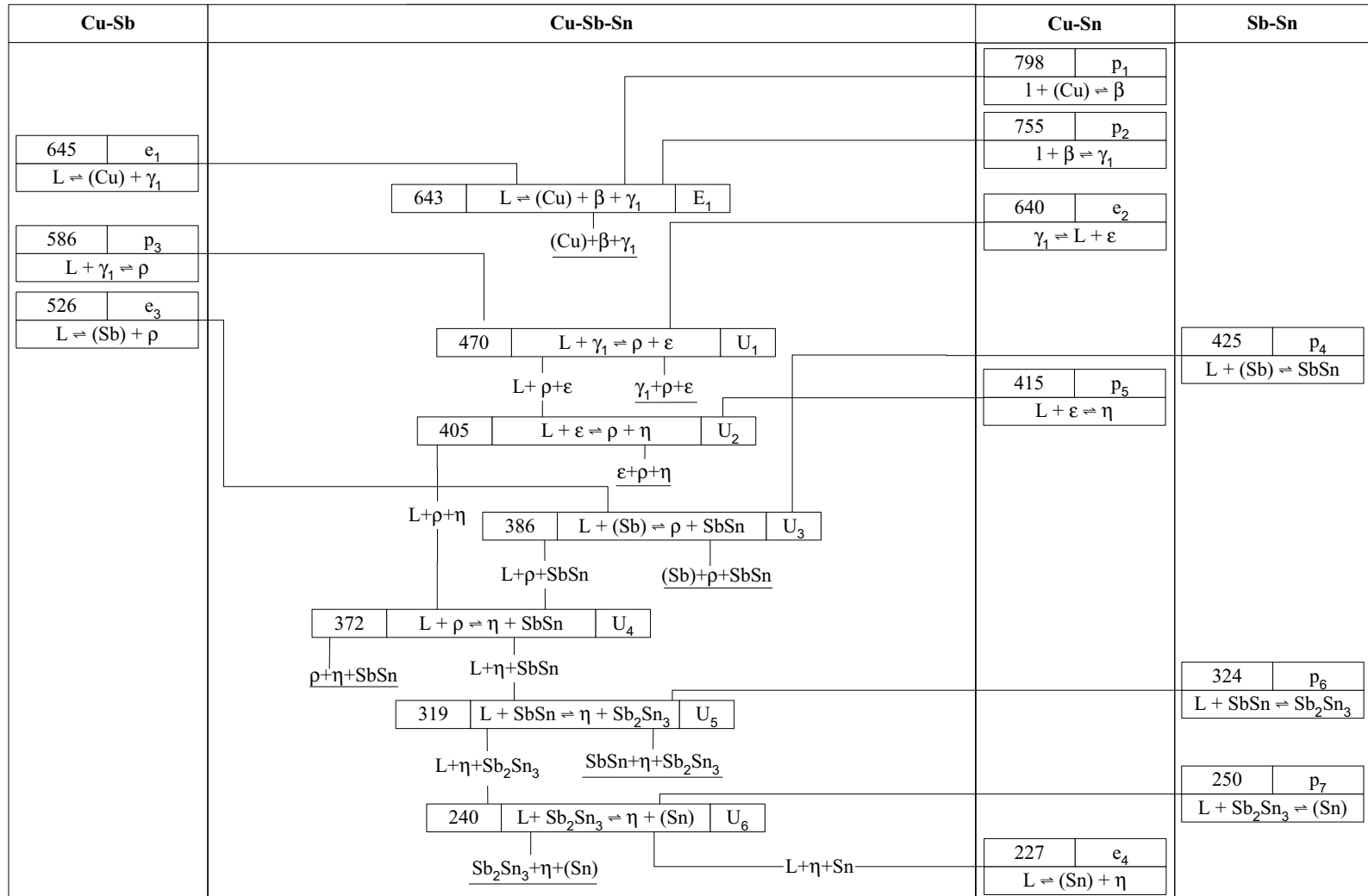


Fig. 1: Cu-Sb-Sn. Reaction scheme for the solidification of Cu-Sb-Sn alloys

Fig. 2: Cu-Sb-Sn.
Liquidus surface

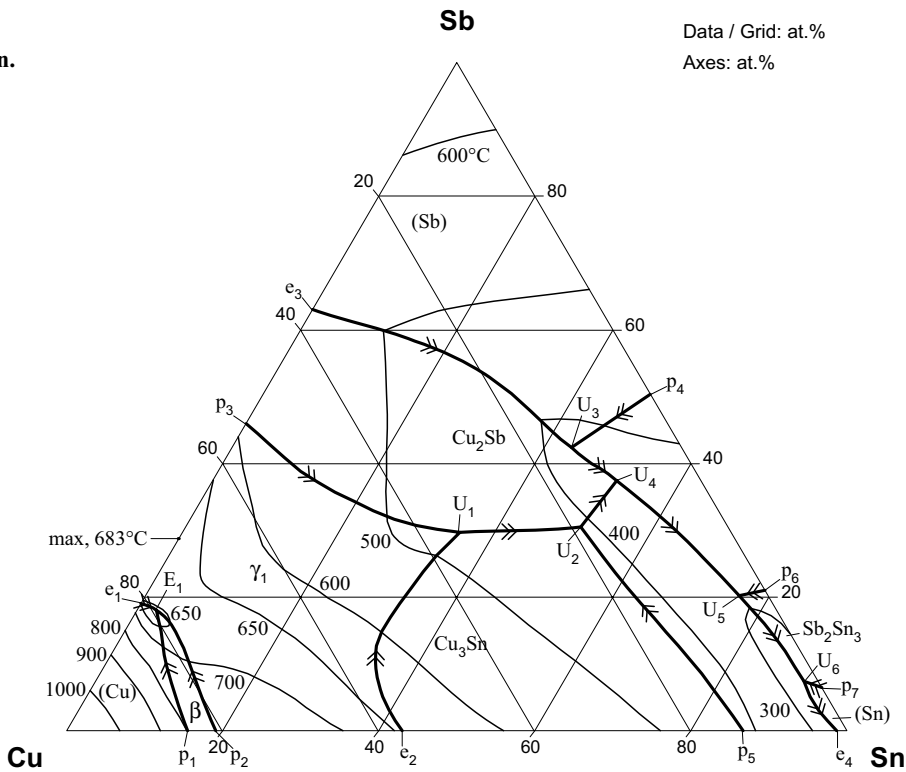


Fig. 3: Cu-Sb-Sn.
Liquidus surface of
Sn corner

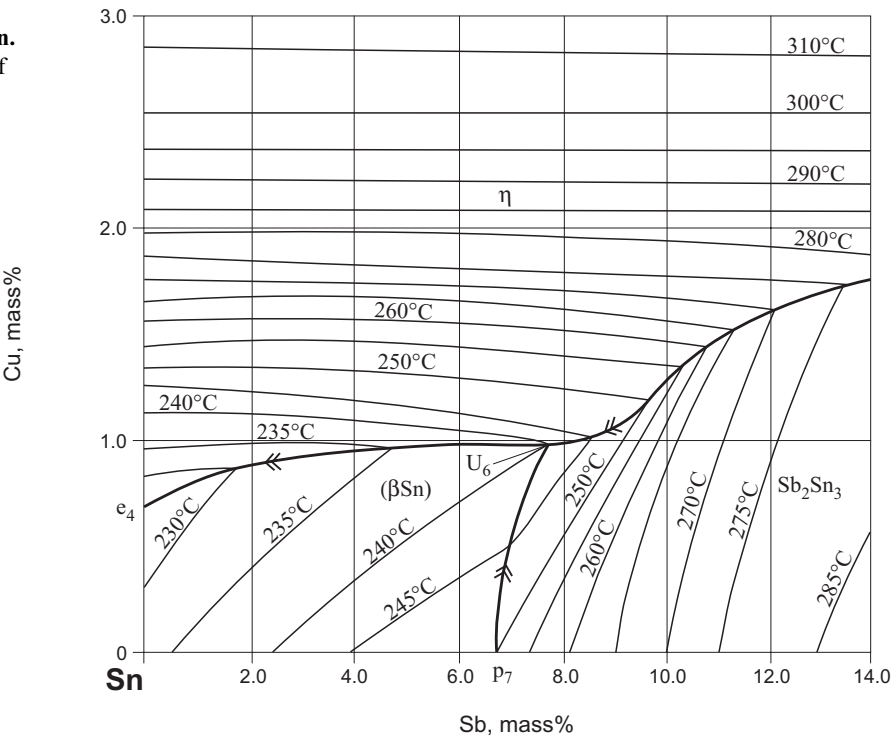


Fig. 4: Cu-Sb-Sn.
Phase relations just below the solidus

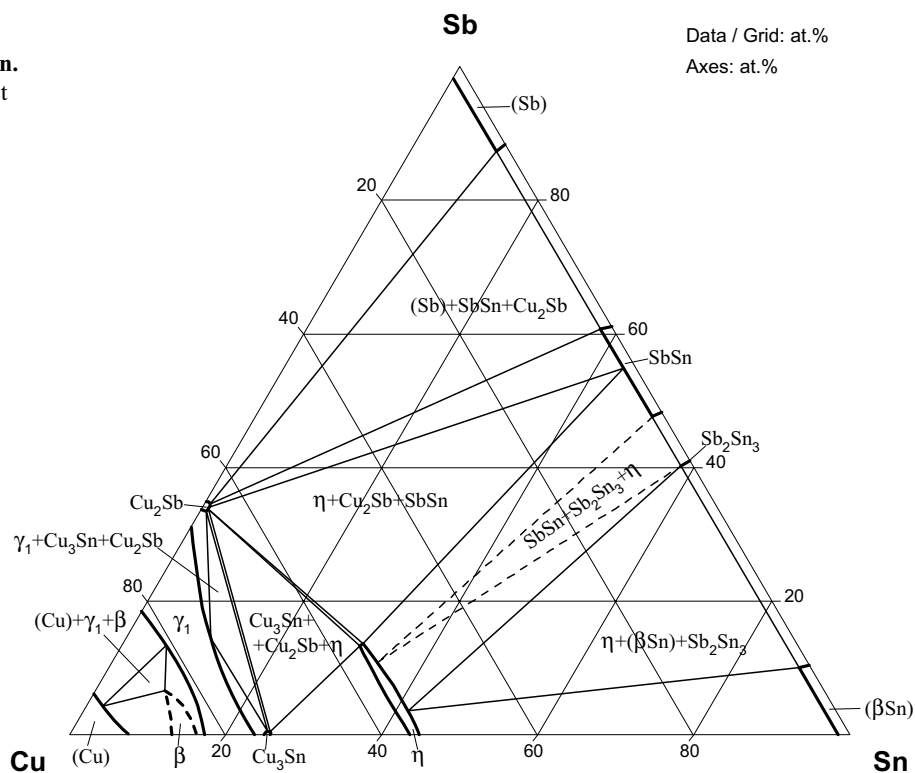


Fig. 5: Cu-Sb-Sn.
Isothermal section of Sn corner at 239°C

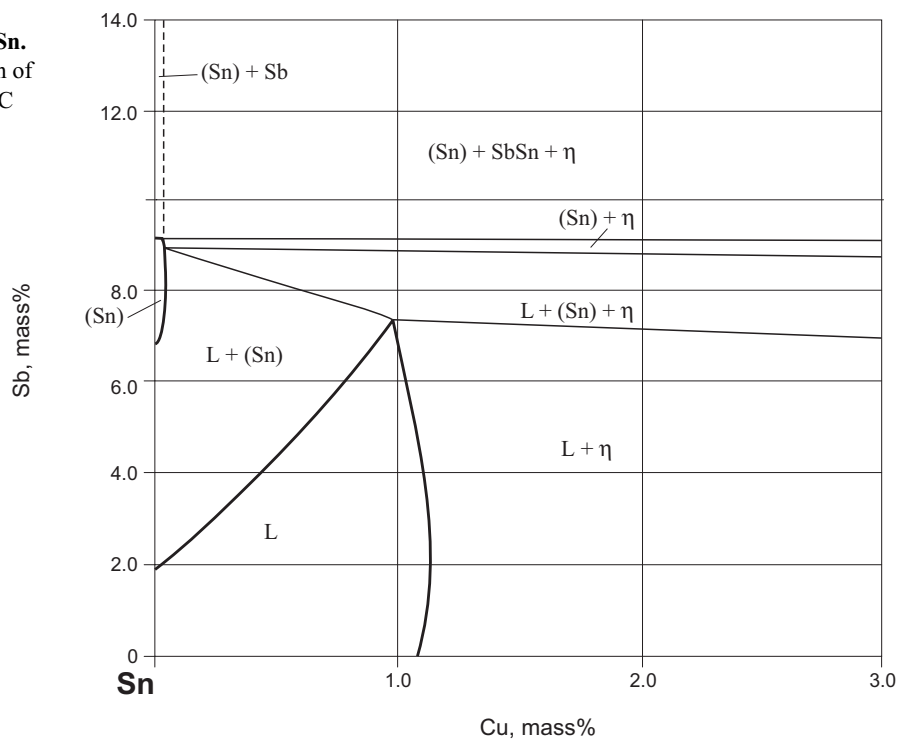


Fig. 6: Cu-Sb-Sn.
Isothermal section of
Sn corner at 230°C

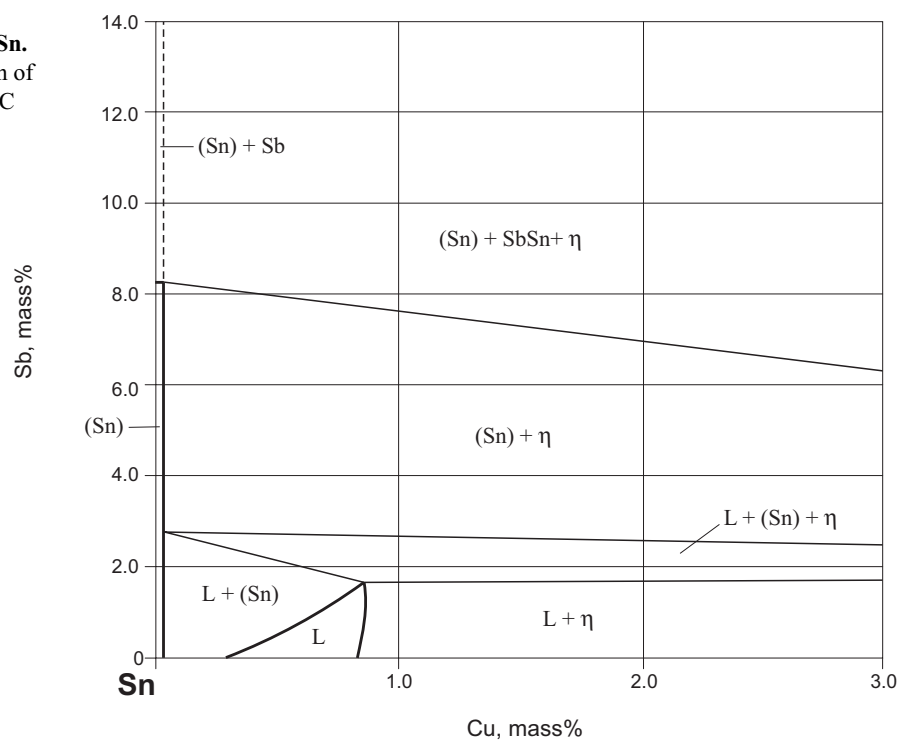


Fig. 7: Cu-Sb-Sn.
Isothermal section of
Sn corner at 200°C

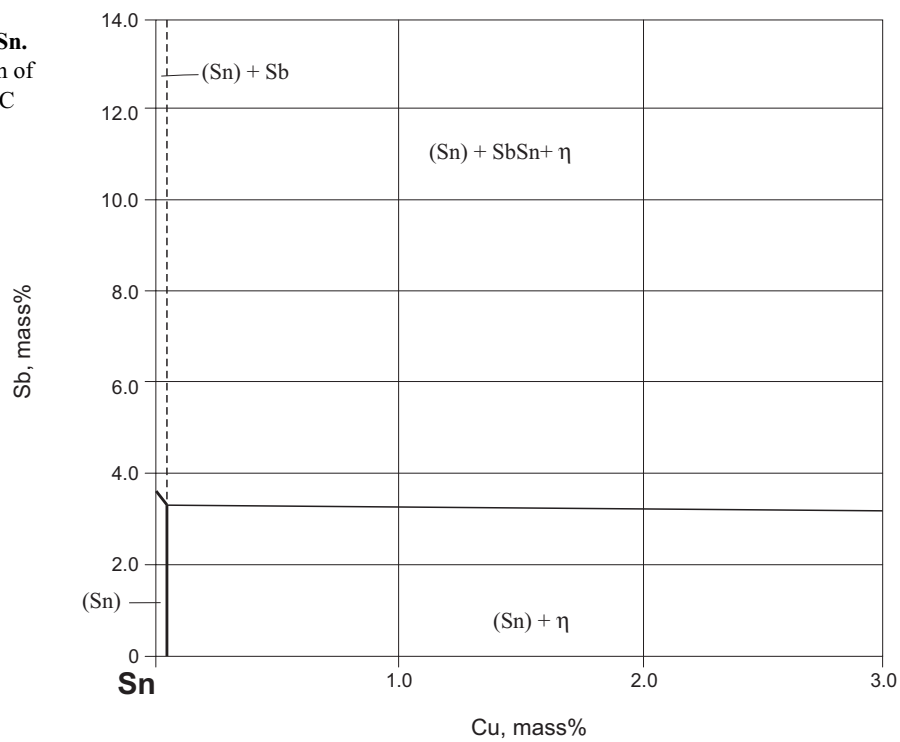
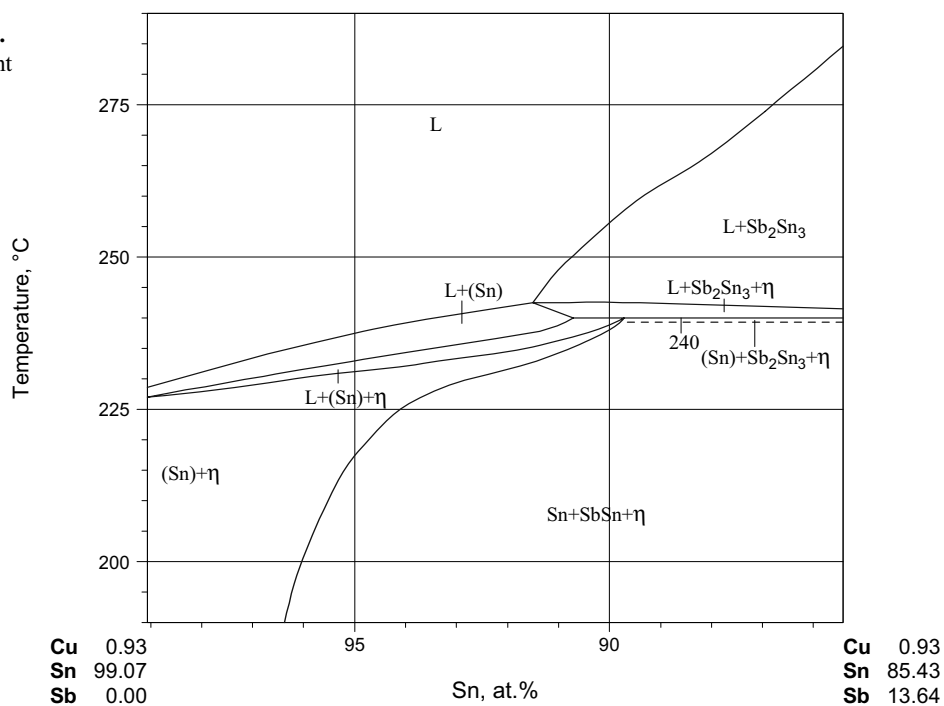


Fig. 8: Cu-Sb-Sn.

Isopleth at a constant
Cu content of 0.5
mass%, plotted in
at.%

**Fig. 9: Cu-Sb-Sn.**

Isopleth at a constant
Cu content of 0.75
mass%, plotted in
at.%

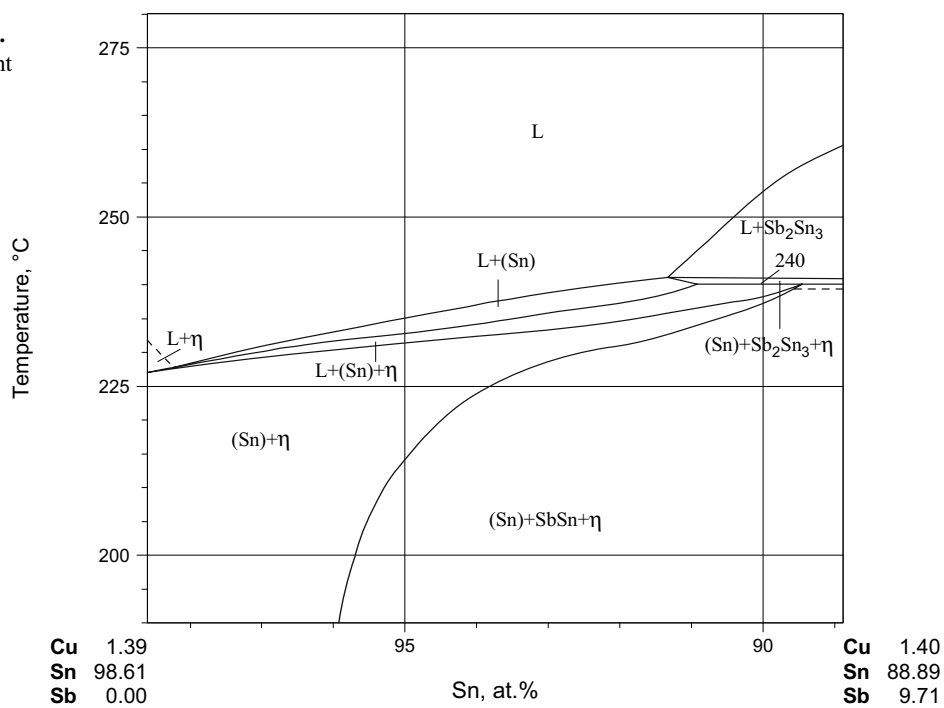
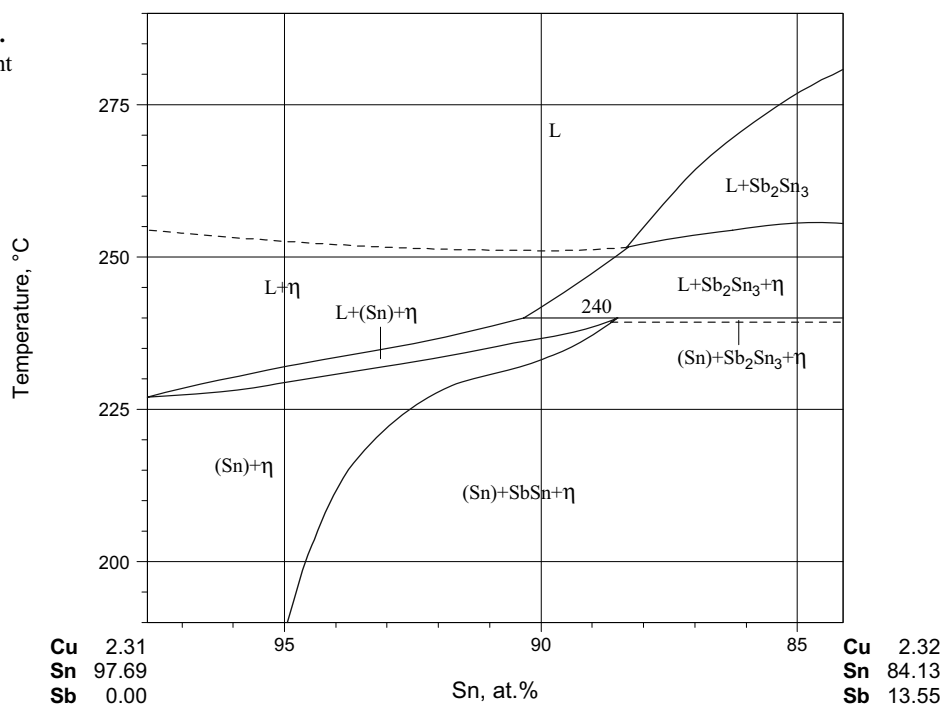


Fig. 10: Cu-Sb-Sn.

Isopleth at a constant
Cu content of 1.25
mass%, plotted in
at.%

**Fig. 11: Cu-Sb-Sn.**

Isopleth at a constant
Cu content of 3
mass%, plotted in
at.%

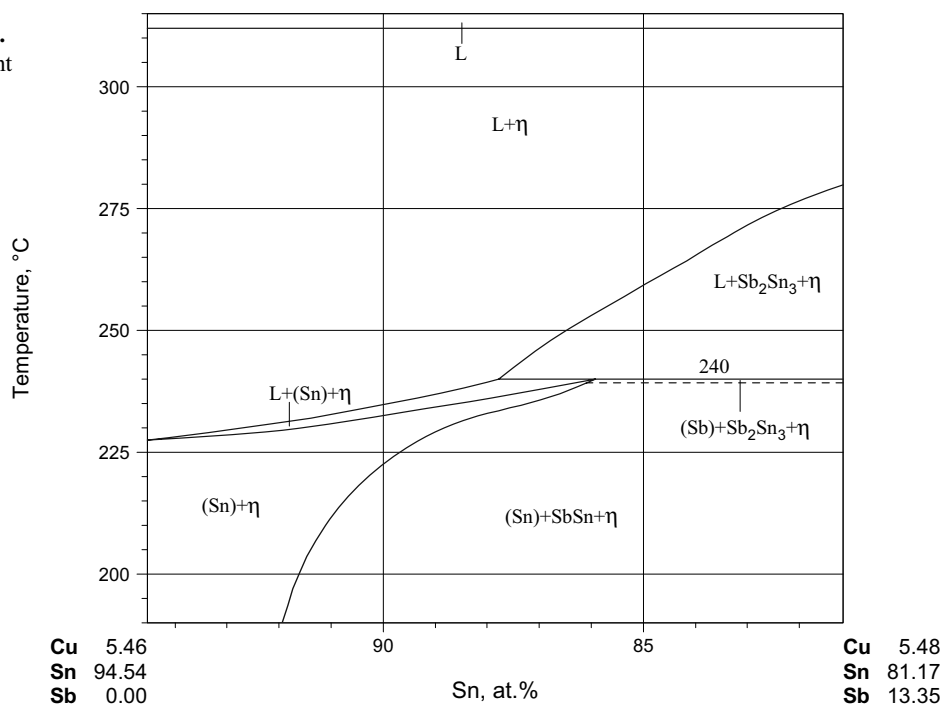
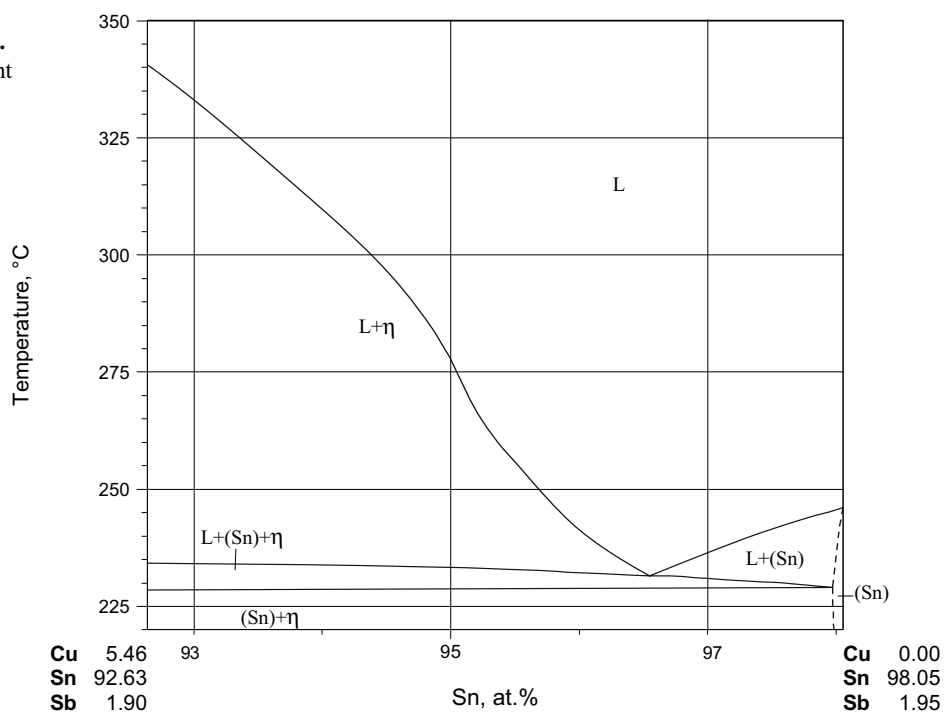


Fig. 12: Cu-Sb-Sn.

Isopleth at a constant
Sb content of 2
mass%, plotted in
at.%

**Fig. 13: Cu-Sb-Sn.**

Isopleth at a constant
Sb content of 4
mass%, plotted in
at.%

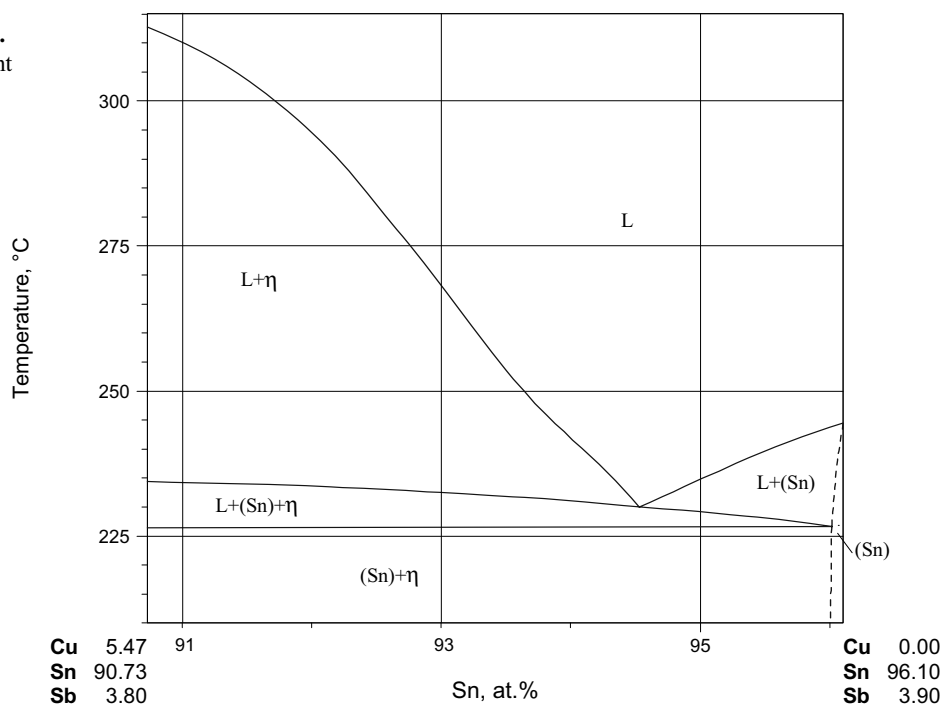
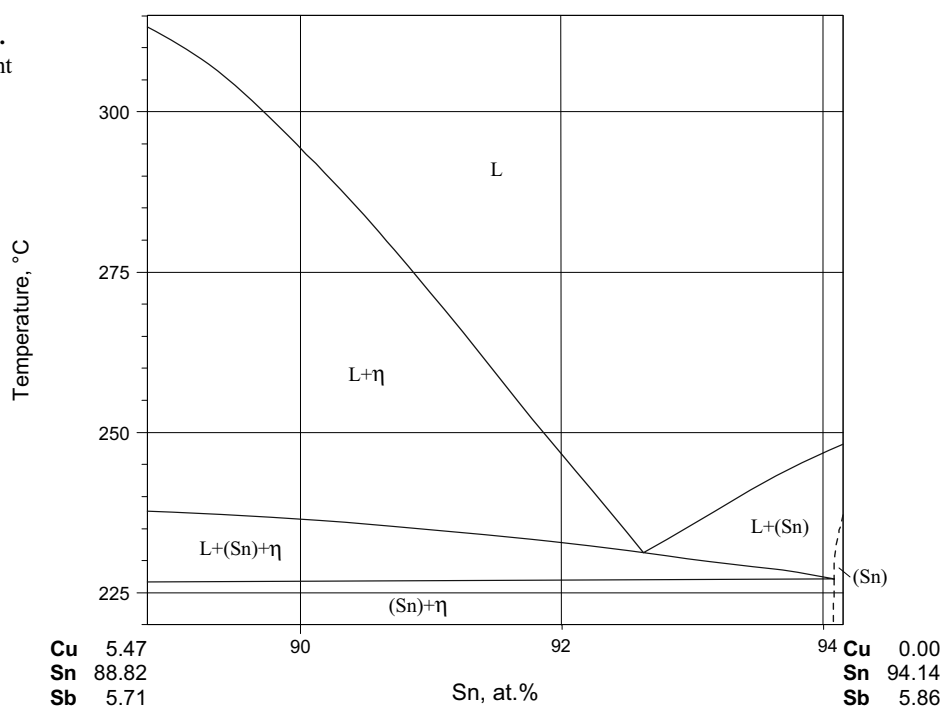
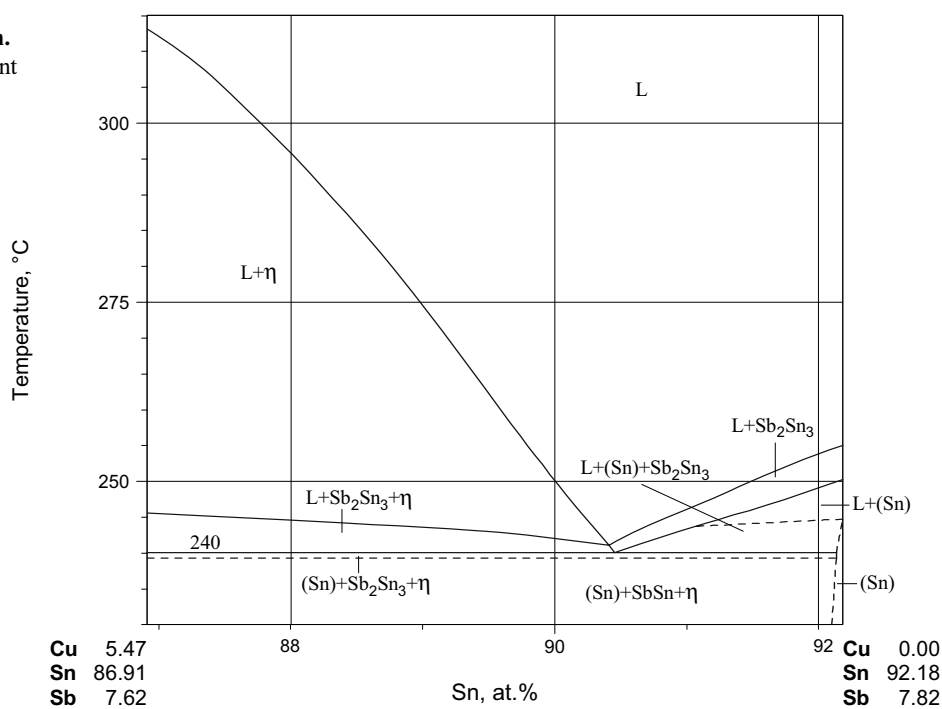


Fig. 14: Cu-Sb-Sn.

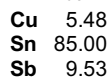
Isopleth at a constant
Sb content of 6
mass%, plotted in
at.%

**Fig. 15: Cu-Sb-Sn.**

Isopleth at a constant
Sb content of 8
mass%, plotted in
at.%



Isopleth at a constant
Sb content of 10
mass%, plotted in
at. %



Isopleth at a constant
Sb content of 14
mass%, plotted in
at. %

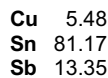


Fig. 18: Cu-Sb-Sn.
Integral enthalpy of
mixing of liquid
alloys at 727°C, in
 $\text{kJ}\cdot\text{mol}^{-1}$

