Silver – Copper – Tin

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Introduction

The phase diagram of the Ag-Cu-Sn system is fundamental to the development of technology of lead-free solder alloys. Recent legislations in many countries to eliminate the use of lead containing solder alloys have created renewed interest in this system, especially in the Sn rich region. Apart from this, Ag-Cu-Sn also forms the basis of several Cu rich dental amalgams.

Constitutional data of the Ag-Cu-Sn system were reviewed by several authors [1967Mcd, 1977Cha, 1977Dar, 1979Dri, 1982Fed, 1988Kub, 2002Lew]. Present report updates an earlier evaluation by [1988Kub] made within the MSIT Evaluation Program. First comprehensive experimental work on the phase equilibria was reported by [1959Geb], employing thermal, metallographic and X-ray analyses. That investigation mainly concentrated in the Ag-Cu region and reported a liquidus projection, isothermal sections at 500 and 600°C, and vertical sections at 5, 10, 15, 20 and 25 mass% Sn. The Sn rich region was investigated by [1959Wag] (thermal analysis) and by [1954Cro] (metallography), results of these studies were incorporated in [1959Geb]. Using X-ray and metallographic techniques [1956Gla] claimed that the intermetallic systems $Ag_3Sn(\theta)$-$Cu_3Sn(\varepsilon_1)$ and $Ag_5Sn(\varepsilon_2)$-$Cu_3Sn(\varepsilon_1)$ show only limited solid solubility. Phase relation in the system at 37°C was reported by [1972Jen, 1973Wir, 1980Kra] independently, that was used to explain characteristics of certain Cu rich dental amalgams. [1994Mil] was the first to report that Sn rich invariant reaction, observed in alloys close to typical solder compositions currently in use, is of eutectic type instead of transition type as stated in the work of [1959Geb]. This was later confirmed by several investigators: [1997And], [2000Loo] (DSC), [2000Moo, 2004Moo] (DTA), and [2002Lew] (SEM, EPMA, LOM). Using DTA [1997He] determined liquidus and solidus of seven Cu rich alloys, typically used as brazing filler. [1997He] also reported that all alloys in the composition range studied (15-25 mass% Ag, 15-24 mass% Sn) contained the δ phase ($Cu_{41}Sn_{11}$), that decreased ductility. Using SEM-EDS and optical microscopy [2000Ohn] established phase relation in nine samples in the temperature range 300-500°C having compositions 20-48 mass% Ag and 15-60 mass% Cu. Isopleths of the ternary Ag-Cu-Sn system were published by two teams, [1959Geb] and [1982Fed]. The vertical sections at constant Sn content, namely 5, 10, 15, 20 and 25 mass% are based on thermal, microscopic and X-ray studies by [1959Geb]. Three isopleths were constructed by [1982Fed]: one parallel to the Ag-Sn system containing 10 mass% Cu, one cross section from Ag to 80Sn-17.8Cu and one from Cu to 50Ag-50Sn (mass%).

Apart from experimental investigations, several authors have reported calculated phase equilibria in the system [1997Lee, 2000Moo, 2000Ohn, 2003Kim, 2003Ohn], employing the CALPHAD method, however, only [2000Moo] reported ternary parameters used for the calculation. The phase diagrams presented in the present evaluation are calculated using the Calphad approach (see section “Thermodynamics”).

[2004Lue] determined isoenthalpy curves for liquid alloys at 500, 700, 900°C using a Calvet type calorimeter. Physical properties of typical solder compositions were also studied by several authors: notably density [2004Gas], viscosity [2001Lee, 2002Wan, 2003Ohn], surface tension [2001Lee, 2002Mos, 2003Ohn, 2004Gas], and wetting properties [2004Gas]. Electrical properties were studied by [2003Coo, 2004Kis]. Coarsening kinetics of intermetallics of the solder alloys were determined by [2004All].

Binary Systems

The binary system Ag-Cu [2002Rom] is from the MSIT Binary Evaluation Program. Ag-Sn is from [1999Oht] and Cu-Sn is from [2000Ohn]. The version of the Cu-Sn phase diagram used here differs from that of [Mas2] with respect to the γ phase. It has been confirmed by the careful investigations by [2000Ohn, 2004Liu] that there is no eutectoid reaction $\beta \rightleftharpoons (Cu) + \gamma$ in the Cu-Sn system, instead the phase boundary between γ and β is of higher-order type involving a two-stage chemical ordering reaction β (*A*2) ⇌ β’ (*B*2) ⇌ γ ($D0_3$). In the evaluation of the ternary system, the order-disorder equilibria are neglected since the ternary extensions of them are undetermined.

Solid Phases

No ternary compounds have been reported. The solid phases of the Ag-Cu-Sn ternary systems are listed in Table 1.

Invariant Equilibria

The invariant equilibria associated with the liquid phase are given in Table 2. All invariant reaction temperatures and compositions are calculated using the Calphad approach. They are in reasonable agreement with experimental data. Figure 1 depicts the reaction scheme for the system relevant to reactions involving the liquid phase.

Liquidus Surface

The calculated liquidus surface is shown in Fig. 2. It should be noted that the calculated phase boundaries along the Ag-Cu side deviate slightly from the accepted version of the Ag-Cu binary system [2002Rom]. Wherever necessary the phase boundaries are adjusted such that it matches with those given in [2002Rom]. The invariant reaction close to the Sn corner occurring at 217.7°C is of eutectic type (E_1). The corresponding eutectic composition is 3.24Ag-0.57Cu-Sn (mass%). These are very close to the very precisely experimentally determined eutectic temperature (217.2°C) and composition (3.58Ag-0.96Cu-Sn) [2004Moo]. Figure 3 is the enlarged Sn rich corner of the liquidus projection in detail.

Isothermal Sections

Figures 4 to 7 show isothermal sections at 600, 500, 400, and 37°C, respectively, calculated using the Calphad method. Here also the phase boundaries along the Ag-Cu side have been adjusted to match with the accepted binary system. It is assumed that except β and ε_2, none of the intermediate phases extends into the ternary region. The calculated diagrams compare well with available experimental data [1959Geb, 1980Kra, 2000Ohn].

Temperature – Composition Sections

Isopleths at 3.66 mass% Ag, 10 mass% Sn, and 25 mass% Sn are shown in Figs. 8, 9 and 10, respectively. These diagrams reproduce fairly well the experimental findings of [1959Geb] and [1982Fed].

Thermodynamics

The phase diagrams presented in the present evaluation are calculated using the Calphad approach. The binary Gibbs energy model parameters are from [1986Hay] (Ag-Cu), [1999Oht] (Ag-Sn), and [2001Liu] (Cu-Sn). The ternary parameters are obtained in the present work and they are listed in Table 3.
Calorimetrically measured [2004Lue] isoenthalpy curves for liquid alloys at 500 and 700°C are shown in Figs. 11 and 12, respectively. The data indicate temperature dependence for the enthalpy of mixing.

Notes on Materials Properties and Applications

Ag-Cu-Sn alloys form the basis of lead-free solders and dental amalgams. Knowledge of the phase relationships in the ternary and quaternary (Ag-Cu-Sn-Hg) systems is essential for the understanding and development of new dental amalgams. $Ag_3Sn(\theta)$ is known as “dental alloy”. If mixed with an equal amount of Hg, the undesirable phase $Sn_{7.8}Hg$ is formed during the hardening process. The addition of Cu to the basic Ag-Sn alloys suppresses the formation of this phase [1980Kra].
Renewed interest in Ag-Cu-Sn system is particularly due to several Sn rich lead-free solder compositions close to the eutectic E_1. Physical properties such as surface tension and viscosity of the liquid phase are important quantities for quantifying wetting properties of solders. Several such studies are mentioned in the section “Introduction”. In Figs. 13 and 14, calculated surface tension contours for the liquid are shown at

800 and 1000°C, respectively [2000Ohn, 2003Ohn]. Figures 15 and 16 show isoviscosity contours for the liquid at 800 and 1000°C [2000Ohn, 2003Ohn], respectively.

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Table 1: Crystallographic Data of Solid Phases

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
(Ag) < 961.93	*cF*4 *Fm*$\bar{3}$*m* Cu	*a* = 408.57 *a* = 414.7	pure, at 25°C [Mas2] 14 at.% Sn [V-C]
(Cu) < 1084.62	*cF*4 *Fm*$\bar{3}$*m* Cu	*a* = 361.46 *a* = 370.46	pure, at 25°C [Mas2] 9.1 at.% Sn, [Mas2]
(βSn) 231.9681 - 13	*tI*4 $I4_1/amd$ Sn	*a* = 583.18 *c* = 318.18	pure, at 25°C [Mas2]
(αSn) < 13	*cF*8 *Fd*$\bar{3}$*m* C (diamond)	*a* = 648.92	pure, [Mas2]
β, $Cu_{13}Sn_3$ 796 - 586	*cI*2 *Im*$\bar{3}$*m* W	*a* = 297.81	13.4 at.% Sn [Mas2] 12.6 - 17 at.% Sn [2004Liu]
β´ 775 - 574	*cP*2 *Pm*$\bar{3}$*m* CsCl		13.7 - 22.8 at.% Sn [2004Liu]
γ, Cu_4Sn 722 - 515	*cF*16 *Fm*$\bar{3}$*m* BiF_3	*a* = 606.05	710°C, 16.6 at.% Sn [Mas2] 13.7 - 27.5 at.% Sn [2004Liu]
δ, $Cu_{41}Sn_{11}$ 569 - 350	*cF*416 *F*$\bar{4}$3*m* $Cu_{41}Sn_{11}$	*a* = 1798	20.5 at.% Sn [Mas2]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
ζ, $Cu_{10}Sn_3$ 635 - 540	*hP*26 $P6_3$ $Cu_{10}Sn_3$	*a* = 733.0 *c* = 786.4	23.1 at.% Sn [Mas2]
η, Cu_6Sn_5 (HT) 415 - 187	*hP*4 $P6_3/mmc$ NiAs	*a* = 419.0 *c* = 508.6	45.45 at.% Sn [Mas2]
η´, Cu_6Sn_5 (LT) < 187	*hR*22	*a* = 2087.0 *c* = 2508.1	45.45 at.% Sn [Mas2] Hexagonal superlattice based on NiAs
ε_1, Cu_3Sn < 666	*oC*80 *Cmcm* Cu_3Sn	*a* = 552.9 *b* = 4775.0 *c* = 432.3	25 at.% Sn [Mas2]
ε_2, Ag_5Sn < 722	*hP*2 $P6_3/mmc$ Mg	*a* = 294.36 *c* = 478.45	11.8-22.85 at.% Sn [1987Kar]
θ, Ag_3Sn < 477	*oP*8 *Pmmn* βCu_3Ti	*a* = 596.82 *b* = 478.02	23.7-25 at.% Sn [1987Kar]

Table 2: Invariant Equilibria

Reaction	*T* [°C]	Type	Phase	Composition (at.%)		
				Ag	Cu	Sn
L + (Cu) ⇌ (Ag) + β	586	U_1	L	37.20	47.06	15.74
			(Cu)	1.10	91.57	7.33
			(Ag)	90.00	3.61	6.39
			β	2.84	82.83	14.33
L + β ⇌ (Ag) + ε_1	556	U_2	L	38.42	41.58	20.00
			β	2.15	79.56	18.29
			(Ag)	88.45	2.33	9.22
			ε_1	0.00	75.00	25.00
L + (Ag) ⇌ ε_1 + ε_2	544	U_3	L	42.10	34.60	23.30
			(Ag)	87.06	1.68	11.26
			ε_1	0.00	75.00	25.00
			ε_2	86.02	0.79	13.19
L + ε_2 ⇌ ε_1 + θ	436	U_4	L	40.88	16.51	42.61
			ε_2	77.98	0.10	21.92
			ε_1	0.00	75.00	25.00
			θ	75.00	0.00	25.00

Reaction	T [°C]	Type	Phase	Composition (at.%)		
				Ag	Cu	Sn
$L + \varepsilon_1 \rightleftharpoons \theta + \eta$	375	U_5	L	26.68	8.75	64.57
			ε_1	0.00	75.00	25.00
			θ	75.00	0.00	25.00
			0	0.00	54.53	45.47
$L \rightleftharpoons \theta + \eta + (\beta Sn)$	217.7	E_1	L	3.54	1.06	95.40
			θ	75.00	0.00	25.00
			η	0.00	54.50	45.50
			(βSn)	0.07	0.01	99.92
$\beta \rightleftharpoons L + \varepsilon_1$	661	e_2 (max)	β	0.27	74.13	25.60
			L	4.54	61.28	34.18
			ε_1	0.00	75.00	25.00

Table 3: Thermodynamic Description of the Ag-Cu-Sn System

Ag-Cu: [1986Hay]
Ag-Sn: [1999Oht]
Cu-Sn: [2001Liu]
Ternary parameters for the liquid phase (in J·mol^{-1})
L(LIQUID,AG,CU,SN;0) = –63565
L(LIQUID,AG,CU,SN;1) = –99000
L(LIQUID,AG,CU,SN;2) = –22418

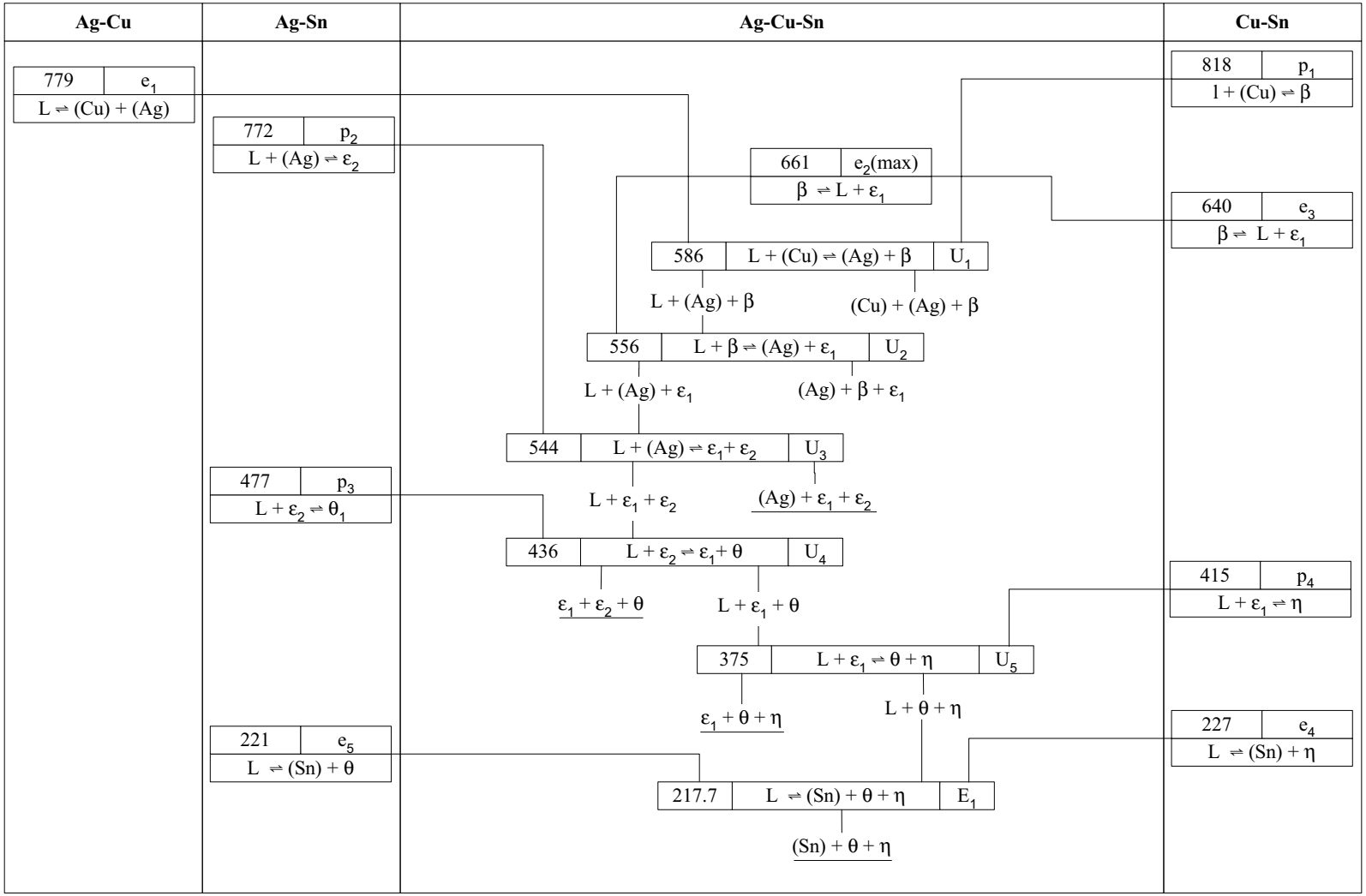

Fig. 1: Ag-Cu-Sn. Part of the reaction scheme involving the liquid phase

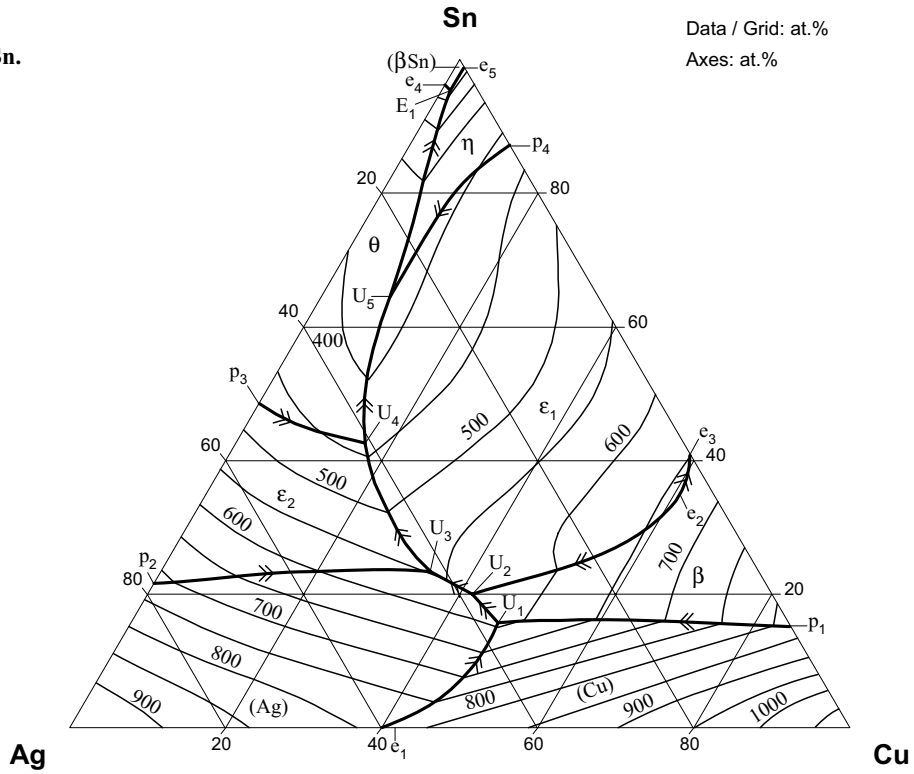

Fig. 2: Ag-Cu-Sn. Liquidus surface projection

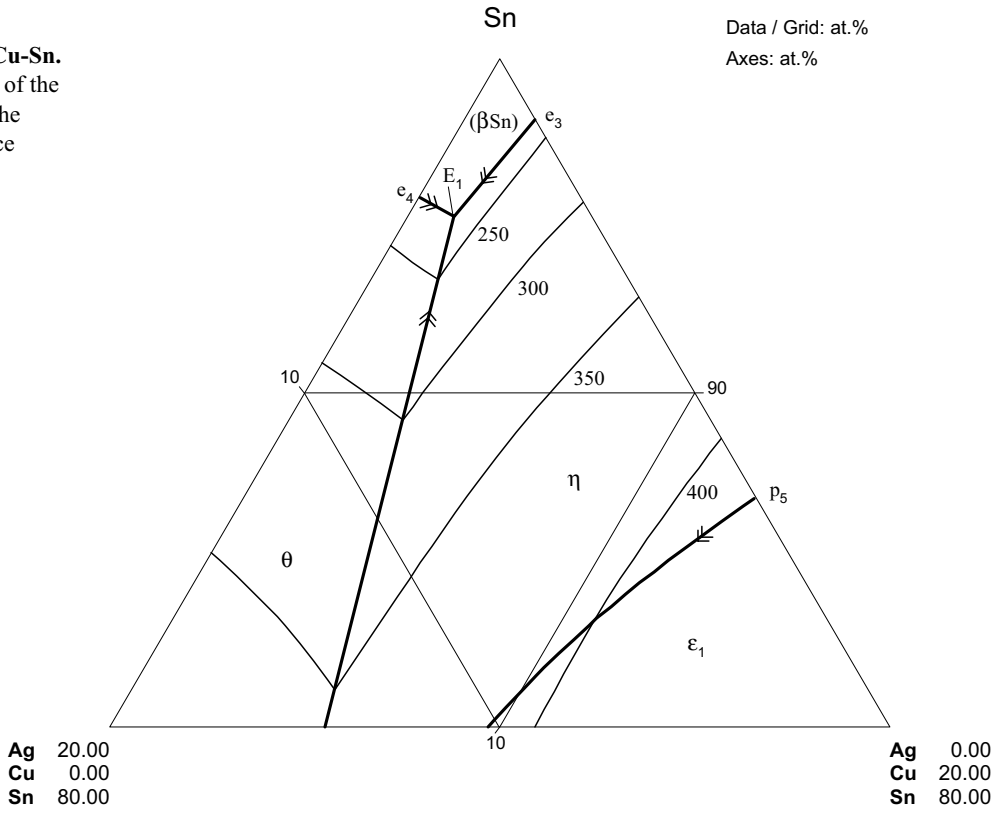

Fig. 3: Ag-Cu-Sn. Sn rich corner of the projection of the liquidus surface

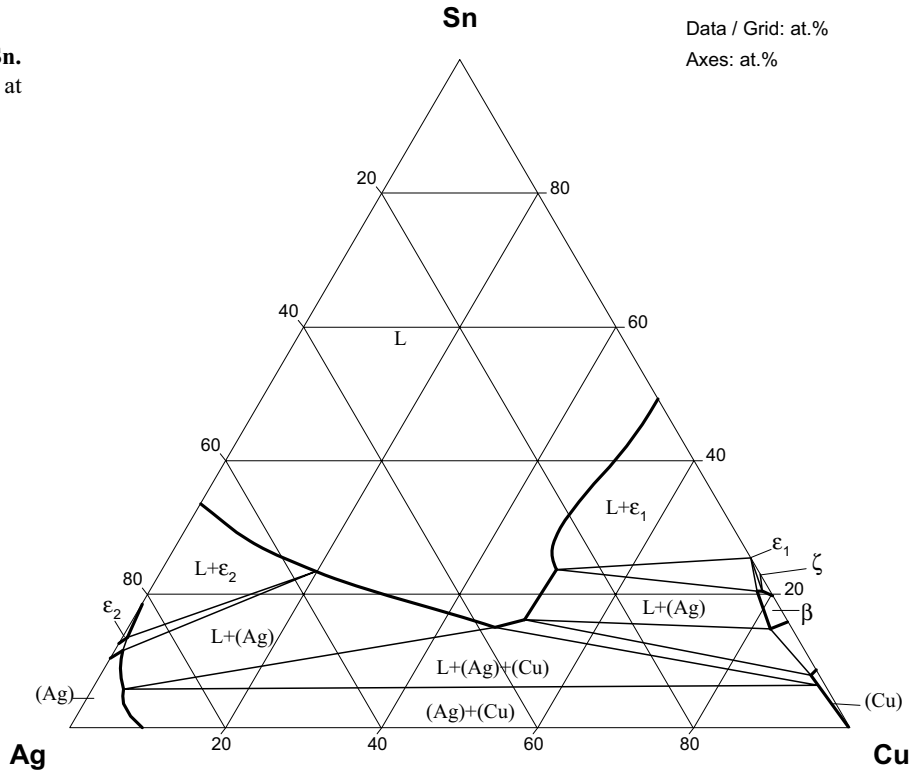

Fig. 4: Ag-Cu-Sn. Isothermal section at 600°C

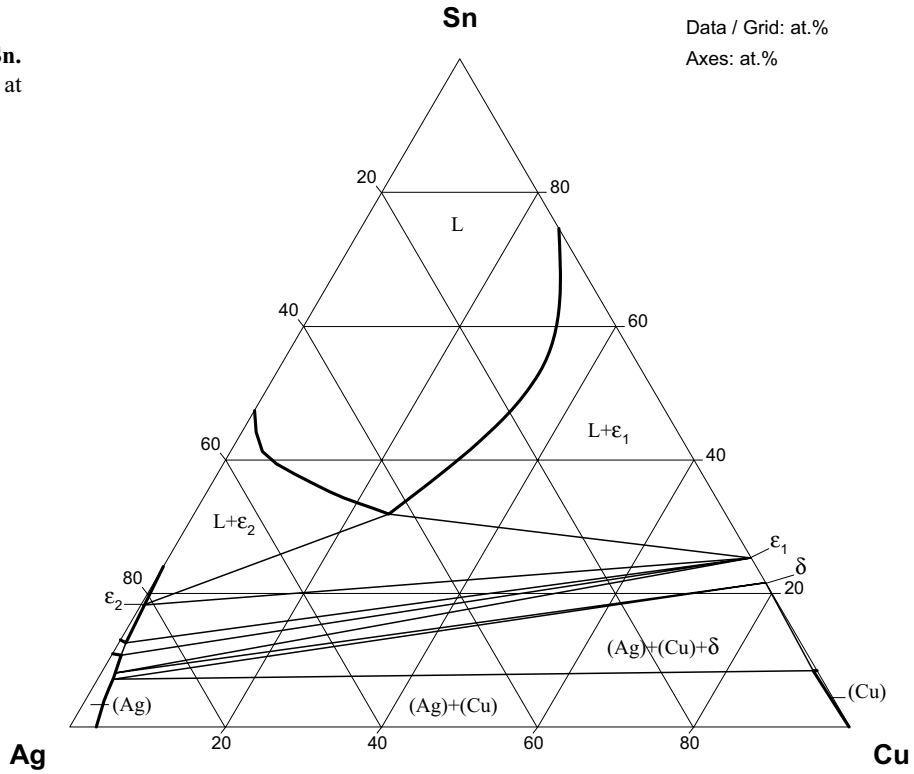

Fig. 5: Ag-Cu-Sn. Isothermal section at 500°C

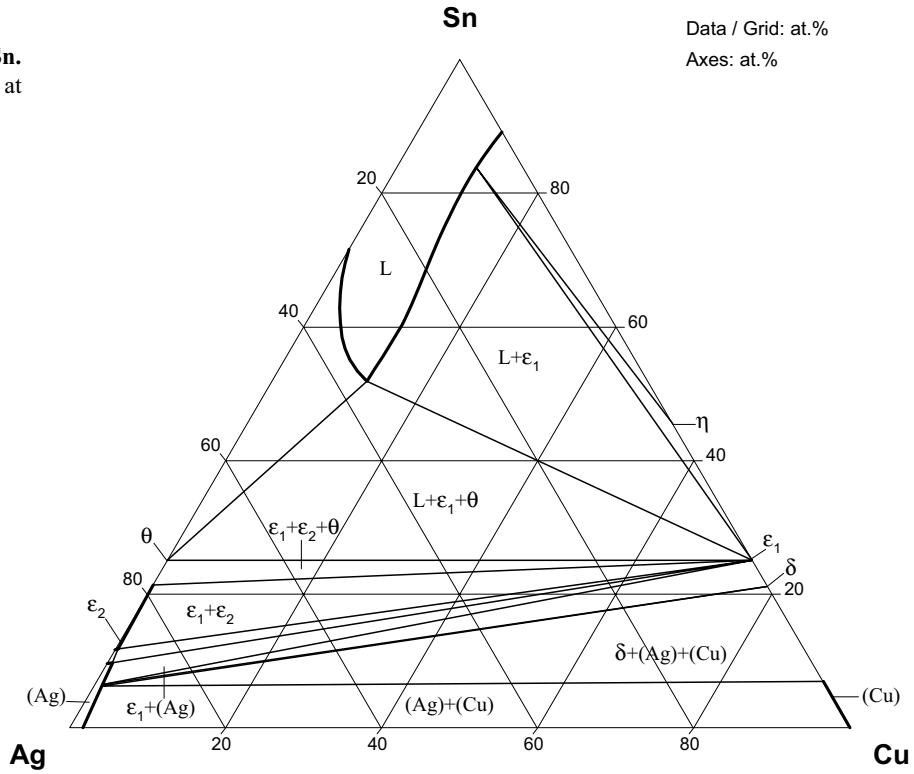

Fig. 6: Ag-Cu-Sn. Isothermal section at 400°C

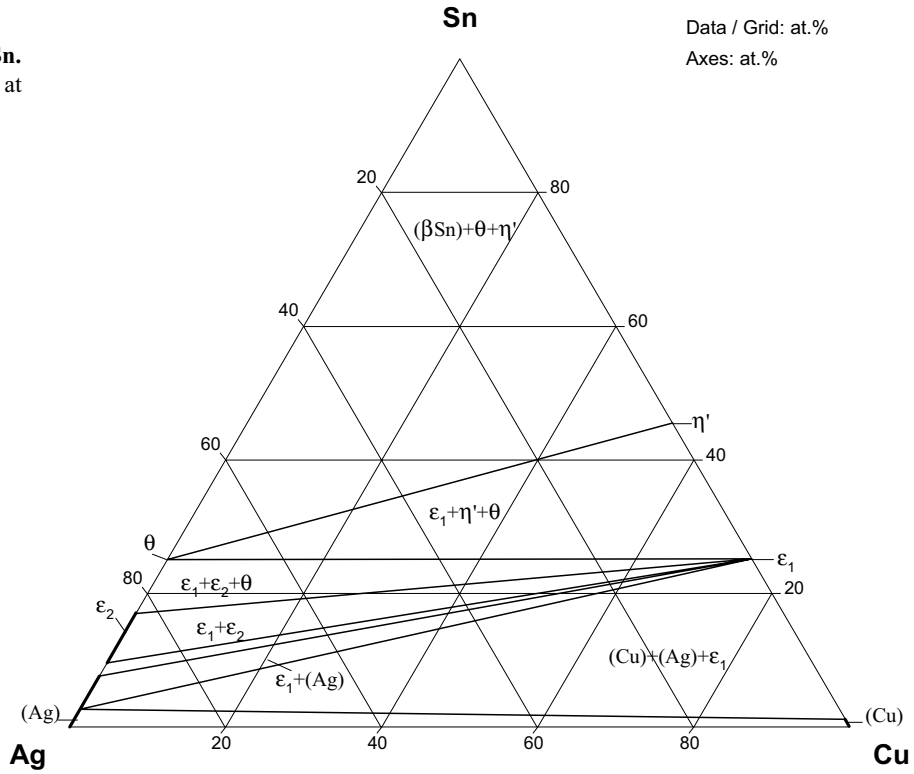

Fig. 7: Ag-Cu-Sn. Isothermal section at 37°C

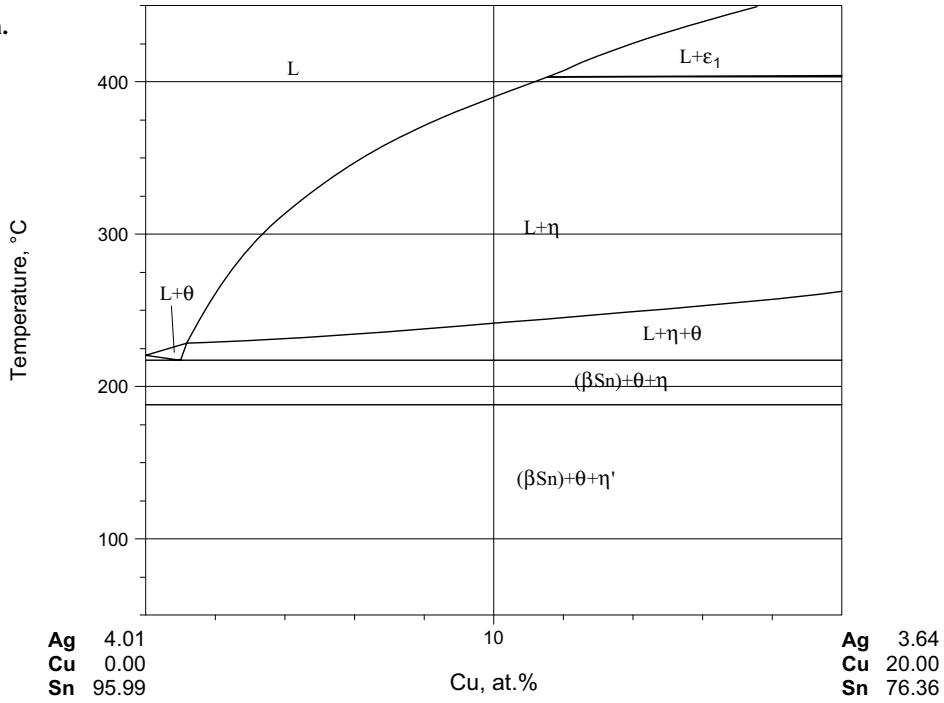

Fig. 8: Ag-Cu-Sn. Vertical section at 3.66 mass% Ag, plotted in at.%

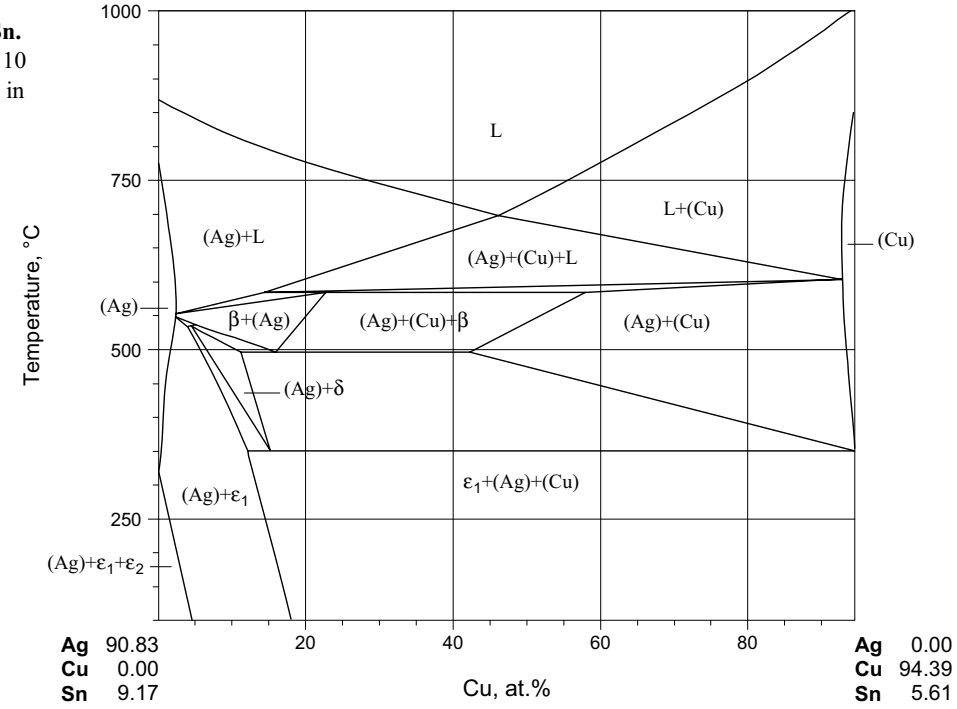

Fig. 9: Ag-Cu-Sn. Vertical section at 10 mass% Sn, plotted in at.%

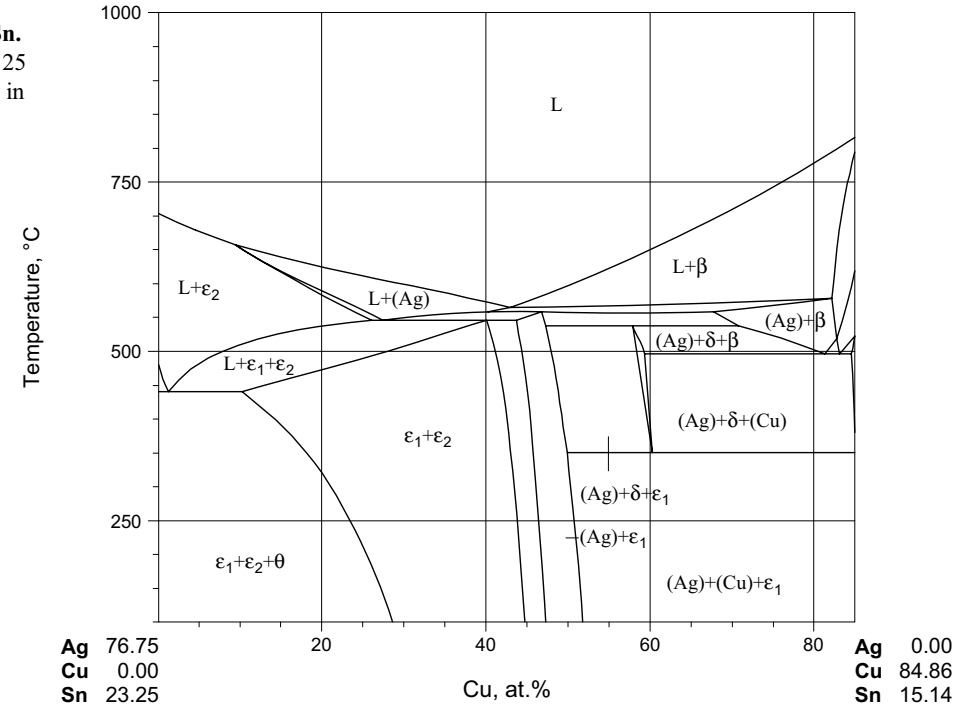

Fig. 10: Ag-Cu-Sn. Vertical section at 25 mass% Sn, plotted in at.%

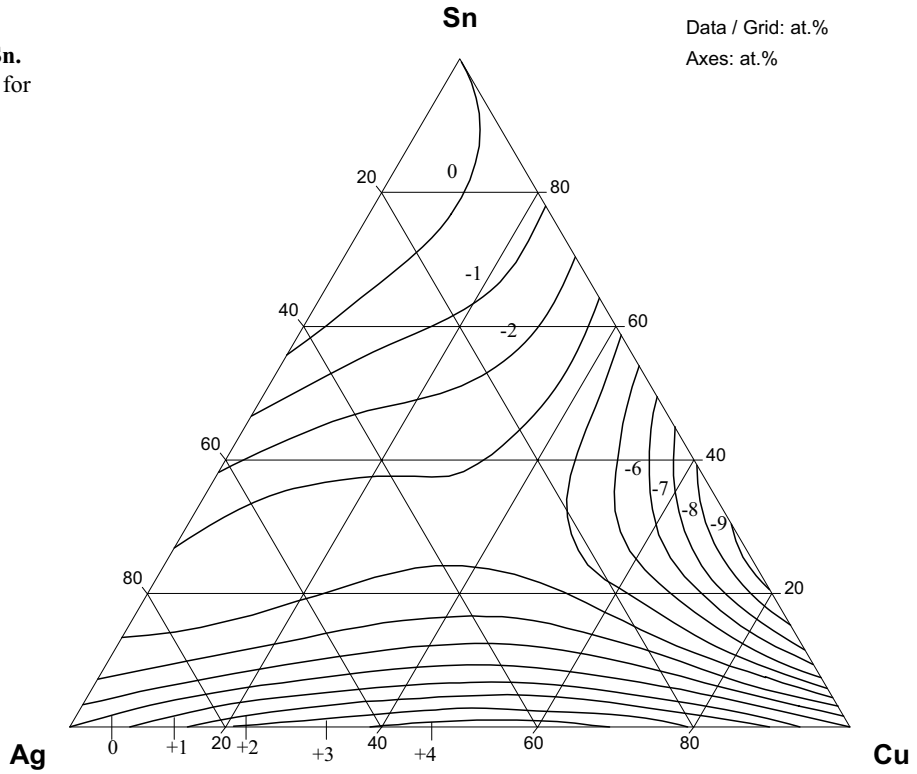

Fig. 11: Ag-Cu-Sn. Isoenthalpy curves for Ag-Cu-Sn liquid alloys at 500°C

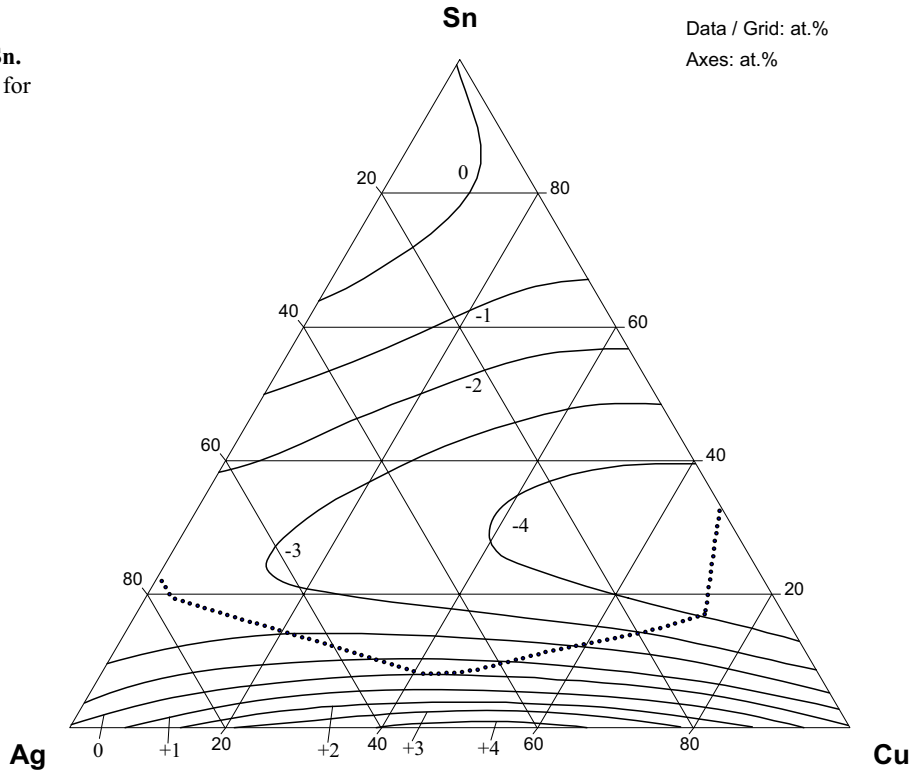

Fig. 12: Ag-Cu-Sn. Isoenthalpy curves for Ag-Cu-Sn liquid alloys at 700°C

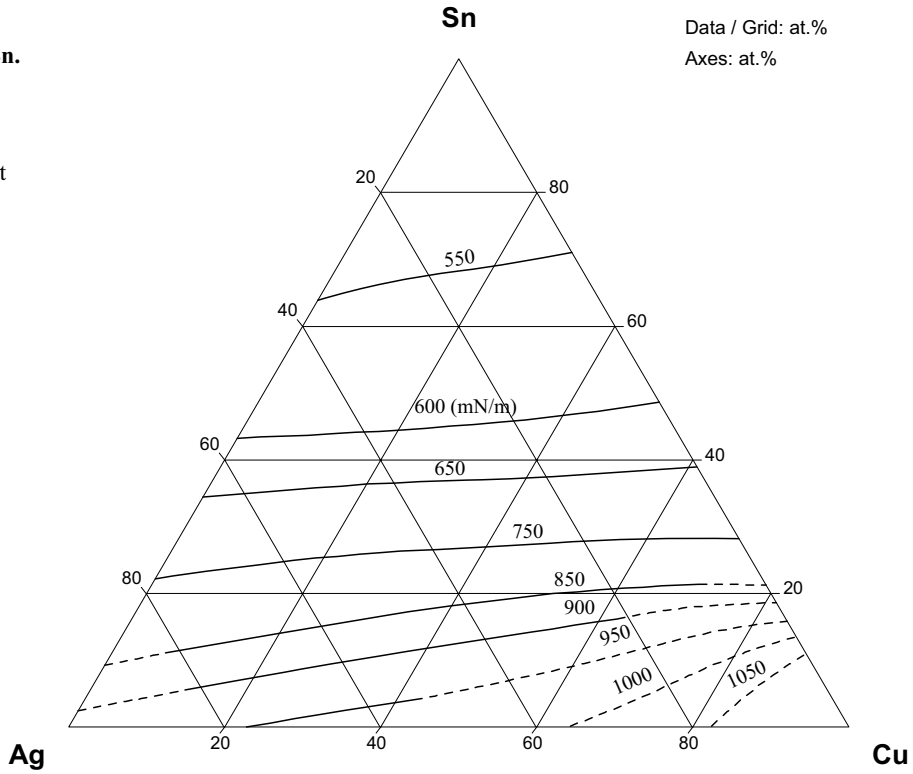

Fig. 13: Ag-Cu-Sn. Calculated surface tension (in mN/m) contours for liquid Ag-Cu-Sn alloys at 800°C

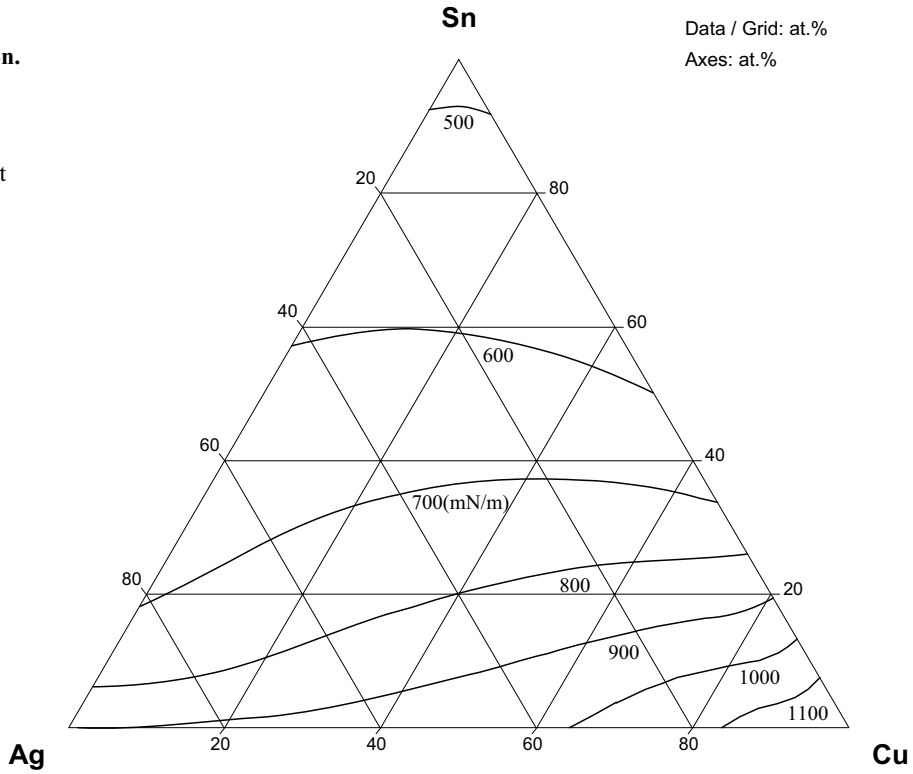

Fig. 14: Ag-Cu-Sn. Calculated surface tension (in mN/m) contours for liquid Ag-Cu-Sn alloys at 1000°C

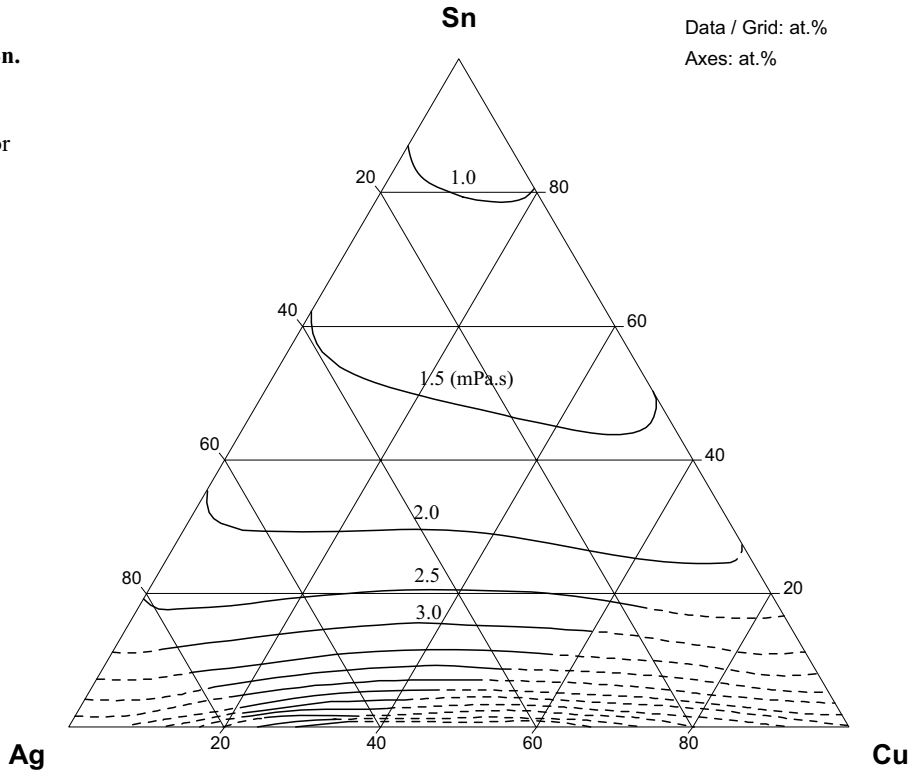

Fig. 15: Ag-Cu-Sn. Calculated isoviscosity (in mPa·s) contours for liquid Ag-Cu-Sn alloys at 800°C

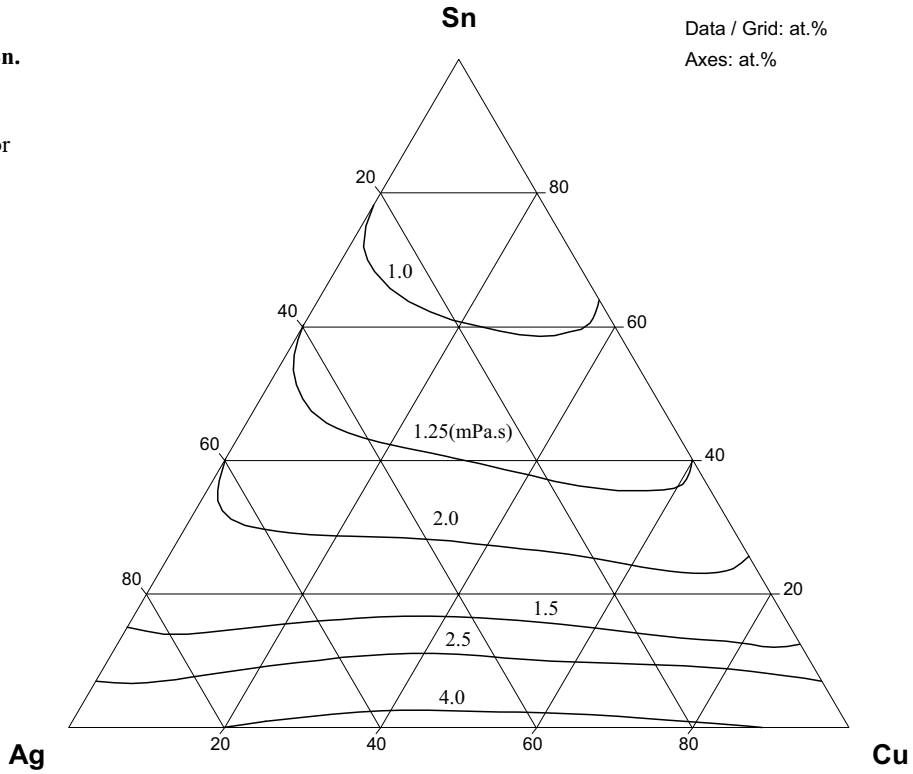

Fig. 16: Ag-Cu-Sn. Calculated isoviscosity (in mPa·s) contours for liquid Ag-Cu-Sn alloys at 1000°C