Copper – Niobium – Tin

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Introduction

Practical interest in the Cu-Nb-Sn ternary system started with the discovery of the bronze process for the production of Nb_3Sn superconductors. Filaments of Nb are drawn down in a Cu-Sn bronze matrix and reacted to produce a fully stabilized Nb_3Sn wire which has very good superconducting properties [1978Dew]. The success of this process for making Nb_3Sn is partly due to the fact that there are no stable ternary compounds in the Cu-Nb-Sn system [1987Nei] and no interference of a more stable phase between Nb_3Sn and (Cu). Isothermal sections have been investigated between 675 and 1100°C mainly by diffusion of tin-copper alloys with niobium.

Binary Systems

Cu-Nb, assessed by [1982Cha] and Cu-Sn, assessed by [1990Sau] are accepted as given by [Mas2]. As for the Nb-Sn system, [Mas2] gives a diagram redrawn from [S], which is anterior to 1969. [1970Cha] reported the most extensive investigation on the Nb-Sn phase diagram by diffusion experiment and detected the formation of Nb_3Sn, Nb_6Sn_5 and $NbSn_2$ in a 6 weeks diffusion experiment at 650°C. Figure 1 gives the Nb-Sn system as proposed by [1970Cha] and later accepted by [1977Kru, 1984Ino, 1987Nei]. Below 800°C, the absence of Nb_3Sn and Nb_6Sn_5 is due to the low diffusion rate rather then to the instability of the compounds.

Solid Phases

Solid phases are given in Table 1. The crystal parameter and the critical temperature of Nb_3Sn, measured at 16.5 K are nearly independent of the stoichiometry [1996Sve]. There is no ternary compounds whose existence has been clearly established. However, diffusion experiment made by [2001Nau] suggests the formation of an intermetallic phase whose approximate composition is $Cu_2Nb_3Sn_8$. The existence of this compound has not been confirmed and its crystal structure is unknown.

Liquidus Surface

The liquidus surface has been qualitatively drawn by [1972Die] and more precisely investigated by [1975Zwi, 1979Cha, 1979Dri]. The melting grooves separating 10 areas of primary crystallization are shown in Fig. 2. The dominant feature is the large field of primary crystallization of Nb_3Sn and (Nb). The 8 remaining fields stand close to the Cu-Sn border, containing less than 0.15 at.% Nb. The invariant equilibria given in Table 2 are from [1979Cha, 1979Dri]. Although these authors present the invariant point at 225°C as a ternary peritectic, it is more surely a eutectic.

Isothermal Sections

The isothermal section at 675°C given Fig. 3 has been mainly investigated by [1987Nei]. Compositional analysis of intermetallic compounds formed in diffusion couples bronze-niobium after 528 h and 675 h shows that Nb_3Sn and Nb_6Sn_5 may dissolve 3 at.% Cu whereas the solubility of copper in $NbSn_2$ does not exceed 0.6 at.%. [1975Zwi] gives an isothermal section at 785°C without formation of the β,$Cu_{17}Sn_3$ phase. [1972Die] presents at 700°C an isothermal section showing a three-phase field Nb-Nb_3Sn-liquid $Cu_{45}Sn_{55}$ which is questionable. On the other hand, [1974Sha] prepares a $Cu_{93}Nb_3Sn_2$ melt followed by an annealing at 700°C and shows the precipitation of Nb_3Sn crystals in the copper matrix. [1975Sch] charges Cu-Nb alloys with Sn by a vapor transport reaction and forms Nb_3Sn by a diffusion reaction during 21 h at 725°C. [1980Pan] prepares 105 alloys by the method of electric arc welding followed by 100 h annealing at 800°C and proposes an isothermal section [1980Pan, 1981Pan] showing solubilities of Nb in liquid Cu-Sn higher

than 10 at.%. Such high solubilities coming from the oversaturation of the melt are not confirmed by further studies. However, [1977Kru] shows that the solubility of Nb in liquid Cu-Sn (0 to 20 at.% Cu) increases with Cu content (see Fig. 1). Figure 4 gives an isothermal section at 800°C, mainly from [1980Pan, 1983Bar] in essential agreement with the isothermal section given at 785°C by [1979Cha].
Phase relations at 1000°C (Fig. 5) and 1100°C (Fig. 6) have been determined from diffusion experiments by [1975Zwi, 1977Hop, 1980Sav]. The isothermal section proposed at 1000°C by [1977Hop] is preferred because [1975Zwi] gives a three-phase field (Nb)-Nb_3Sn-liquid $Cu_{95}Sn_5$. Such a section prohibits the existence of a tie line between (Cu) and Nb_3Sn, which contradicts further experimental evidences given by [1977Hop, 1978Dew, 1980Pan, 1983Bar, 1987Nei]. The solubility of Nb in liquid is small; less than 5 at.% Nb are sufficient to crystallize the Nb_3Sn phase from most liquid compositions.

Notes on Materials Properties and Applications

The Cu-Nb-Sn system is important because the alloys are used in engineering as superconducting materials for the production of high magnetic fields and in the transmission of alternative current electrical power [1999Hel]. The Nb_3Sn based composite superconductors are prepared by the so-called “bronze technique” [1996Pop]: multifilamentary Nb is prepared in a Cu-Sn matrix and superconducting Nb_3Sn layers are obtained by diffusion. The plastic deformation behavior of such composites has been investigated by [2000Pop] which showed that, with increasing the degree of deformation, the dislocation density decreases in the bronze matrix and increases in the Nb filaments. The critical current increases with the Sn concentration up to the single phase limit of Sn in Nb_3Sn, then decreases with the excess of Sn once the solubility limit reached [1974Sue]. The structure formed by long term diffusion of Cu-Sn alloys and Nb metal has been shown to consist of fcc (Cu) and A15 ($Pm\bar{3}n$) Nb_3Sn phases. Small filaments of Nb_3Sn fabricated in a ductile copper base matrix may present a critical current density of 10 kA·cm^{-2} at 4.2 K under a magnetic field of 12 T [1977Fon]. Similar results are obtained when preparing Cu-Nb alloys by a chill casting technique. After drawing a fine wire, Nb_3Sn is formed by plating and diffusion of Sn [1978Ver].
Superconducting Nb_3Sn presents a high critical temperature, from 17.6 K ($Nb_{81}Sn_{19}$) to 18.3 K ($Nb_{75}Sn_{25}$) and carries large [1975Gol1, 1975Gol2] superconducting currents in very high magnetic fields. The dissolution of copper does not change significantly the critical temperature [1979Efi, 1984Ino], but increases by one order of magnitude the critical current density [1974Sha] and improves the capacity of shielding high magnetic fields of Nb_3Sn [1976Lef].
High cooling rates (10^6-10^7 K·s^{-1}) from the Nb-Sn melts in presence of Cu favors the appearance of some quantities (10-20 vol%) of a metastable superconducting phase whose critical temperature lies between 13.8 and 14.1 K [1978Sav, 1980Sav]. If pure copper is not a superconductor, even at low temperatures, a small amount of dispersed superconducting particles in a copper matrix induces superconductivity. The best superconducting properties (critical temperature of 12.0 K and critical current density of 130 kA·cm^{-2}) were observed in a $Cu_{99.55}Nb_{0.30}Sn_{0.15}$ alloy quenched from the liquid state and aged at 550°C for 384 h [1978Nag]. Superconductivity may also be obtained from a $Cu_{92.5}Nb_5Sn_{2.5}$ alloy rapidly quenched into a powder by ultrasonic gas atomization [1991Ino]. The powder has been consolidated by hot extrusion at 650°C then annealed for 100 h at the same temperature in order to synthesize Nb_3Sn. The critical temperature of the as quenched alloy is 8.0 K, but increases to 15.6 K after annealing. The critical current density is 74 kA·cm^{-2} at zero applied field and 4.2 K.
The solid state diffusion behavior in a layered Cu-Nb/Cu couple was investigated by using very thin Nb filaments as a marker for studying the Kirkendall phenomena [1998Luo]. Results show that the Nb filaments, during annealing at 400-450°C, shift towards the Cu-Nb side, which is due to the volume expansion resulting from the formation of the ε,Cu_3Sn phase prior to A15 (Nb_3Sn) formation. A similar observation has been performed by [2001Nau] which pointed out the impossibility of a complete mixing of Cu and Sn before the formation of Nb_3Sn, so that the Nb filaments have to react with different Cu-Sn phases, however, without any consequences for the superconducting properties of the fully reacted samples.
Formation of an amorphous phase may be obtained by mechanical alloying of the $Cu_{44}Nb_{42}Sn_{14}$ and $Cu_{45}Nb_{35}Sn_{20}$ alloys, which are in a domain where no amorphous phase may be obtained by rapid quenching, but not with the $Cu_{45}Nb_{50}Sn_5$ alloy, not rich enough in tin [1987Ino]. Indeed, amorphization

requires a negative heat of mixing providing the necessary chemical driving force, which is not realized in an alloy in which the two major constituents, Cu and Nb present repulsive interactions. The amorphous alloy $Cu_{44}Nb_{42}Sn_{14}$ present, in differential scanning calorimetry, a broad exothermic peak between 480 and 590°C, which corresponds to the precipitation of copper [1988Mat].

Miscellaneous

Ingots of the ternary composites (Cu-13% Sn)-30% Nb and (Cu-8% Sn)-30%Nb has been prepared by direct crystallization in a high temperature gradient (~40 $K \cdot mm^{-1}$) and quenched at a cooling rate (~100 $K \cdot s^{-1}$) high enough to prevent the formation of the brittle compound Nb_3Sn [1998Arz]. The internal friction measurements showed the existence of a minimum around ~175°C, which is correlated with the temperature at which the "plasticity dip" occurs in tin bronzes. Wetting angles of liquid Sn or Cu-Sn alloys on Cu, Nb or Cu-Nb substrates was measured by [1988Mor]. The fraction of Nb or Cu in the solid substrate was considered as a controlling factor in wetting behavior.

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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
(Cu) < 1084.62	*cF*4 $Fm\bar{3}m$ Cu	 *a* = 361.6 *a* = 368.8	0 to 9.1 at.% Sn [Mas2] 0 to 0.1 at.% Nb at 1080°C 0 at.% Sn [V-C2] 6.8 at.% Sn [V-C2]
(Nb) < 2469	*cI*2 $Im\bar{3}m$ W	*a* = 330.04	0 to 1.2 at.% Cu at 1080°C [Mas2]
(βSn)	*tI*4 $I4_1/amd$ βSn	*a* = 583.18 *c* = 318.18	~ 0 at.% Cu [Mas2] ~ 0 at.% Nb [Mas2]
(αSn) < 13	*cF*8 $Fd\bar{3}m$ C diam.	*a* = 648.92	~ 0 at.% Cu [Mas2] ~ 0 at.% Nb [Mas2]
β, ~$Cu_{17}Sn_3$ 798-586	*cI*2 $Im\bar{3}m$ W	*a* = 302.61	13.1 to 16.5 at.% Sn [Mas2] at 710°C and 15 at.% Sn [V-C2]
γ, ~Cu_3Sn 755 - 520	*cF*16 $Fm\bar{3}m$ BiF_3	 *a* = 611.66	15.5 to 27.5 at.% Sn [Mas2] at 710°C [V-C2]
δ, ~$Cu_{41}Sn_{11}$ 585 - 350	*cF*416 $F\bar{4}3m$ $Cu_{41}Sn_{11}$	*a* = 1798.0	20 to 21 at.% Sn [Mas2, V-C2]
ζ, ~Cu_7Sn_2 640 - 582	*hP*26 $P6_3$ $Cu_{10}Sn$	*a* = 733.0 *c* = 786.4	20.3 to 22.5 at.% Sn [Mas2, V-C2]
ε, ~Cu_3Sn < 676	*oC*80 *Cmcm* Cu_3Sn	*a* = 552.9 *b* = 4777.5 *c* = 432.3	24.5 to 25.9 at.% Sn [Mas2, V-C]
η, ~Cu_6Sn_5(h)	*hP*4 $P6_3/mmc$ NiAs	*a* = 419.2 *c* = 503.7	43.5 to 45.5 at.% Sn [Mas2, V-C2]
η', ~Cu_6Sn_5(r) < 189	*h***	*a* = 2087 *c* = 2508.1	44.8 to 45.5 at.% Sn [Mas2] superstructure of η [V-C2]
Nb_3Sn < 2130	*cP*8 $Pm\bar{3}n$ Cr_3Si	*a* = 528.5	16.1 to 26.7 at.% Sn [1970Cha], Dissolves up to 3 at.% Cu [1987Nei]. Crystal parameter from [1996Sve]
Nb_6Sn < 930	*oI*44 *Immm* Sn_5Ti_6	*a* = 565.6 *b* = 919.9 *c* = 1684.3	44.9 to 46.4 at.% Sn [1970Cha] Dissolves 3.4 at.% Cu [1987Nei]
$NbSn_2$ < 845	*oF*48 *Fddd* $CuMg_2$	*a* = 986.0 *b* = 564.77 *c* = 1912.7	66 to 68.6 at.% Sn [1970Cha] [V-C2]

Table 2: Invariant Equilibria

Reaction	T [°C]	Type	Phase	Composition (at.%)		
				Cu	Nb	Sn
L + (Nb) ⇌ (Cu) + Nb_3Sn	1075	U_1	L	99.8	0.15	0.05
L + (Cu) ⇌ β + Nb_3Sn	788	U_2	L	84.4	0.1	15.5
L + β ⇌ γ + Nb_3Sn	750	U_3	L	80.9	0.1	19
L + Nb_3Sn ⇌ γ + Nb_6Sn_5	670	U_4	L	~ 70	0.1	~30
L + Nb_6Sn_5 ⇌ γ + $NbSn_2$	650	U_5	L	~ 63	0.1	~37
L + γ ⇌ ε + $NbSn_2$	630	U_6	L	56.9	0.1	43
L + ε ⇌ η + $NbSn_2$	410	U_7	L	13.3	0.1	86.6
L ⇌ η + $NbSn_2$ + (βSn)	225	E	L	1.3	0.1	98.6

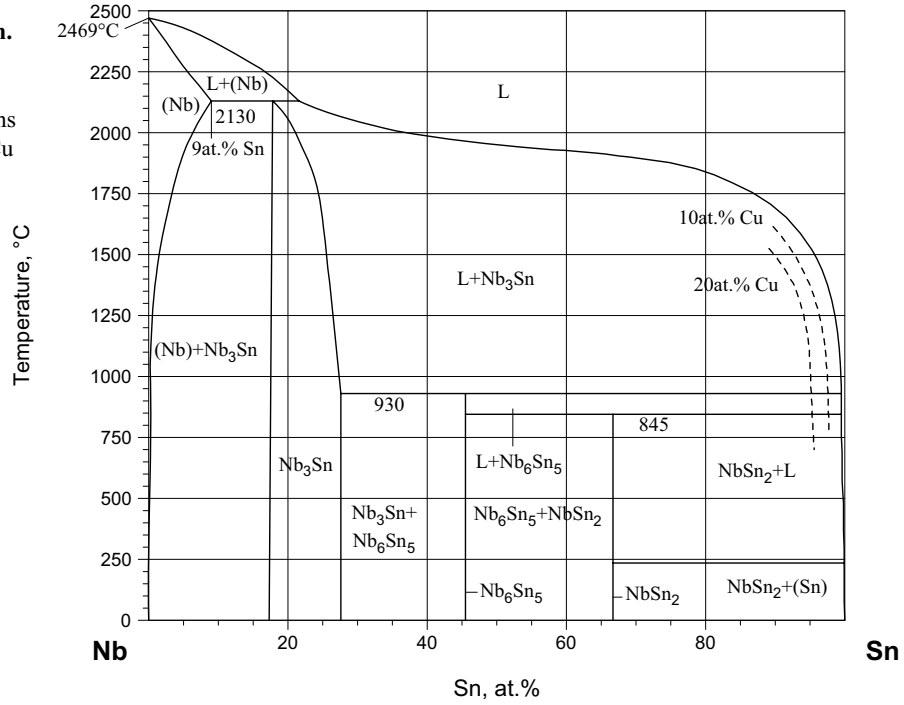

Fig. 1: Cu-Nb-Sn. The Nb-Sn binary system [1970Cha] with vertical sections at 10 and 20 at.% Cu

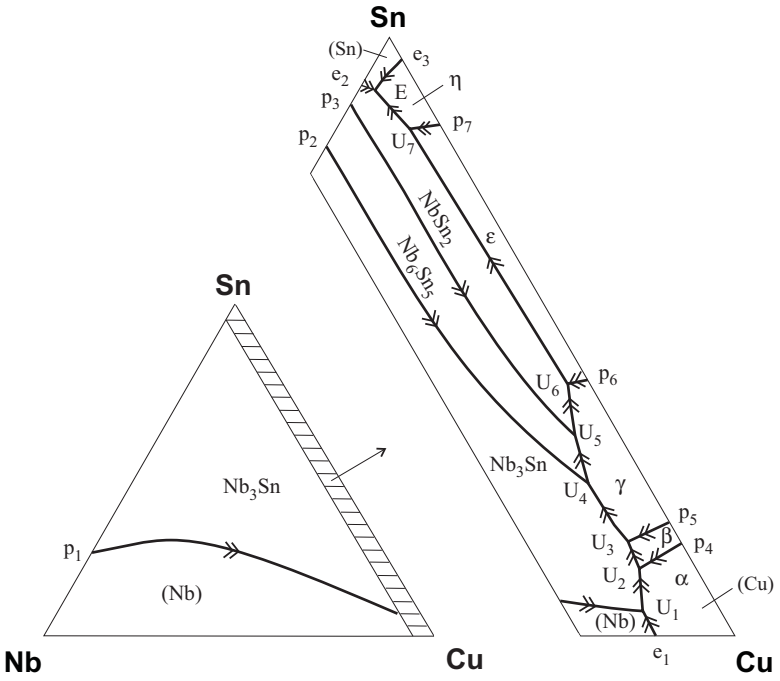

Fig. 2: Cu-Nb-Sn. Melting grooves and fields of primary crystallization (schematic)

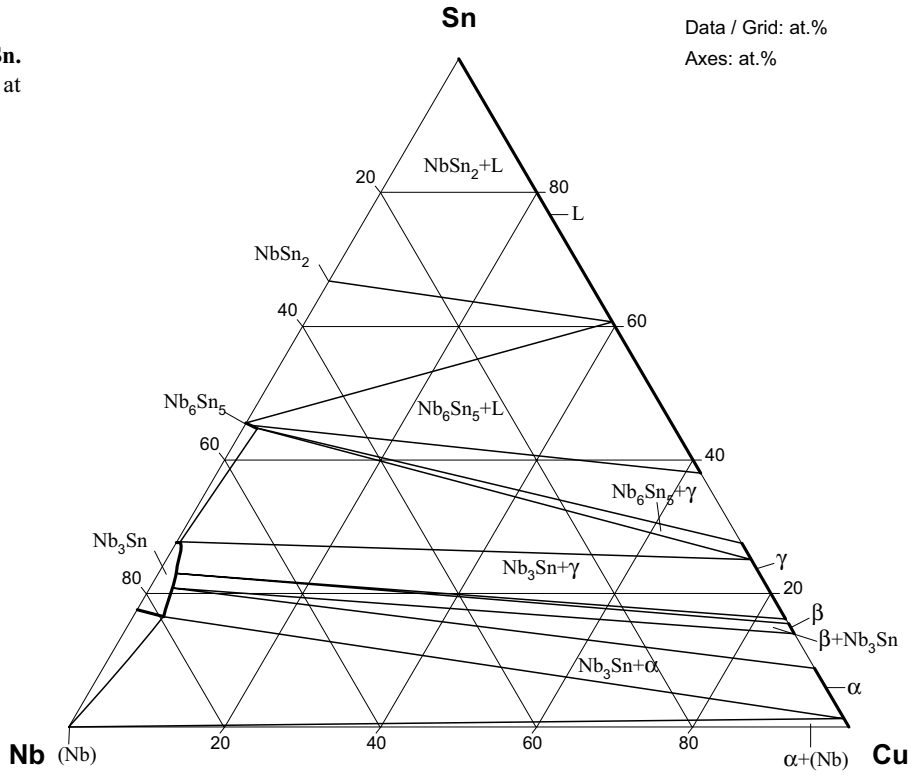

Fig. 3: Cu-Nb-Sn. Isothermal section at 675°C

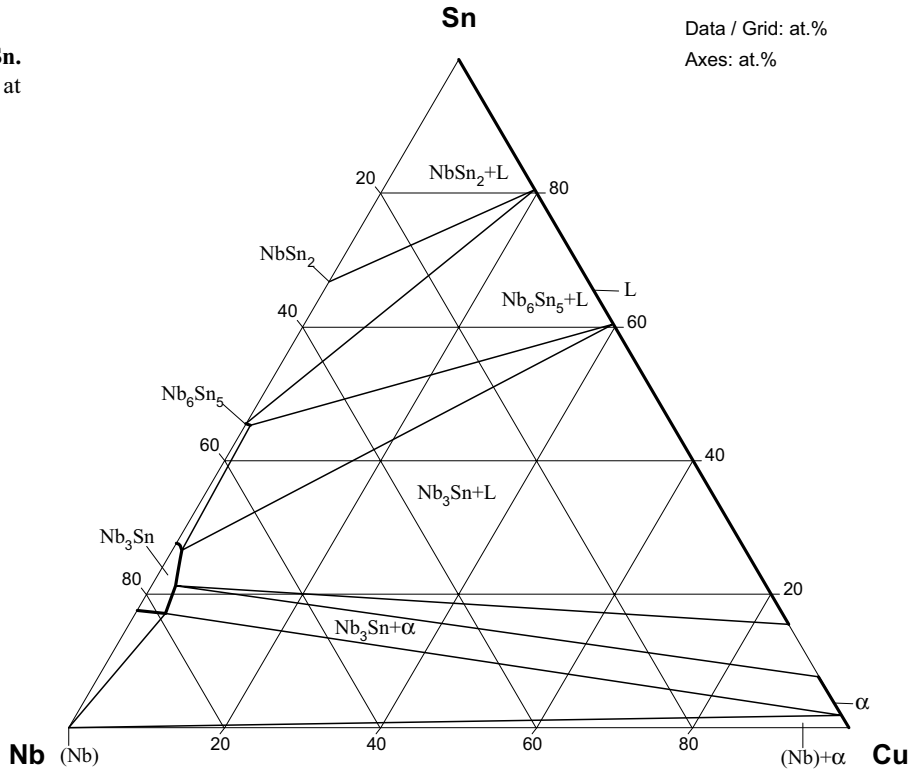

Fig. 4: Cu-Nb-Sn. Isothermal section at 800°C

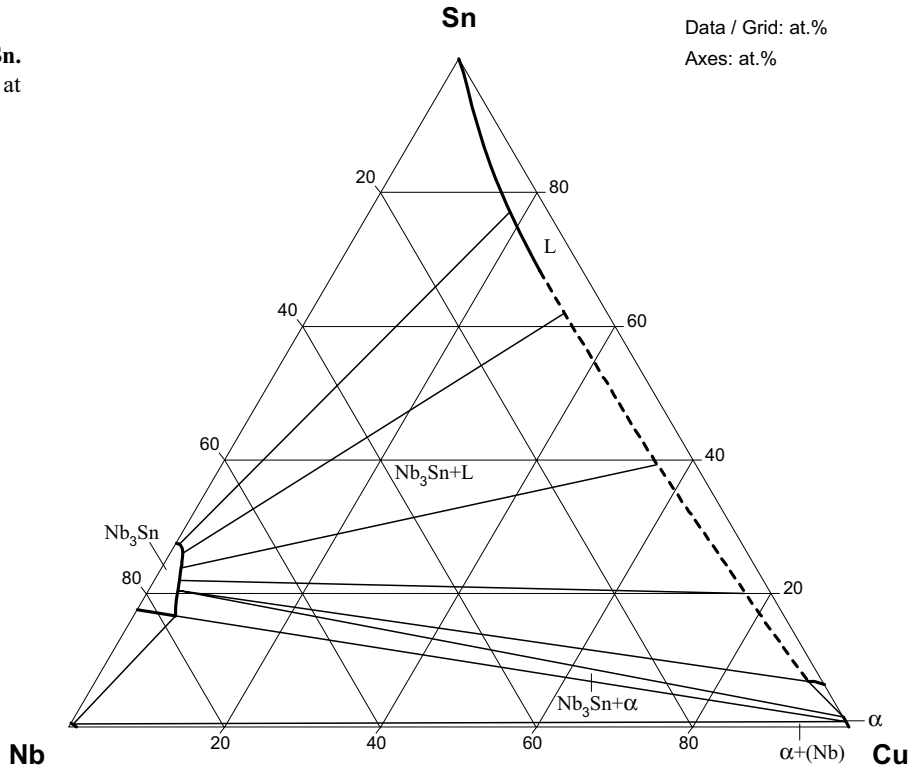

Fig. 5: Cu-Nb-Sn. Isothermal section at 1000°C

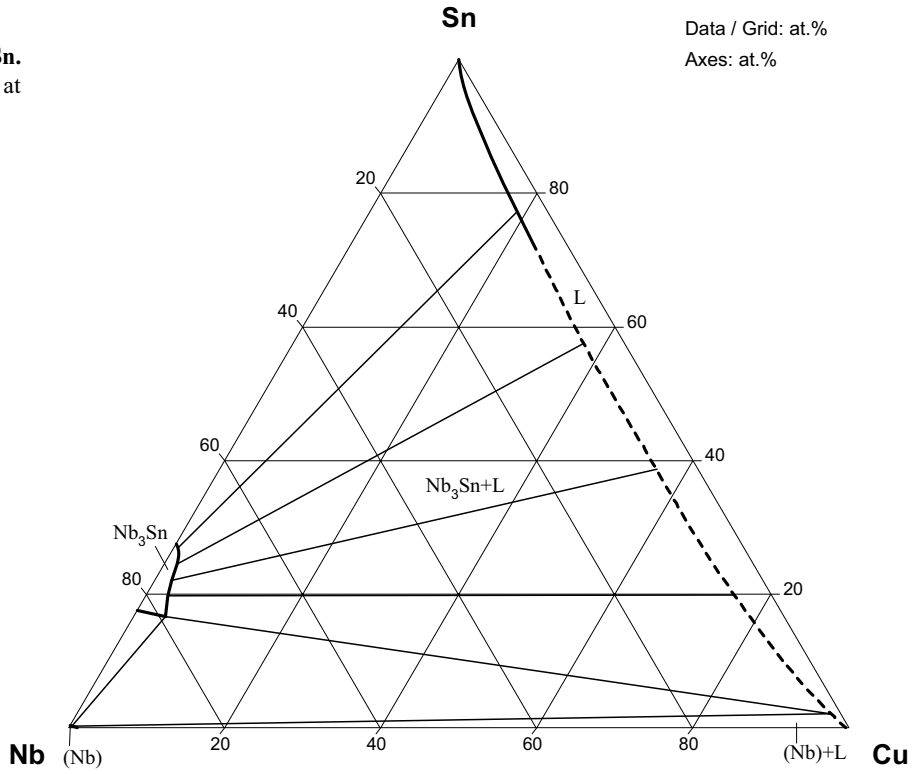

Fig. 6: Cu-Nb-Sn. Isothermal section at 1100°C