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## Phase equilibrium diagram of Y-Cu-Sb system at 870 K

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The interaction of the components in the Y-Cu-Sb ternary system was investigated using the methods of X-ray phase analysis, microstructure, and energy-dispersive X-ray spectroscopy in the whole concentration range at 870 K. At the temperature of investigation Y-Cu-Sb system is characterized by the formation of three ternary compounds: Y<sub>3</sub>Cu<sub>22</sub>Sb<sub>9</sub> (Dy<sub>3</sub>Cu<sub>20+x</sub>Sb<sub>11-x</sub> structure type, space group *F-43m*, *a*=1.6614(3) nm), Y<sub>3</sub>Cu<sub>3</sub>Sb<sub>4</sub> (Y<sub>3</sub>Au<sub>3</sub>Sb<sub>4</sub> structure type, space group *I-43d*, *a* = 0.95357(5) nm), YCuSb<sub>2</sub> (HfCuSi<sub>2</sub> structure type, space group *P4/nmm*, *a* = 0.42580(1), *c* = 0.98932(3) nm). The solubility of copper in the binary compound YSb (NaCl structure type) extends up to 8 at. %.

**Keywords:** intermetallics, ternary system, phase equilibria, crystal structure.

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## Introduction

The basis for the search for new materials is the study of the chemical interaction of elements in multicomponent systems. Rare earth metals (R=Y, La-Sm, Gd-Er) with copper and antimony form R<sub>3</sub>Cu<sub>3</sub>Sb<sub>4</sub> compounds with the Y<sub>3</sub>Au<sub>3</sub>Sb<sub>4</sub> structure type (space group *I-43d*) [1], which is a derivative of the Th<sub>3</sub>P<sub>4</sub> structure type [2]. As well as half-Heusler RNiSb phases (MgAgAs structure type, space group *F-43m*) [3, 4], R<sub>3</sub>Cu<sub>3</sub>Sb<sub>4</sub> compounds are characterized by semiconductor properties [1, 5, 6]. Unlike half-Heusler RNiSb phases, the crystal structure of R<sub>3</sub>Cu<sub>3</sub>Sb<sub>4</sub> compounds is more complex, which ensures low thermal conductivity of these phases. It is known that both the methods of synthesis and the presence of impurity phases have a significant influence on the behavior of the electrical transport properties of semiconductor compounds. Therefore, when studying the interaction of components in the corresponding systems, it is possible to obtain important information about the conditions of formation, temperature and concentration stability, microstructure, and crystal structure of the original ternary compounds, as a basis for the search for new semiconductor materials. Phase diagrams of the R-Cu-Sb

ternary systems were constructed for R=Ce, Nd, Ho, and Tm [7-9]. Separate ternary compounds were studied for other rare earth elements [7].

The purpose of this work is an experimental study of the interaction of components in the Y-Cu-Sb system and the construction of a phase equilibrium diagram at 870 K.

## I. Experimental

To construct a phase diagram of the Y-Cu-Sb system, the alloys were synthesized by the arc-melting of a charge of metals (the content of the basic component is not less than 99.9 wt.%) in an argon atmosphere (sponge titanium was used as a getter). For better homogenization, the samples were remelted twice. Losses of Sb during melting were compensated by an excess of 2-3 wt. % of antimony. Homogenizing annealing of the alloys was carried out in vacuumed quartz ampoules at a temperature of 870 K for 700 hours with subsequent quenching in cold water.

The phase composition of the synthesized samples was determined based on the analysis of experimental powder diffraction patterns (DRON-4.0 diffractometer, FeK $\alpha$  radiation) by comparing them with theoretical

diffraction patterns of pure components, binary compounds, and known ternary antimonides (PowderCell program [10]). The method of energy dispersive X-ray spectroscopy (EDRS) (electron microscope TESCAN VEGA 3 LMU, equipped with an X-ray analyzer with energy dispersive spectroscopy) was used to control the chemical composition of the synthesized samples and determine the exact content of components in the phases. At least five measurements were performed for each phase in each sample. Structural calculations were performed using WinCSD [11] and FullProf Suite [12] program packages.

The temperature limits of the stability of ternary compounds were investigated by the method of differential thermal analysis (DTA, synchronous thermal analyzer LINSEIS STA PT 1600). The samples were heated in an argon atmosphere up to 900°C (1170 K) at a rate of 10°C/min.

## II. Experimental results

To study the Y-Cu-Sb ternary system, information on phase diagrams of Y-Cu, Y-Sb, and Cu-Sb binary systems, which limit the Y-Cu-Sb system, was used from Refs. [13, 14]. At the temperature of the investigation, the formation of  $\text{Cu}_3\text{Sb}$  ( $\text{BiF}_3$  structure type) and  $\text{Cu}_2\text{Sb}$  ( $\text{Cu}_2\text{Sb}$  structure type) compounds was confirmed in the Cu-Sb system. The homogeneity range of the  $\text{Cu}_3\text{Sb}$  binary compound was determined by EDX analysis and is limited by the compositions  $\text{Cu}_{78.11}\text{Sb}_{21.89}$  and  $\text{Cu}_{67.45}\text{Sb}_{32.55}$ . Three binary compounds are formed in the Y-Sb system:  $\text{Y}_3\text{Sb}$  ( $\text{Ti}_3\text{P}$  structure type),  $\text{Y}_5\text{Sb}_3$  ( $\text{Mn}_5\text{Si}_3$  structure type), and  $\text{YSb}$  ( $\text{NaCl}$  structure type). The  $\text{Y}_4\text{Sb}_3$  compound ( $\text{Th}_3\text{P}_4$  structure type) exists at high temperatures (above 1660 K) [13], and the high-temperature modification of the  $\text{Y}_5\text{Sb}_3$  compound with the  $\text{Yb}_5\text{Sb}_3$  type structure was obtained at a temperature of 1538 K [15]. The existence of the  $\text{Y}_3\text{Sb}$ ,  $\text{Y}_5\text{Sb}_3$ , and  $\text{YSb}$  compounds in the Y-Sb system at a temperature of 870 K was confirmed by us during the study of the Y-Ni-Sb system [4]. The Y-Cu system is characterized by the formation of five binary phases:  $\text{YCu}_5$  ( $\text{CaCu}_5$  structure type),  $\text{Y}_{0.8}\text{Cu}_{5.4}$  ( $\text{Tb}_{0.78}\text{Cu}_{5.44}$  structure type),  $\text{YCu}_2$  ( $\text{KHg}_2$  structure type),  $\text{YCu}$  ( $\text{CsCl}$  structure type), and  $\text{Y}_2\text{Cu}_7$  with unknown structure. In the course of the study, the formation of binary compounds was confirmed according to literature data, except for the  $\text{Y}_2\text{Cu}_7$  phase. Under the conditions of our study, the sample of the corresponding composition contained  $\text{YCu}_5$  and  $\text{YCu}_2$  compounds in equilibrium.

The phase equilibrium diagram of the Y-Cu-Sb system was constructed at 870 K based on the data from the X-ray phase, microstructural analyses, and scanning electron microscopy of the synthesized samples (Fig. 1). Microstructural and EDX analyses were used to confirm the chemical and phase composition of the samples. The phase compositions of selected alloys of the Y-Cu-Sb system according to X-ray phase and EDX analyses are given in Table. 1. Photographs of microstructures for some samples are shown in Fig. 2. At a temperature of 870 K in the Y-Cu-Sb system, the formation of three ternary compounds was confirmed [1, 16, 17], the crystallographic characteristics of which are given in the Table. 2.

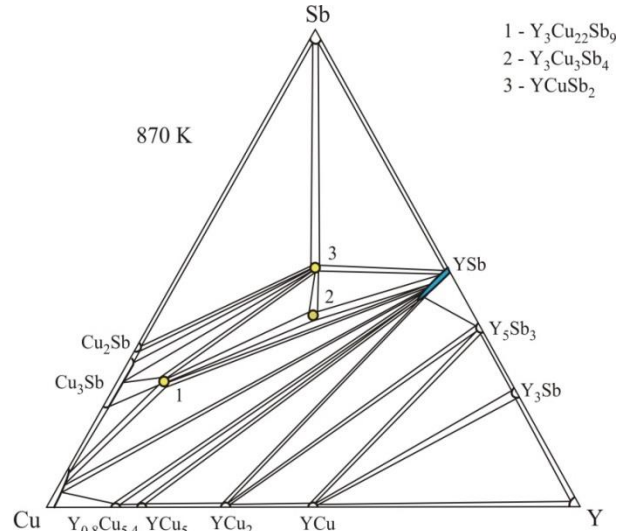


Fig. 1. Isothermal section of the Y-Cu-Sb system at 870 K.

Information concerning the compound  $\text{YCuSb}_2$  with  $\text{HfCuSi}_2$  structure type is given in Ref. [16]. The authors of Ref. [18] consider the structure of the  $\text{YCuSb}_2$  compound as defective with a structure of the  $\text{CaBe}_2\text{Ge}_2$  type. The performed calculation of the crystal structure of the compound  $\text{YCuSb}_2$  confirmed its belonging to the  $\text{HfCuSi}_2$  structure type (space group  $P4/nmm$ ,  $R_{\text{Bragg}} = 0.0387$ ,  $R_f = 0.0258$ ) (Fig. 3). The calculated atomic coordinates in the  $\text{YCuSb}_2$  structure are given in Table 3. According to the performed calculations and EDX data ( $\text{Y}_{26.25}\text{Cu}_{24.21}\text{Sb}_{49.54}$ ), the compound  $\text{YCuSb}_2$  under used conditions is realized at a stoichiometric composition.

The solubility of the third component in the binary compounds of the Cu-Sb and Y-Cu systems under the conditions of the study is insignificant (up to ~1.5 at. %). In the Y-Sb system, the binary compound  $\text{YSb}$  ( $\text{NaCl}$  structure type) dissolves up to ~8 at. % Cu, which is confirmed by the data of EDX analysis ( $\text{Y}_{46.18}\text{Cu}_{8.03}\text{Sb}_{45.79}$ ). The lattice parameters vary from  $a = 0.6184(2)$  (for  $\text{YSb}$ ) to  $a = 0.6192(2)$  nm (for the  $\text{Y}_{45}\text{Cu}_{10}\text{Sb}_{45}$  sample). Significant solubility of copper in other binary compounds of the Y-Sb system was not observed.

The  $\text{Y}_3\text{Cu}_{22}\text{Sb}_9$  and  $\text{Y}_3\text{Cu}_3\text{Sb}_4$  compounds were studied using differential thermal analysis (DTA), which confirmed the limited temperature interval of the compounds stability (Fig. 4, heating regime).

Temperature transitions (heating mode) at temperatures of 1064 K for  $\text{Y}_3\text{Cu}_{22}\text{Sb}_9$  phase (temperature transitions at 923 K corresponds to the melting temperature of the impurity phase  $\text{Cu}_3\text{Sb}$ ) and 968 K for  $\text{Y}_3\text{Cu}_3\text{Sb}_4$  phase indicate the decomposition of both compounds above the specified temperatures. There is practically no change in the mass of the tested samples during the measurement.

Table 1.

Phase composition and EDX data for selected alloys of the Y–Cu–Sb system

Nominal composition/EDX composition, at. %	Phase	Structure type	Lattice parameters, nm			EDX data, at. %		
			<i>a</i>	<i>b</i>	<i>c</i>	Y	Cu	Sb
Y <sub>40</sub> Cu <sub>40</sub> Sb <sub>20</sub>	YSb	NaCl	0.6189(3)					
	YCu <sub>2</sub>	KHg <sub>2</sub>	0.4309(4)	0.6893(3)	0.7307(4)			
Y <sub>65</sub> Cu <sub>15</sub> Sb <sub>20</sub>	YCu	CsCl	0.3472(3)					
	Y <sub>5</sub> Sb <sub>3</sub>	Mn <sub>5</sub> Si <sub>3</sub>	0.8882(3)		0.6287(3)			
	Y <sub>3</sub> Sb	Ti <sub>3</sub> P	1.2363(5)		0.6178(3)		100.0	
Y <sub>35</sub> Cu <sub>45</sub> Sb <sub>20</sub> Y <sub>37.47</sub> Cu <sub>45.50</sub> Sb <sub>17.03</sub>	YCu <sub>3</sub> Sb	NaCl	0.61891(3)			45.15	8.38	46.47
	YCu <sub>2</sub>	KHg <sub>2</sub>	0.4308(3)	0.6893(4)	0.7307(4)	34.66	64.64	0.70
	YCu <sub>5</sub>	CaCu <sub>5</sub>	0.4989(4)		0.4099(4)	16.54	83.46	
Y <sub>40</sub> Cu <sub>40</sub> Sb <sub>20</sub> Y <sub>37.47</sub> Cu <sub>45.50</sub> Sb <sub>17.03</sub>	YCu <sub>2</sub>	KHg <sub>2</sub>	0.4308(3)	0.6892(4)	0.7307(3)	34.66	64.65	0.69
	YCu <sub>3</sub> Sb	NaCl	0.6190(3)			47.68	5.02	47.30
	YCu <sub>5</sub>	CaCu <sub>5</sub>	0.4988(4)		0.4097(4)	16.44	83.56	
Y <sub>20</sub> Cu <sub>55</sub> Sb <sub>25</sub> Y <sub>21.22</sub> Cu <sub>55.28</sub> Sb <sub>23.50</sub>	YCu <sub>3</sub> Sb	NaCl	0.6190(3)			46.18	8.04	45.78
	Y <sub>3</sub> Cu <sub>22</sub> Sb <sub>9</sub>	Dy <sub>3</sub> Cu <sub>22</sub> Sb <sub>9</sub>	1.6614(7)			8.76	65.42	25.82
	(Cu)	Cu	0.3632(3)				100.0	
Y <sub>10</sub> Cu <sub>55</sub> Sb <sub>35</sub>	Y <sub>3</sub> Cu <sub>22</sub> Sb <sub>9</sub>	Dy <sub>3</sub> Cu <sub>22</sub> Sb <sub>9</sub>	1.6613(7)					
	Cu <sub>3</sub> Sb	BiF <sub>3</sub>	0.5967(3)					
	YCuSb <sub>2</sub>	HfCuSi <sub>2</sub>	0.4259(3)		0.9936(4)			
Y <sub>20</sub> Cu <sub>40</sub> Sb <sub>40</sub> Y <sub>19.93</sub> Cu <sub>41.99</sub> Sb <sub>38.08</sub>	Y <sub>3</sub> Cu <sub>22</sub> Sb <sub>9</sub>	Dy <sub>3</sub> Cu <sub>22</sub> Sb <sub>9</sub>	1.6614(5)			9.19	63.75	27.06
	Y <sub>3</sub> Cu <sub>3</sub> Sb <sub>4</sub>	Y <sub>3</sub> Au <sub>3</sub> Sb <sub>4</sub>	0.9536(3)			30.05	30.26	39.69
	YCuSb <sub>2</sub>	HfCuSi <sub>2</sub>	0.4260(2)		0.9936(4)	27.11	24.62	48.27
Y <sub>48</sub> Cu <sub>12</sub> Sb <sub>40</sub> Y <sub>47.65</sub> Cu <sub>11.25</sub> Sb <sub>41.10</sub>	YCu <sub>3</sub> Sb	NaCl	0.6189(3)			48.61	2.93	48.46
	YCu <sub>2</sub>	KHg <sub>2</sub>	0.43091(3)	0.6891(4)	0.7307(4)	33.48	65.26	1.26
Y <sub>55</sub> Cu <sub>5</sub> Sb <sub>40</sub> Y <sub>55.42</sub> Cu <sub>3.43</sub> Sb <sub>41.15</sub>	Y <sub>5</sub> Sb <sub>3</sub>	Mn <sub>5</sub> Si <sub>3</sub>	0.8881(3)		0.6288(3)	61.46	0.36	38.18
	YCu <sub>3</sub> Sb	NaCl	0.6187(3)			49.02	2.25	48.73
	YCu <sub>2</sub>	KHg <sub>2</sub>	0.4309(3)	0.6891(3)	0.7308(5)	33.57	66.05	0.38
Y <sub>15</sub> Cu <sub>35</sub> Sb <sub>50</sub>	YCuSb <sub>2</sub>	HfCuSi <sub>2</sub>	0.4260(3)		0.9941(3)			
	Cu <sub>3</sub> Sb	BiF <sub>3</sub>	0.5968(3)					
	(Sb)	As	0.4308(2)		1.1256(4)			

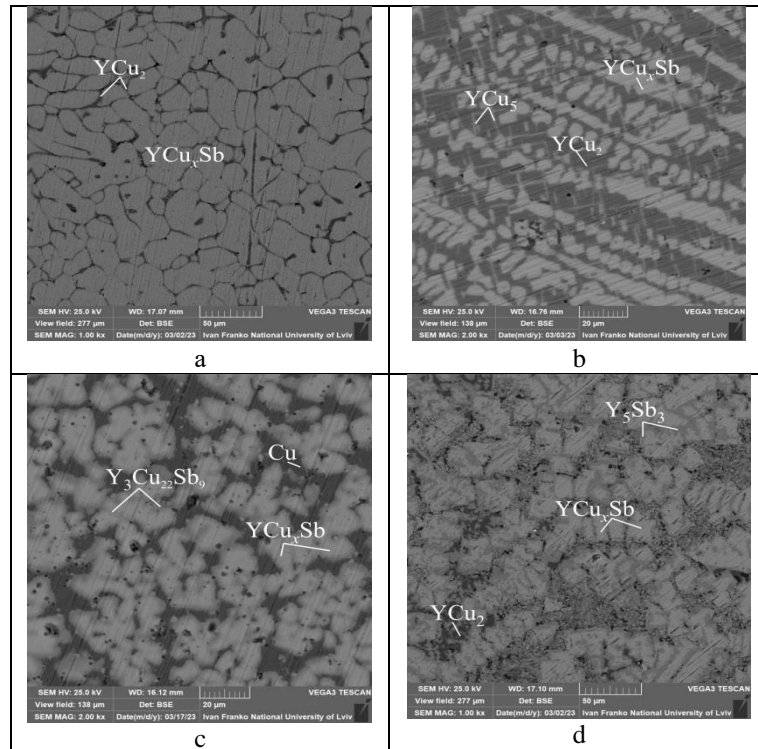
Fig. 2. Microphotographs of the alloys: Y<sub>48</sub>Cu<sub>12</sub>Sb<sub>40</sub> (a), Y<sub>35</sub>Cu<sub>45</sub>Sb<sub>20</sub> (b), Y<sub>20</sub>Cu<sub>55</sub>Sb<sub>25</sub> (c), Y<sub>50</sub>Cu<sub>20</sub>Sb<sub>30</sub> (d).

Table 2.

Crystallographic characteristics of the ternary compounds in the Y-Cu-Sb					
Compound	Structure type	Space group	L		
			<i>a</i>	<i>b</i>	<i>c</i>
Y <sub>3</sub> Cu <sub>22</sub> Sb <sub>9</sub>	Dy <sub>3</sub> Cu <sub>20+x</sub> Sb <sub>11-x</sub>	<i>F-43m</i>			
Y <sub>3</sub> Cu <sub>3</sub> Sb <sub>4</sub>	Y <sub>3</sub> Au <sub>3</sub> Sb <sub>4</sub>	<i>I-43d</i>			
YCuSb <sub>2</sub>	HfCuSi <sub>2</sub>	<i>P4/nmm</i>	8		8

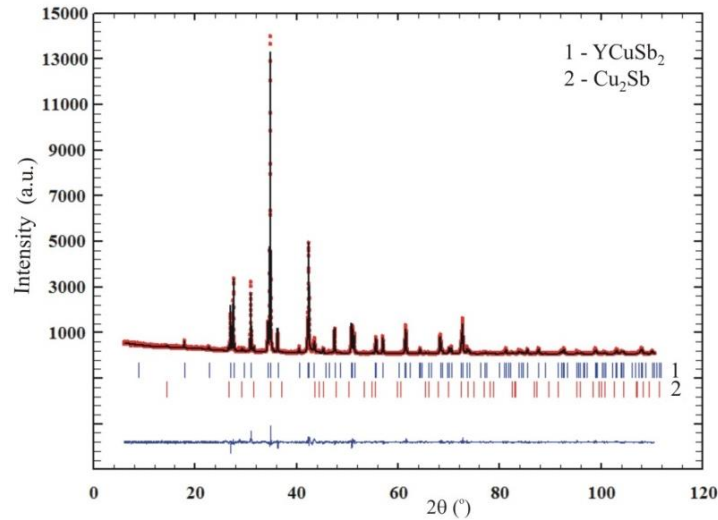
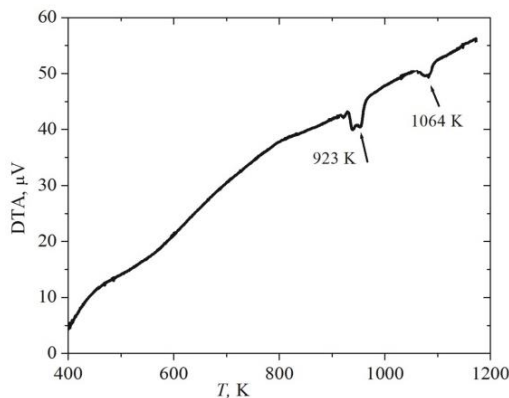


Fig. 3. The experimental (circles), calculated (line) and difference (bottom) X-ray diffraction patterns for Y<sub>25</sub>Cu<sub>25</sub>Sb<sub>50</sub> sample.

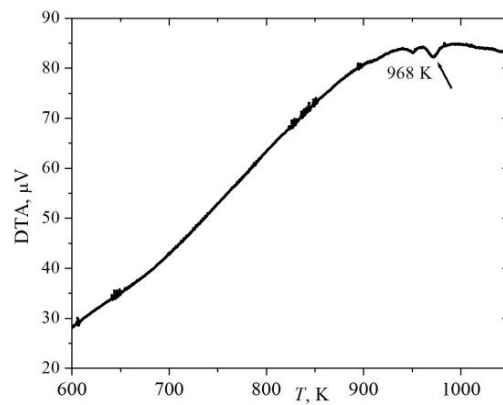
Table 3.

Fractional atomic coordinates and isotropic displacement parameters  $B_{iso}$  for YCuSb<sub>2</sub> compound

Atom	Wyckoff position	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	$B_{iso} \cdot 10^2$ (nm <sup>2</sup> )
Y	2 <i>c</i>	1/4	1/4	0.2462(2)	0.83(3)
Cu	2 <i>b</i>	3/4	1/4	1/2	0.92(1)
Sb1	2 <i>c</i>	1/4	1/4	0.6626(1)	0.41(3)
Sb2	2 <i>a</i>	3/4	1/4	0	0.52(4)



a



b

F  
i

The analysis of the investigated system Y-Cu-Sb, literature data of the studied R-Cu-Sb systems (R = Ce, Nd, Ho, Tm), and selected ternary compounds shows that antimonides RCuSb<sub>2</sub> (HfCuSi<sub>2</sub>-type) are formed with all rare earth metals, instead, compounds R<sub>3</sub>Cu<sub>3</sub>Sb<sub>4</sub> (Y<sub>3</sub>Au<sub>3</sub>Sb<sub>4</sub>-type) with Tm, Yb, and Lu are not realized.

For R=Tm, Lu, the formation of RCu<sub>3</sub>Sb<sub>2</sub> compounds with TmCu<sub>3</sub>Sb<sub>2</sub> structure type was observed. A feature of R-Cu-Sb systems is the absence of compounds with equiatomic composition, except RCuSb compounds where R = Eu and Yb with ZrBeSi structure type [19, 20].

## Conclusions

The phase equilibrium diagram of the ternary system Y–Cu–Sb was constructed at a temperature of 870 K in the whole concentration range based on the results of an experimental study of the interaction of components. The investigated system at 870 K is characterized by the formation of three ternary compounds:  $Y_3Cu_{22}Sb_9$ ,  $Y_3Cu_3Sb_4$ , and  $YCuSb_2$ . A solid solution is formed on the basis of the binary compound  $YSb$  (NaCl-type) up to a content of 8 at. % of copper. Differential thermal analysis indicated that compounds  $Y_3Cu_{22}Sb_9$  and  $Y_3Cu_3Sb_4$  are stable up to temperatures of 1064 K and 968 K, respectively.

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## Діаграма фазових рівноваг системи Y-Cu-Sb при 870 К

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Взаємодія компонентів у потрійній системі Y-Cu-Sb досліджена методами рентгенівського, мікроструктурного аналізу та енергодисперсійної рентгенівської спектроскопії в повному концентраційному інтервалі за температури 870 К. За умов дослідження система характеризується існуванням трьох тернарних сполук:  $Y_3Cu_{22}Sb_9$  (структурний тип  $Du_3Cu_{20+x}Sb_{11-x}$ , просторова група  $F-43m$ , (структурний тип  $HfCuSi_2$ , просторова група  $P4/nmm$ ,  $a = 0.42580(1)$ ,  $c = 0.98932(3)$  нм). Розчинність купруму в бінарній сполуці YSb (структурний тип NaCl) сягає 8 ат. %.

**Ключові слова:** інтерметаліди, потрійна система, фазові рівноваги, кристалічна структура.