Gold – Copper – Tin

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Introduction

Interdiffusion in a diffusion sample of the composition electrolytic Cu (99.95%, 5000 μm)/Ag (15μm)/Au (13 μm)/Sn-Pb solder (41.5% Pb, 100 μm) resulted in the occurrence of three ternary compounds, $AuCu_5Sn_5$, $Au_2Cu_4Sn_5$ and $Au_3Cu_3Sn_5$. These were formed between 110 and 212°C [1971Cre]. However, the existence of the first two phases was not confirmed in subsequent studies.

The Au poor part of the Au-Cu-Sn system was investigated by [1988Roe1, 1988Roe2] using metallography and differential scanning calorimetry. Starting materials were 99.999% pure Au, Cu and Sn. Alloys were induction melted under an argon atmosphere, annealed under vacuum in silica or pyrex ampoules for various time periods and water quenched. Diffusion couples were prepared from binary Au-Cu alloys which were hot-worked and annealed for 48 h at temperatures ~25 K below the solidus temperature. After polishing they were pressed against sheets of Sn and annealed at 170°C in evacuated capsules. 232 h was the annealing time for the AuCu/Sn and Au_3Cu/Sn couples and 256 h for the $AuCu_3$/Sn couple. The specimens were water quenched and examined by electron probe microanalysis.

Solid state diffusion couples and ternary alloys were examined to establish an isothermal section at 170°C. Three ternary phases, Φ (~25 at.% Au, 55 at.% Cu, 20 at.% Sn), χ (~33.3 at.% Au, 33.3 at.% Cu, 33.3 at.% Sn), and κ (approximately, $Au_3Cu_3Sn_5$) were found and their crystal structures were determined by convergent beam electron diffraction. The Φ phase, denoted τ_1 in this assessment, extends along the Au-Cu axis. The χ compound - τ_2 in this review - has an AuCuSn formula. The κ ($Au_3Cu_3Sn_5$) compound, denoted τ_3 in the present review, was earlier reported by [1971Cre]. A liquidus surface and a reaction scheme for the Au poor part of the system (< 50 at.% Au) were proposed.

[1990Kar] and [1992Kar1] performed density measurements and used X-ray diffraction, metallography, scanning electron microscopy and differential thermal analysis to study the phase equilibria of the Au-Cu-Sn system. They prepared specimens by fusing (for 1 min at 1100 to 1200°C under shaking) 99.99% Au, 99.9% Cu, and 99.98% pure Sn in evacuated and sealed silica-glass tubes. The molten specimens were quenched into water and then annealed at 360°C. An isothermal section at 360°C was established. It contains three ternary compounds A, B, and C labeled τ_4, τ_1 and τ_2 respectively, in the present review. B and C correspond to those phases detected by [1988Roe1, 1988Roe2] and A was found to contain 20 at.% Sn, 42 to 47% Au and 38 to 33% Cu.

According to [1990Kar, 1992Kar1, 1992Kar2, 1992Kar3] τ_1 forms through a solid-state reaction, whereas [1988Roe2] revealed this phase in equilibrium with the liquid (see Figs. 2 and 3). τ_2 is formed through a solid state reaction at about 370°C. [1990Kar] and [1992Kar1] found the δ(AuSn) and η_1(Cu_6Sn_5) phases to form a continuous series of solid solutions and the phases β and ζ (both Au-Sn) as well as ε_1 (Cu_3Sn), to extend into the ternary system. [1992Kar1] examined the phase constitution in the section $Au_{80-x}Cu_xSn_{20}$. Two invariant four-phase equilibria at ~515 and ~320°C were revealed. Based on these data, [1992Kar1] derived tentative partial isothermal sections at ~450°C, ~320°C as well as just above and below 320°C. [1990Kar, 1992Kar2] and [1992Kar3] determined the crystal structure of the ternary phases τ_1, τ_2 and τ_4.

Using X-ray powder diffraction, scanning electron microscopy/electron microprobe analysis, and other analytical techniques, [1994Pep] revealed the ternary $Au_8Cu_8Sn_4$ and found it homogeneous in the range from $Au_7Cu_9Sn_4$ to $Au_9Cu_7Sn_4$. The alloys were prepared from pure metals by melting in corundum crucibles. The samples were molted for a while, and cooled with the rate of 2 $K \cdot min^{-1}$ to room temperature. The ingots were annealed at 175°C for 7 and 240 d. The above $Au_8Cu_8Sn_4$ ternary phase exhibited the crystal structure same as the [1988Roe1, 1988Roe2] and [1990Kar, 1992Kar1] compounds, both denoted τ_1 in the present assessment.

Based on the X-ray diffraction data, [2002Luc] constructed a partial vertical section AuCu-Sn up to 8 at.% Sn at the temperatures below 450°C. [2002Luc] prepared the alloys by melting the charge sealed in evacuated silica capsules in a high-frequency induction furnace. The ingots were forged at room

temperature, annealed during 20000 min at different temperatures from 450 to 200°C, and quenched into ice brine from an annealing temperature. In the AuCu-Sn section [2002Luc] showed two low-temperature ternary phases. One of them, AuCu(III) (τ_5 in the present assessment) appeared orthorhombic with eight atoms per unit cell similar to the ternary phase of the Au-Cu-Ga system and isomorphous to a Au_2CuZn phase of the Au-Cu-Zn system. The other low-temperature ternary AuCu(III)′ phase exhibited the cubic βMn type structure like the Φ [1988Roe1, 1988Roe2], [1990Kar, 1992Kar1], or $Au_8Cu_8Sn_4$ [1994Pep], which we denote τ_1 here.
[2002Huh] examined three alloys in the Sn corner of the Au-Cu-Sn system with 1, 2 and 3 mass% Au and constant 0.7 mass% Cu content, using differential scanning calorimetry, scanning electron microscopy, electron probe microanalysis and X-ray diffraction. [2002Huh] confirmed the solidification of ternary (Sn) + η + η′ eutectic and a large solid solubilities of Au in the η′ phase and Cu in the η phase.
In a review on employing X-ray microprobe to study diffusion, [1989Gol] discussed the data reported by [1988Roe1, 1988Roe2].

Binary Systems

The binary systems, Au-Cu and Cu-Sn, were accepted as given by [Mas2] and the Au-Sn binary phase diagram, as given by [1993Oka] (Fig. 1).

Solid Phases

Five ternary compounds were confirmed to exist in the Au-Cu-Sn system. [1988Roe2] reported the τ_1 and τ_3 compounds to precipitate from the liquid; however no data on the formation reactions is available. In the solidified alloys, the τ_1 compound was found to exist at 360°C [1990Kar, 1992Kar1], below 320°C [2002Luc], at 175°C [1994Pep], and 170°C [1988Roe1, 1988Roe2]; the τ_2 compound, at 170°C [1988Roe1, 1988Roe2] and 360°C [1990Kar, 1992Kar1]; τ_3, between 110 and 220°C [1971Cre, 1988Roe1, 1988Roe2]; τ_4, at 360°C [1990Kar, 1992Kar1], and τ_5, below 270°C [2002Luc]. The τ_1 compound has a homogeneity range that, at 360°C, extends from 11 to 38 at.% Au, from 69 to 42 at.% Cu, and from 18 to 20.5 at.% Sn. This homogeneity range becomes wider at 300°C, its lower limit shifting to 35 at.% Cu, and seems to reach even lower Cu contents below 300°C [1992Kar1]. The homogeneity range of the τ_2 phase extends from 33 to 37 at.% Au (33.7 to 29.7 at.% Cu) at 33.3 at.% Sn at 360°C [1990Kar, 1992Kar1]. The τ_3 phase was found homogeneous between 22.1 and 25.9 at.% Au, 22.5 and 26.8 at.% Cu, and 51.1 and 51.7 at.% Sn at 170°C [1988Roe1, 1988Roe2]. The τ_4 phase is homogeneous from 42 to 47 at.% Au and from 38 to 33 at.% Cu for 20 at.% Sn and 360°C. The minimum gold limit of the homogeneity range shifts to 15 at.% Au (65 at.% Cu) at 550°C [1990Kar].
Most of the binary phases extend into the ternary system. δ (AuSn) and η_1 (Cu_6Sn_5) form a continuous series of solid solutions $(Au_{1-x}Cu_x)Sn_{1-y}$ at 360°C [1990Kar, 1992Kar1], denoted δ in the present assessment. At 170°C, the δ phase dissolves about 15 at.% Cu and the η' phase, about 8 at.% Au [1988Roe1, 1988Roe2]. At 360°C, the ζ phase dissolves up to 50 at.% Cu, the ε_1 phase (Cu_3Sn), up to 17 at.% Au, and the β phase, up to 20 at.% Cu. The structural data for the binary and ternary phases are given in Table 1.

Invariant Equilibria

[1988Roe2] proposed a solidification reaction scheme for the Au poor part of the Au-Cu-Sn system. It is presented slightly modified in Fig. 2. [1988Roe2] deduced nine transition (U_1 to U_9) and one eutectic (E_2) reaction from the metallographic data obtained for the ternary alloys. Besides, the U_9 and E_2 reactions were also revealed by differential scanning calorimetry. Reactions U_9, $L + \tau_3 \rightleftharpoons \eta + \eta_1$, at 279°C, and U_8, $L + \varepsilon \rightleftharpoons \tau_3 + \eta$, above the U_9 temperature [1988Roe2], are unlikely as implying the η phase to be stable at higher temperatures in the ternary than in the binary system, which would only be possible with Cu soluble in η. Therefore, we describe the U_9 reaction as P and the U_8 reaction, as E_1. The solid-state invariant reactions are unknown and omitted in Fig. 2.

Two four-phase reactions in the Au rich part of the system were reported by [1992Kar1]. These are $\beta \rightleftharpoons Au_{1-x}Cu_x + \zeta + L$ at ~515°C and $L + \tau_4 \rightleftharpoons \zeta + \delta'$ at ~320°C. The binary reactions p_3, p_4 and e_2 indicated in Fig. 2 are connected with these four-phase equilibria.

Liquidus Surface

Based on microstructural examination and differential scanning calorimetry, a liquidus projection for the partial system below 35 at.% Au was mapped [1988Roe2] and is shown here (Fig. 3) corrected with respect to the P and E_1 reactions.

Isothermal Sections

Figure 4 demonstrates the isothermal section at 360°C in the Au-Cu-Sn system [1992Kar1], with corrections based on the binary data quoted by [Mas2, 1993Oka]. The section features the existence of the liquid phase in two single-phase, four two-phase and two three-phase regions. The two-phase regions are marked with tie lines. The δ_1 phase and region which involves ordered $AuCu_3$(I) have not been examined by experiment and are added tentatively by dashed lines.
Figure 5 depicts the partial isothermal section at 170°C based on [1988Roe2]. Phase equilibria in the Sn corner up to 50 at.% Cu and 50 at.% Au as well as the homogeneity ranges of the ternary τ_1, τ_2 and τ_3 compounds were experimentally determined by [1988Roe2]. We added a τ_5 ternary phase in the section shown in Fig. 5, reported by [2002Luc]. The binary phases on the Au-Cu side are added by dashed lines according to [Mas2]. Also [1988Roe2] indicated diffusion paths which may be interpreted as tie lines showing equilibrium between the phases τ_1 and τ_2, τ_2 and δ, τ_1 and (Au), τ_1 and $AuCu_3$(I), τ_1 and AuCu(I), τ_1 and Au_3Cu, τ_1 and η', τ_1 and ε_1. But there is no determination of the three phase equilibria with less than 50 at.% Sn.

Temperature – Composition Sections

Figure 6 demonstrates a partial vertical section AuCu-Sn [2002Luc] up to 8 at.% Sn in a temperature range from 450 to 200°C. The section reveals single-phase and multiphase regions including ordered AuCu(I), AuCu(II), τ_1, and τ_5 phases and disordered $Au_{1-x}Cu_x$ solid solution. The τ_5 phase appears below 320°C for the Sn contents of and above 2 at.% in the alloys, and the τ_1 phase, below 270°C with the Sn content growing from 4 at.%.

Thermodynamics

[1969She] determined heats of solution of gold and copper in dilute Au-Cu-Sn alloys at 720°C, using a liquid metal solution calorimeter.

Notes on Materials Properties and Applications

[1974Alc] measured the chemical potentials of tin in $Au_{1-x}Cu_x$ solid solutions with Cu + Au alloys using a gas-solid equilibration technique.
[1984Kim] examined the microstructure, electrical resistivity, hardness and soldering contact strength of the Cu based alloys added with about 1 mass% Au and from 1 to 25 mass% Sn; [1984Kim] also examined the microstructure of thin films of these alloys obtained by e-beam evaporation onto aluminium oxide substrates. All samples were finally annealed for 30 min at 250°C. [1984Kim] pointed out that the structures of annealed thin-film and bulky alloys are similar to one another and, therefore, the thin-film properties can be predicted as measured with the bulky specimens and respectively corrected for the scale factor.
[2002Huh] examined influence of small (to 1 mass%) additions of Au on the tensile properties of a Sn-0.7Cu (mass%) alloy. The best combination of strength and ductility was achieved with 0.3 mass% Au owing to solution hardening.

[2004Kis] measured the electrical resistivity and solder tensile strength of solder eutectic Au-Sn alloy with 0.46 at.% Cu and shown the promise of this alloy for providing a replacement for Pb-Sn alloys in electronic assemblies.

Miscellaneous

[1993Zak] examined the effect of Kirkendall pore formation in the Au-Cu-Sn system on the adhesion strength of the inner lead bond contacts. The contact-diffusion layer was formed between contacting 80/20 eutectic Au-Sn alloy and Cu. The sandwich specimens were annealed at the temperatures between 125 and 200°C for up to 2000 h. Adhesion in the inner lead bond contacts was found to primarily depend on the mechanism of pore formation, growth, and coagulation. [1993Zak] reported on two new ternary phases, defined as ternary compounds. The first phase consisted of 40 to 50 at.% Cu, 40 to 50 at.% Au, and up to 20 at.% Sn and the second phase, of 15 to 20 at.% Cu, 65 to 75 at.% Au and 10 to 12 at.% Sn. Judging from the [1992Kar1, 1992Kar2, 1992Kar3] data, we can identify the ternary τ_1 compound with the first phase, whereas the second phase should be considered as a ternary solid solution based on the binary ζ phase of the Au-Sn system.
[1997Ven] used differential scanning calorimetry to investigate the solidification kinetics of a transient liquid phase in Au and Sn layers electroplated on Cu foil. [1997Ven] showed that this liquid phase had a gold rich eutectic composition for the used thicknesses of Sn (2 μk) and Au (6 μk) and a heating rate of 10 $K \cdot min^{-1}$. The solidification reaction takes place between this liquid and the excess solid gold. The solidification time is a strong function on the gold-to-tin thickness ratio and tends to infinity as the net composition approaches the ζ phase solidus. At temperatures up to 295°C, the diffusion of copper into gold is not significant enough to affect the solidification kinetics.
[2001Lee, 2003Dua] investigated the effect of the under bump metallurgy (UBM) structure on the redeposition rate of Au-containing intermetallic compounds at the solder/UBM interface during the aging treatments. Au/Ni/Cu [2001Lee] and Ni(P)/Au and Ni(V)/Cu [2003Dua] compositions were used as the UBM structures and Sn-37Pb (mass%), Sn-3.5Ag (mass%), Sn-3.5Ag- 0.7Cu (mass%), as the solder alloys. Copper dissolution from UBM into the solder, as well as the addition of Cu into the solder, were found to be effective in retarding the redeposition of the Au containing intermetallic compounds and in preventing the Au embrittlement.

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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
$Au_{1-x}Cu_x$	*cF*4 *Fm*$\bar{3}$*m* Cu		$0 \le x \le 1$
(Au) < 1064.43		*a* = 407.82	at *x* = 0 and 25°C [Mas2]
(Cu) < 1084.87		*a* = 361.46	at *x* = 1 and 25°C [Mas2]
Sn(r) 231.97 - 13	*tI*4 $I4_1/amd$ Sn(r)	*a* = 583.18 *c* = 318.18	at 25°C [Mas2]
Sn(l) < 13	*cF*8 *Fm*$\bar{3}$*m* C(diamond)	*a* = 648.92	[Mas2]
β, $Au_{10}Sn$ < 532	*hP*16 $P6_3/mmc$ $TiNi_3$		8.2 to 9.1 at.% Sn [1993Oka], dissolves up to 20 at.% Cu at 360°C [1992Kar1]
		a = 290.2 *c* = 951.0	[V-C]
ζ < 521	*hP*2 $P6_3/mmc$ Mg	-	9.1 to 17.6 at.% Sn [1993Oka], dissolves up to 50 at.% Cu at 360°C [1992Kar1]
ζ′, Au_5Sn < 190	*hR*6 *R*$\bar{3}$ Au_5Sn	 *a* = 509.2 *c* = 1433.3	[1993Oka] [V-C]
δ′, $(Au_{1-x}Cu_x)Sn_{1-y}$	*hP*4 $P6_3/mmc$ NiAs	-	at 360°C, $0 \le x \le 1$ and $0 \le y \le 0.17$ [1990Kar, 1992Kar1]
δ, AuSn < 419.3			at *x* = 0, *y* = 0 [1993Oka], dissolves up to 18 at.% Cu at 170°C [1988Roe2]
		a = 432.18 *c* = 552.30	[V-C2]
η_1, ≈Cu_6Sn_5 415 - 186			at *x* = 1, *y* = 0.17, [1993Oka], dissolves up to 15 at.% Au at 170°C [1988Roe2] quenched
		a = 419.2 *c* = 503.7	[Mas2, V-C2]
ε, $AuSn_2$ < 309	*oP*24 *Pbca* $AuSn_2$	*a* = 690.9 *b* = 703.7 *c* = 1178.9	[1993Oka, V-C2]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
η, $AuSn_4$ 252 - ~60	*oC*20 *Aba*2 $PtSn_4$	*a* = 650.2 *b* = 654.3 *c* = 1170.5	[1993Oka, V-C2]
β_1, $Cu_{17}Sn_3$ 798 - 586	*cI*2 $Im\bar{3}m$ W	*a* = 302.61	13.1 to 16.5 at.% Sn [Mas2, V-C2]
γ_1, Cu_3Sn 755 - 520	*cF*16 $Fm\bar{3}m$ BiF_3	 *a* = 611.66	15.5 to 27.5 at.% Sn [Mas2] at 700°C [V-C2]
δ_1, $Cu_{41}Sn_{11}$ 590 - ~350	*cF*416 $F4\bar{3}m$ $Cu_{41}Sn_{11}$	*a* = 1798.0	20 to 21 at.% Sn [Mas2, V-C2]
ζ_1, $Cu_{10}Sn_3$ 638 - 582	*hP*26 $P6_3$ $Cu_{10}Sn_3$	*a* = 733.0 *c* = 786.4	20.3 to 22.5 at.% Sn [Mas2, V-C2]
ε_1, Cu_3Sn < 676	*oC*80 *Cmcm* Cu_3Sn	 *a* = 552.9 *b* = 477.5 *c* = 432.3	24.5 to 25.9 at.% Sn [Mas2], dissolves up to 17 at.% Au at 360°C [1992Kar1] at 25 at.% Sn [V-C2]
η´, ≈ Cu_6Sn_5 < 189	*h*** - superstructure of NiAs	 *a* = 2087.0 *c* = 2508.1	45 at.% Sn [Mas2], dissolves up to 15 at.% Au at 170°C [1988Roe2] [1973Gan]
Au_3Cu < 240	*cP*4 $Pm\bar{3}m$ $AuCu_3$	*a* = 396.5	10 to 38.5 at.% Cu [Mas2, V-C2]
AuCu(II) < 410	*oI*40 *Imma* AuCu(II)	*a* = 367.6 *b* = 395.6 *c* = 397.2	38.5 to 63 at.% Cu [Mas2, V-C2]
AuCu(I) < 385	*tP*4 *P*4/*mmm* AuCu	*a* = 396.3 *c* = 367.1	42 to 57 at.% Cu [Mas2, V-C2]
$AuCu_3$(II) 390 - 255(?)	*tP*28 *P*4*mm* $PdCu_3$	-	66 to ? at.% Cu [Mas2]
$AuCu_3$(I) < 390	*cP*4 $Pm\bar{3}m$ $AuCu_3$	*a* = 374.8	67 to 81 at.% Cu [Mas2, V-C2]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
* τ_1, $Au_{80-x}Cu_xSn_{20}$	*cP*20 $P4_132$ βMn		$42 \le x \le 69$, at 360°C [1992Kar1,1992Kar3]
		$a = 676.2 \pm 0.2$	at $x = 45$ [1992Kar3]
		$a = 666.2 \pm 0.2$	at $x = 60$ [1992Kar3]
		$a = 673.61 \pm 0.01$	at $x = 47$, annealed at 175°C [1994Pep]
		$a = 678.32 \pm 0.03$	at $x = 40$ [1994Pep]
		$a = 681.29 \pm 0.01$	at $x = 30$ [1994Pep]
* τ_2, AuCuSn	*tI*12 *I*4/*mmm* La_2Sb	$a = 407.8 \pm 0.1$ $c = 1293.8 \pm 0.1$	at 360°C for $Au_{24.9}Cu_{31.8}Sn_{33.3}$ [1992Kar2]
		$a = 408.8 \pm 0.3$ $c = 1300.7 \pm 0.6$	at 360°C for $Au_{37}Cu_{29.7}Sn_{33.3}$ [1990Kar]
* τ_3, $Au_3Cu_3Sn_5$ or $AuCuSn_2$	-	-	[1971Cre, 1988Roe1, 1988Roe2]
* τ_4, $Au_{80-x}Cu_xSn_{20}$	*cI*52 $I\bar{4}3m$ Cu_5Zn_8		$31 \le x \le 37$, at 360°C or $31 \le x \le 65$, at 550°C [1990Kar]
		$a = 918.4 \pm 0.3$	at $x = 60$, quenched from 550°C
		$a = 948.4 \pm 0.6$	at $x = 32$, quenched from 360°C
* τ_5	*oP*8 *Pbam* Au_2CuZn	$a = 899.1$ $b = 454.2$ $c = 285.9$	[2002Luc]

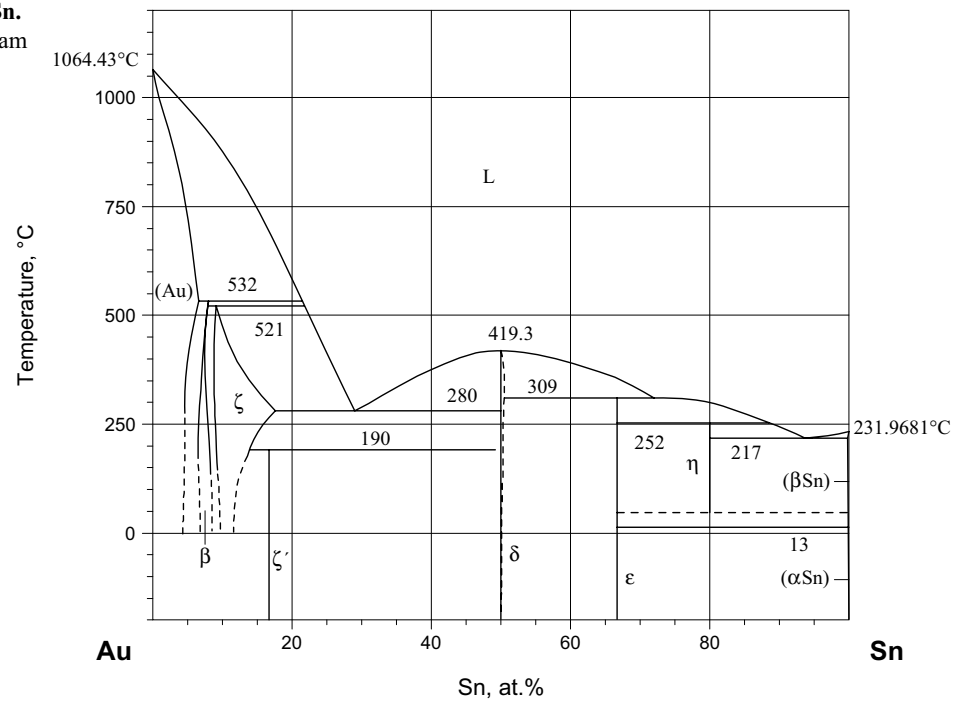

Fig. 1: Au-Cu-Sn. Au-Sn phase diagram [1993Oka]

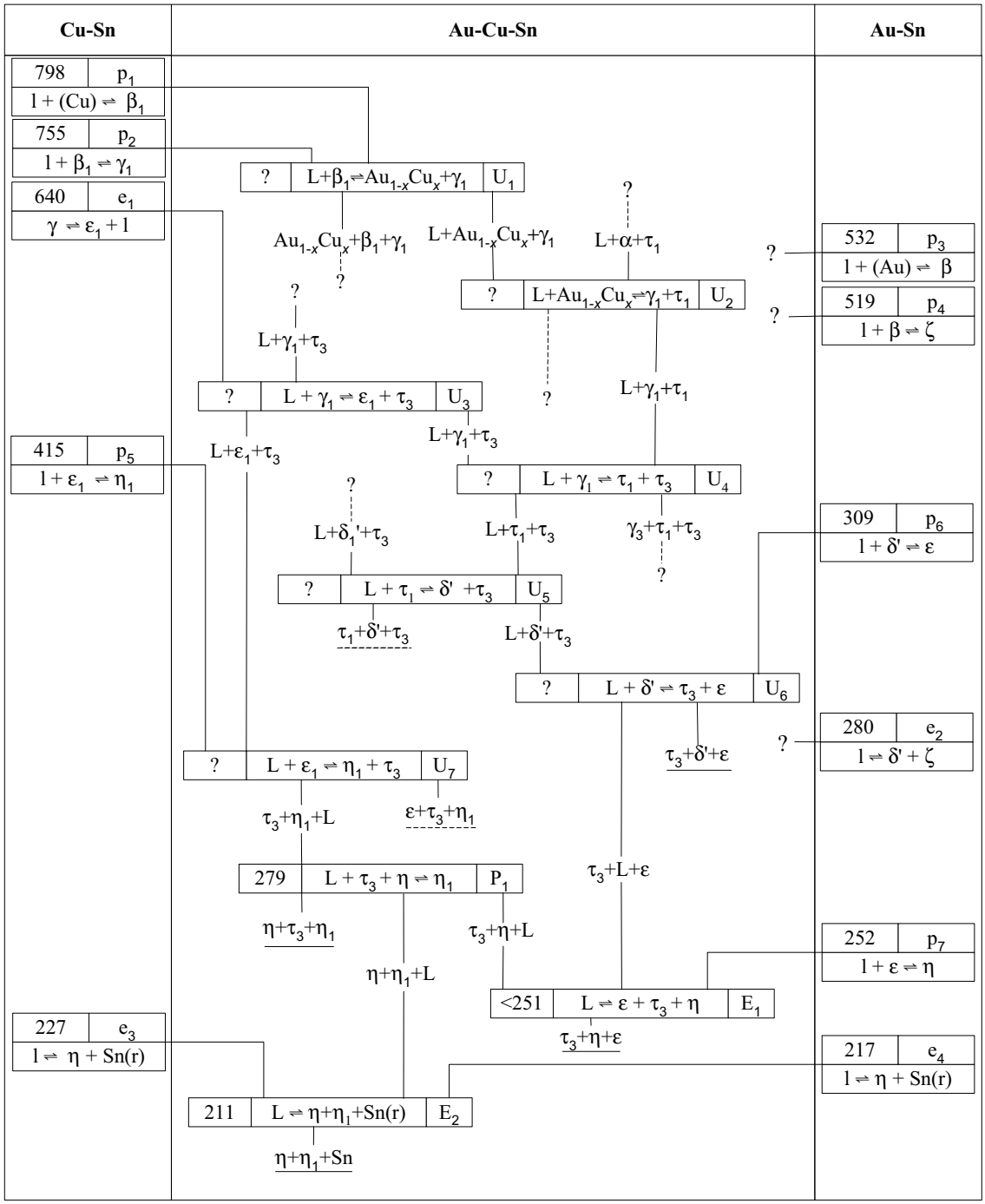

Fig. 2: Au-Cu-Sn. Reaction scheme

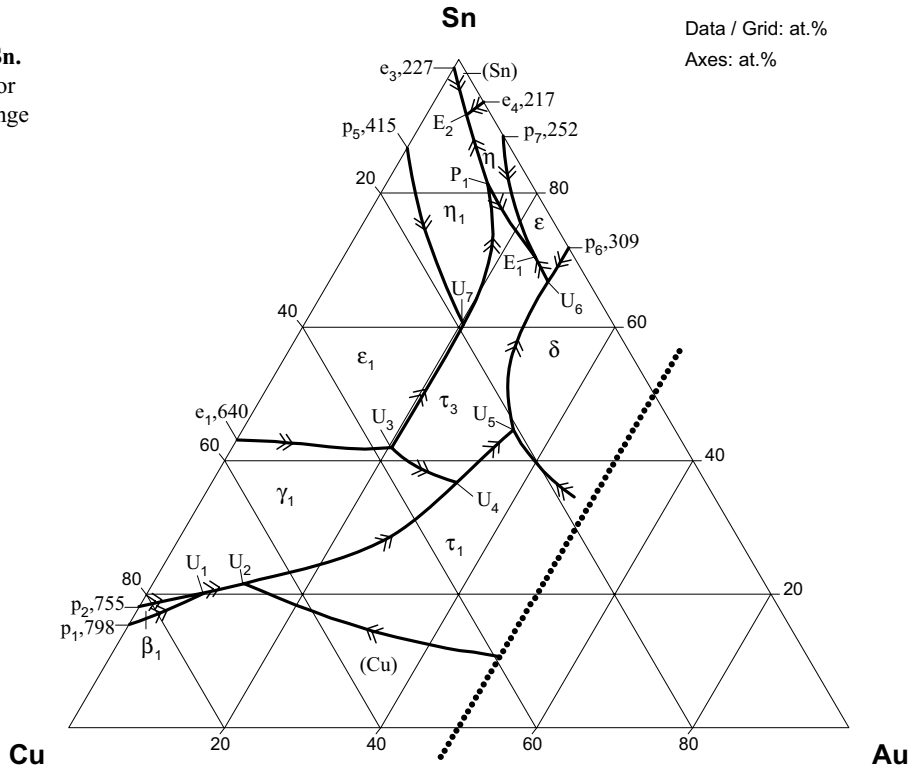

Fig. 3: Au-Cu-Sn. Liquidus surface for the composition range below 35 at.% Au

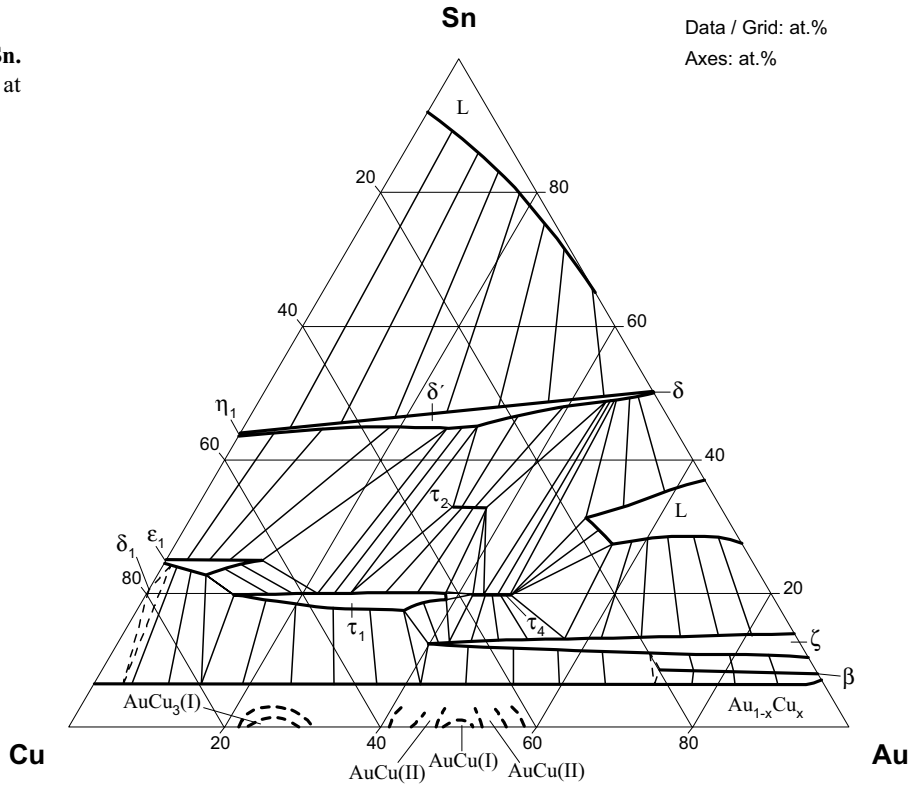

Fig. 4: Au-Cu-Sn. Isothermal section at 360°C

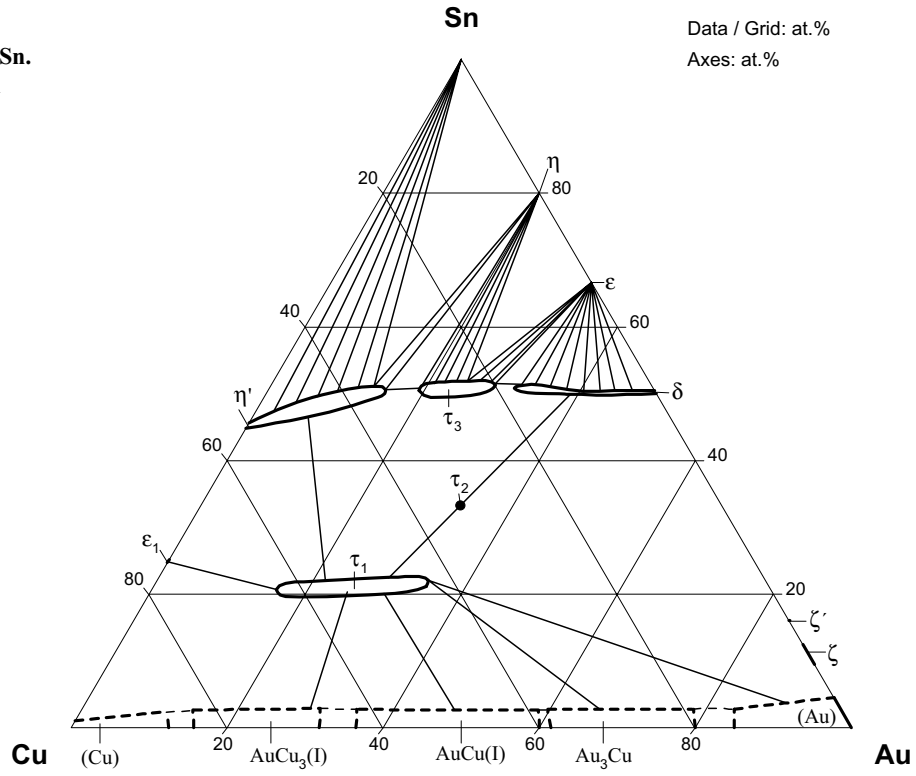

Fig. 5: Au-Cu-Sn. Partial isothermal section at 170°C

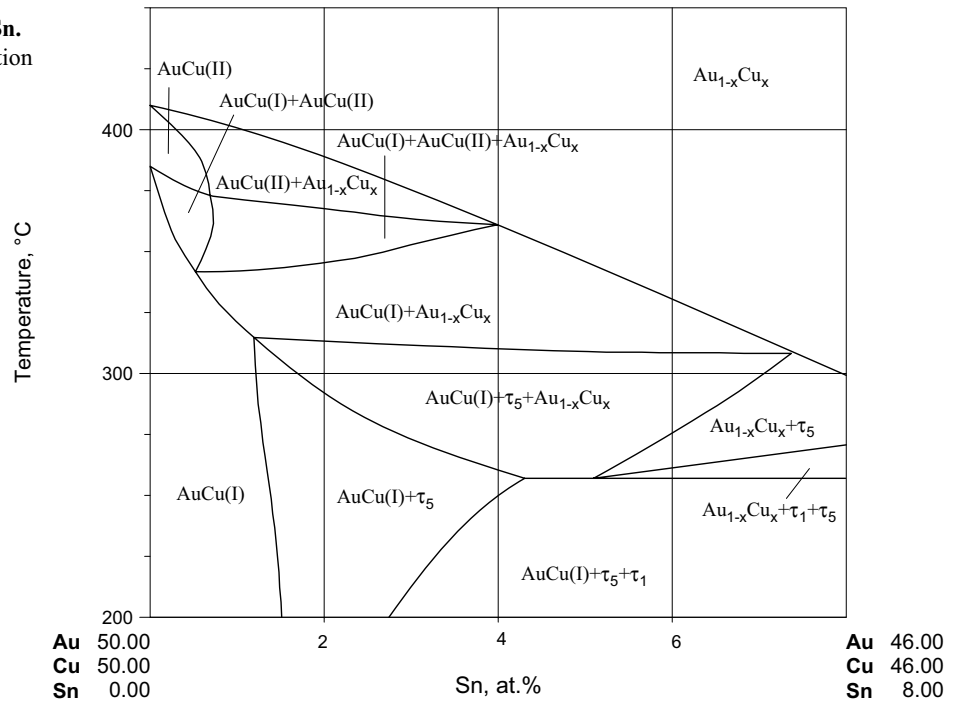

Fig. 6: Au-Cu-Sn. Partial vertical section AuCu - Sn