

The crystal structure of the new ternary antimonide $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$)

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Abstract

New ternary antimonide $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$) was synthesized and its crystal structure was determined by direct methods from X-ray powder diffraction data (diffractometer DRON-3M, $\text{CuK}\alpha$ -radiation, $R_1 = 6.99\%$, $R_p = 12.27\%$, $R_{wp} = 11.55\%$). The compound crystallizes with the own cubic structure type: space group $F43m$, Pearson code $cF272$, $a = 16.6150(2)$ Å, $Z = 8$. The structure of the $\text{Dy}_3\text{Cu}_{20}\text{Sb}_{11-x}$ ($x \approx 2$) can be obtained from the structure type BaHg_{11} by doubling of the lattice parameter and subtraction of 16 atoms. The studied structure was compared with the structures of known compounds, which crystallize in the same space group with similar cell parameters.

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1. Introduction

The existence of the ternary compounds of approximate composition $\sim R\text{Cu}_6\text{Sb}_3$ ($R = \text{La}, \text{Ce}, \text{Pr}$) with unknown structures were reported in Refs. [1,2]. During the systematic investigation of the phase equilibria in the system Dy–Cu–Sb we synthesized a new ternary compound at higher copper content. The crystal structure of this new ternary antimonide $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$) was solved by direct methods from X-ray powder diffraction data. By the composition (low rare earth metal content) and symmetry (cubic syngony) this compound is similar to the known ternary compounds with the high content of transition metals in the systems R – T –{Si, Ge} (R —rare earth metals, T —transition metal) [3–5]. Such ternaries are of interest because of variety of their physical properties [6–8].

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The results of the crystal structure determination of the $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$) are presented in this article.

2. Experimental details

The sample of nominal composition $\text{Dy}_9\text{Cu}_{64}\text{Sb}_{27}$ was prepared by arc melting of the constituent metals (dysprosium, 99.83 wt%; copper, 99.99 wt%; antimony, 99.99 wt%) in a water-cooled copper bottom under a purified argon atmosphere with Ti as a getter. To ensure homogeneity the sample was remelted twice. Then the ingot was wrapped into tantalum foil, annealed at 870 K in quartz ampoule under vacuum during 720 h and subsequently quenched in cold water. During the sample preparation the weight losses were less than 1% of the total mass, which was about 1 g.

The crystal structure of the ternary compound was determined and refined using the X-ray powder diffraction data obtained on DRON-3M diffractometer ($\text{CuK}\alpha$ -radiation). The data were collected with a scanning step of 0.02° and collecting time of 15 s in a

2θ range of 16–120°. All the procedures, including indexing, refinement of the profile and structural parameters and calculations of the interatomic distances were performed with the *WinCSD* program package [9]. The atomic positions were standardized with the *STRUCTURE TIDY* program [10].

The automatic indexing of the powder pattern of $\text{Dy}_9\text{Cu}_{64}\text{Sb}_{27}$ indicated a cubic cell with the lattice parameter: $a = 16.6150(2)\text{Å}$. Analysis of the systematic extinctions of 216 possible reflections in the measured 2θ range ($hkl: h+k, h+l, k+l = 2n+1; 0kl: k, l = 2n+1, hhl: h+l = 2n+1, h00: h = 2n+1$) leads to five possible space groups $F23$, $F432$, $F3m$, $F\bar{4}3m$ and $Fm\bar{3}m$. The crystal structure was solved by direct methods in the space group $F\bar{4}3m$. The refinement of the structural and profile parameters were performed by the Rietveld method ($R_1 = 6.99\%$, $R_p = 12.27\%$, $R_{wp} = 11.55\%$). In the final refinement cycles 32 parameters were allowed to vary: one scale factor, one cell parameter, six profile parameters (Pseudo-Voigt

peak shape function), twelve positional parameters, ten isotropic thermal displacement parameters and two occupancy factors for Sb3 and Sb5. Attempts to decrease the symmetry did not improve the refinement. Observed, calculated and differential X-ray powder diffraction patterns of the $\text{Dy}_9\text{Cu}_{64}\text{Sb}_{27}$ sample are shown in Fig. 1.

Electron microprobe analysis of the sample carried out using CAMEBAX plant at the probe current 0.01 μA and accelerating voltage 20 kV. For the analysis analytical lines $K\alpha$ (Cu) and $L\alpha$ (Dy and Sb) were used. Pure metals were used as standards.

3. Results and discussion

Atomic coordinates, isotropic temperature displacement and occupational parameters in the structure of $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$) are listed in Table 1. Two out of 10 atomic positions are occupied by

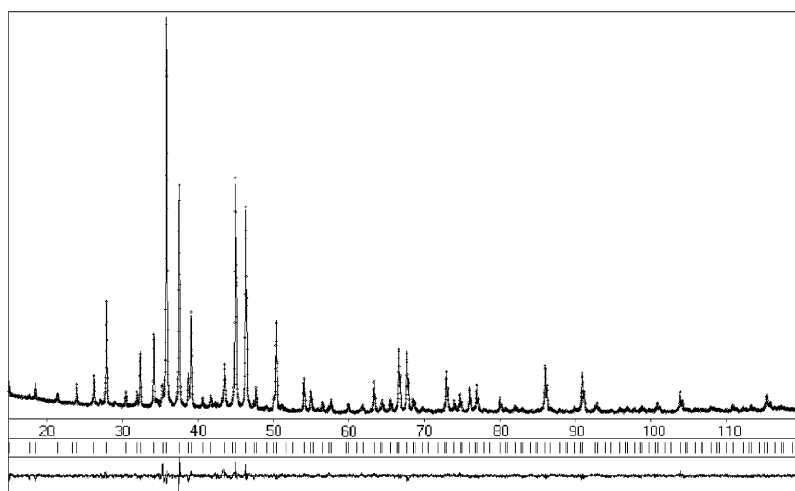


Fig. 1. Experimental (points), calculated (continuous lines) and difference between experimental and calculated (continuous line on bottom) X-ray powder diffraction patterns of $\text{Dy}_9\text{Cu}_{64}\text{Sb}_{27}$ sample. Vertical bars indicate the positions of hkl reflections for $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ phase.

Table 1

Atomic coordinates, isotropic thermal displacement parameters and occupancies of $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$, space group $F\bar{4}3m$, Pearson code $cF272$, $a = 16.6150(2)\text{Å}$)

Atom	Site	x/a	y/b	z/c	$B_{\text{iso}} \times 10^2 (\text{Å}^2)$
Dy	24g	0.0016(2)	1/4	1/4	0.92(2)
Cu1	16e	0.8199(2)	x	x	1.17(13)
Cu2	48h	0.1161(3)	x	0.4995(3)	1.53(12)
Cu3	48h	0.1142(3)	x	0.0054 (3)	0.74(11)
Cu4	48h	0.0759(1)	x	0.2462(5)	0.78(6)
Sb1	4a	0	0	0	0.7(2)
Sb2	4b	1/2	1/2	1/2	1.1(2)
Sb3 ^a	16e	0.3368(2)	x	X	0.9(2)
Sb4	48h	0.0816(1)	x	0.7493(3)	1.21(3)
Sb5 ^b	16e	0.1659(3)	x	X	1.0(2)

^aSb3 = 0.58(2)Sb + 0.42(2)Cu.

^bSb5 = 0.51(2)Sb + 0.49(2)Cu.

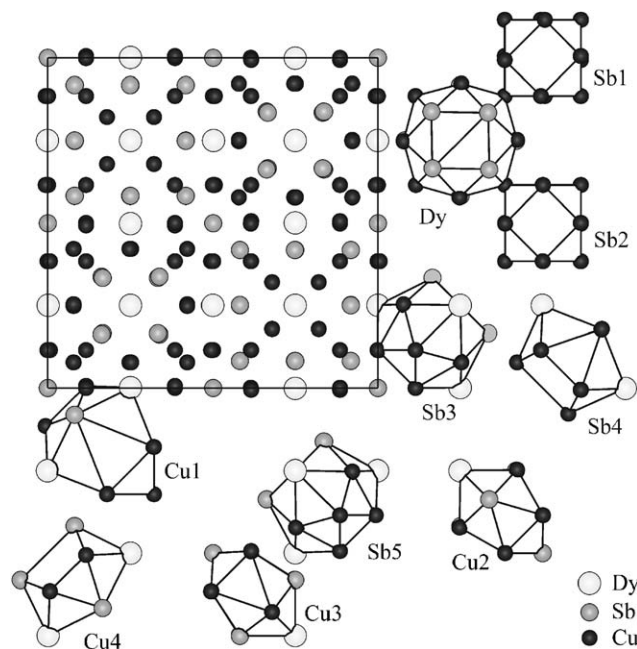


Fig. 2. Projection of the unit cell of the $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$) and coordination polyhedra of atoms on xy plane.

statistical mixtures — $16e$ ($0.58(2)\text{Sb} + 0.42(2)\text{Cu}$) and $16e$ ($0.51(2)\text{Sb} + 0.49(2)\text{Cu}$), respectively. The composition found during the electron microprobe analysis ($\text{Dy}_{3.0}\text{Cu}_{22.0}\text{Sb}_{9.0}$) correlate well with the composition calculated during the structure refinement ($\text{Dy}_3\text{Cu}_{21.8}\text{Sb}_{9.2}$). The projection of the unit cell and coordination polyhedra of atoms in the structure of $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$) along four-fold axis are shown in Fig. 2. Interatomic distances (δ), coordination numbers for all atoms (CN) and differences between the calculated distances and the sums of atomic radii of elements (Δ) are presented in Table 2. The differences between the calculated distances and the sums of the Dy or Cu atomic radii and the ionic radius of Sb^{3+} (Δ') are also listed for comparison. All interatomic distances correlate with the sums of atomic radii of components ($r_{\text{Dy}} = 1.80 \text{ \AA}$, $r_{\text{Cu}} = 1.28 \text{ \AA}$ and $r_{\text{Sb}} = 1.61 \text{ \AA}$ [11]). The most shortening was observed between Cu4–Sb5 (13.49%), Cu4–Sb3 (11.94%) and Sb3–Sb5 (11.80%). That can be explained by partially covalent bonds, and partially valent state of the Sb atoms +3. According to the classification of the structure types of

Table 2

Interatomic distances (δ) within the coordination polyhedra, coordination numbers of atoms (CN), differences between δ and sums of the atomic radii of elements (Δ) in the structure of $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$) and between δ and the sums of the Dy or Cu atomic radii and the ionic radius of Sb^{3+} (Δ')

Atom	δ (Å)	CN	Δ (%)	Δ' (%)	Atomii	δ (Å)	CN	Δ (%)	Δ' (%)
Dy–4Sb4	3.121(2)		–7.66	16.89	Cu1–3Sb4	2.595(4)	12	–10.21	19.04
Dy–4Cu4	3.146(2)		3.15		Cu1–3Cu1	3.287(5)		28.40	
Dy–2Cu2	3.147(4)		3.18		Cu1–3Dy	3.436(4)		12.66	
Dy–2Cu3	3.191(4)	18	4.62		Cu1–3Cu3	3.448(6)		34.69	
Dy–2Sb5	3.370(5)		–0.30	26.22					
Dy–2Sb3	3.372(4)		–0.24	26.29	Cu2–2Sb4	2.663(7)	11	–7.86	22.16
Dy–2Cu1	3.436(4)		12.66		Cu2–2Cu4	2.693(9)		5.20	
					Cu2–2Cu2	2.716(7)		6.09	
Sb1–12Cu3	2.686(4)	12	–7.06	23.21	Cu2–1Sb2	2.728(4)		–5.61	25.14
					Cu2–2Cu2	2.740(7)		7.03	
Sb2–12Cu2	2.728(4)	12	–5.61	25.14	Cu2–1Sb3	2.921(6)		1.07	33.99
					Cu2–1Dy	3.147(4)		3.18	
Sb3 ^b –3Cu4	2.545(6)		11.94	16.74					
Sb3–3Sb5	2.840(6)	12	–11.80	57.77	Cu3–2Cu3	2.557(6)	12	–0.12	
Sb3–3Cu2	2.921(6)		1.07	33.99	Cu3–2Cu4	2.565(9)		0.20	
Sb3–3Dy	3.372(4)		–0.24	26.29	Cu3–1Sb1	2.686(4)		–7.06	23.21
					Cu3–2Sb4	2.743(7)		–5.09	25.83
Sb4–1Cu1	2.595(4)	9	–10.21	19.04	Cu3–2Cu3	2.811(6)		9.81	
Sb4–2Cu4	2.619(2)		–9.38	20.14	Cu3–1Sb5	2.930(7)		1.38	34.40
Sb4–2Cu2	2.663(7)		–7.86	22.16	Cu3–1Dy	3.191(4)		4.62	
Sb4–2Cu3	2.743(7)		–5.09	25.83	Cu3–1Cu1	3.448(5)		34.69	
Sb4–2Dy	3.121(2)		–7.66	16.89					
Sb5 ^c –3Cu4	2.500(7)	12	–13.49	14.68	Cu4–1Sb5	2.500(7)	10	–13.49	14.68
Sb5–3Sb3	2.840(6)		–11.80	57.77	Cu4–1Sb3	2.545(6)		–11.94	16.74
Sb5–3Cu3	2.930(7)		1.38	34.40	Cu4–2Cu3	2.565(9)		0.20	
Sb5–3Dy	3.370(5)		–0.30	26.22	Cu4–2Sb4	2.619(2)		–9.38	20.14
					Cu4–2Cu2	2.693(9)		5.20	
					Cu4–2Dy	3.146(2)		3.15	

¹ $\Delta = (\delta - \sum r) / \sum r \cdot 100\%$; $r(\text{Dy}) = 1.77 \text{ \AA}$, $r(\text{Cu}) = 1.28 \text{ \AA}$, $r(\text{Sb}) = 1.61 \text{ \AA}$; Δ' : $r(\text{Sb}^{3+}) = 0.90 \text{ \AA}$.

^bSb3 = $0.58(2)\text{Sb} + 0.42(2)\text{Cu}$.

^cSb5 = $0.51(2)\text{Sb} + 0.49(2)\text{Cu}$.

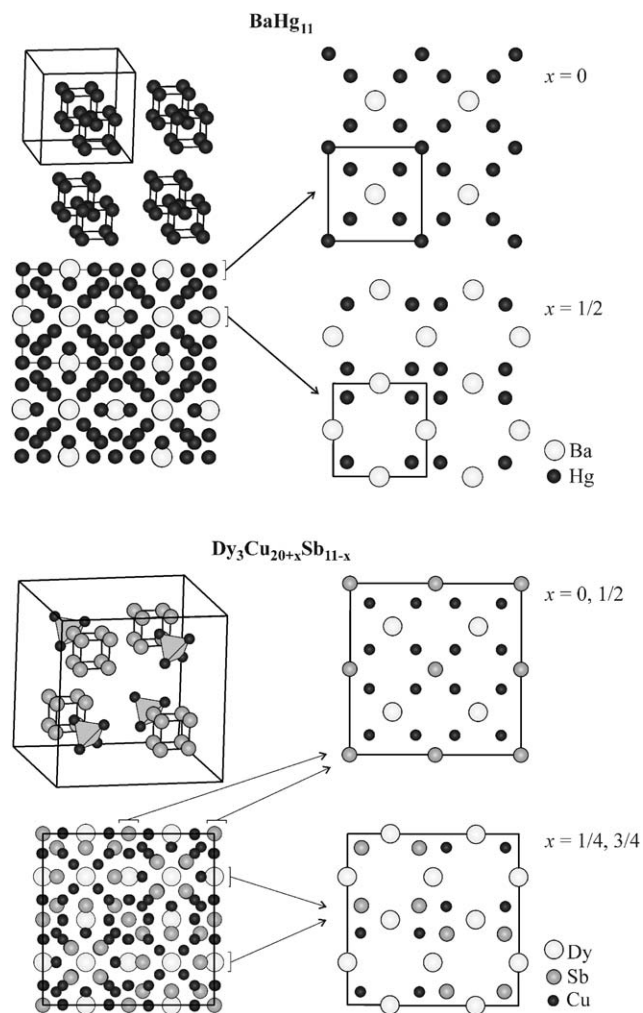


Fig. 3. Atoms distribution in the plain nets perpendicular to the four-fold axes and empty cubes and tetrahedra in the structures of BaHg_{11} and $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$.

intermetallic compounds, proposed by Kripyakevich [12], structure of $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$) belongs to the classes with an icosahedral and cubooctahedral coordination of the smaller atoms. Dy atoms are surrounded by polyhedrons consisting of 18 atoms $[\text{Dy}(\text{Sb}4)_4(\text{Cu}4)_4(\text{Cu}2)_2(\text{Cu}3)_2(\text{Sb}5)_2(\text{Sb}3)_2(\text{Cu}1)_2]$. The coordination polyhedra of Sb1 and Sb2 atoms are cubooctahedra ($CN = 12$) formed by Cu atoms— $[\text{Sb}1(\text{Cu}3)_{12}]$ and $[\text{Sb}2(\text{Cu}2)_{12}]$, respectively; Sb3 and Sb5—icosahedra $[\text{Sb}3(\text{Cu}4)_3(\text{Sb}5)_3(\text{Cu}2)_3(\text{Dy})_3]$ and $[\text{Sb}5(\text{Cu}4)_3(\text{Sb}3)_3(\text{Cu}3)_3(\text{Dy})_3]$, respectively; Sb4—polyhedra consisting 9 atoms $[\text{Sb}4\text{Cu}1(\text{Cu}4)_2(\text{Cu}2)_2(\text{Cu}3)_2(\text{Dy})_2]$. The closest atomic environment of Copper atoms Cu1, Cu2 and Cu3 are distorted icosahedra $[\text{Cu}1(\text{Sb}4)_3(\text{Cu}1)_3(\text{Dy})_3(\text{Cu}3)_3]$, $[\text{Cu}2(\text{Sb}4)_2(\text{Cu}4)_2(\text{Cu}2)_2\text{Sb}2(\text{Cu}2)_2\text{Sb}3\text{Dy}]$ and $[\text{Cu}3(\text{Cu}3)_2(\text{Cu}4)_2\text{Sb}1(\text{Sb}4)_2(\text{Cu}3)_2\text{Sb}5\text{DyCu}1]$, respectively. Coordination polyhedra of Cu4 atoms $[\text{Cu}4\text{Sb}5\text{Sb}3(\text{Cu}3)_2(\text{Sb}4)_2(\text{Cu}2)_2(\text{Dy})_2\text{Cu}4]$ consist 10 atoms and can be obtained from

icosahedra by subtraction of two atoms and small distortion. The coordination polyhedra of some atoms in the structure of $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$) are similar to the polyhedra of atoms in the BaHg_{11} type of structure [13].

The structure of $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ ($x \approx 2$) is derivative from the known structure type BaHg_{11} by composition as well as by symmetry and lattice parameters. The investigated structure can be obtained from the BaHg_{11} by doubling of the lattice parameter and subtraction of 16 atoms. In both structures atoms forms two types of similar plain nets perpendicular to the four-fold axes (Fig. 3): the first one at $x = 0$ (in BaHg_{11}) and $x = 0, 1/2$ (in $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$) and the second—at $x = 1/2$ (in BaHg_{11}) and $x = 1/4, 3/4$ (in $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$). Hg atoms which are located above and below the layers at $x = 1/2$ in the BaHg_{11} structure form empty cubes. In the structure of $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ half of the similar cubes formed by Sb3 and Sb5 are replaced by empty tetrahedra $[(\text{Cu}1)_4]$. Hg positions in the structure of BaHg_{11} are distributed between the Cu and Sb atoms or their statistical mixtures in the $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ -type.

Another derivative from the structure type BaHg_{11} is $\text{Ce}_3\text{Pt}_{23}\text{Si}_{11}$ [5]. It can be obtained from the BaHg_{11} by inclusion of the atoms of transition metals. There are more differences between the above-mentioned structure types than between structures of BaHg_{11} and

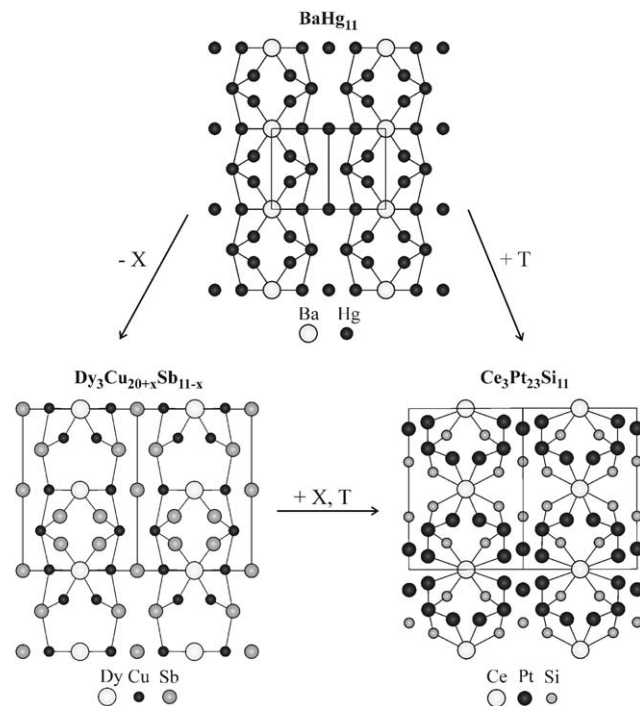


Fig. 4. Atoms arrangements in the plains nets perpendicular to the two-fold axes in the structures of BaHg_{11} , $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ and $\text{Ce}_3\text{Pt}_{23}\text{Si}_{11}$.

Table 3

Atomic coordinates in the structures with space group $F\bar{4}3m$ and lattice parameters $\sim 16 \text{ \AA}$

Site	xyz	$\text{Tl}_4\text{Ag}_{18}\text{Te}_{11}$ [14] $a = 18.717 \text{ \AA}$	$\text{Ce}_3\text{Pt}_{23}\text{Ge}_{11}$ [3] $a = 17.1833 \text{ \AA}$	$\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ $a = 16.6150 \text{ \AA}$	TmNi_2 [15] $a = 14.190 \text{ \AA}$
48 <i>h</i>	xxz	Ag $x = 0.1219$ $z = 0.9908$	Pt $x = 0.1659$ $z = 0.9979$	Cu $x = 0.1142$ $z = 0.0054$	
48 <i>h</i>	xxz	Te $x = 0.0915$ $z = 0.7456$		Sb $x = 0.0816$ $z = 0.7493$	Ni $x = 0.0708$ $z = 0.3169$
48 <i>h</i>	xxz	Ag $x = 0.1139$ $z = 0.5058$	0.78Pt + 0.22Ge $x = 0.1653$ $z = 0.5041$	Cu $x = 0.1161$ $z = 0.4995$	
48 <i>h</i>	xxz	Ag $x = 0.0581$ $z = 0.2450$		Cu $x = 0.0759$ $z = 0.2462$	Ni $x = 0.0646$ $z = 0.8181$
24 <i>g</i>	$x1/41/4$	Tl $x = 0.0195$	Ge $x = 0.0785$	Dy $x = 0.001$	Tm $x = 0.0128$
24 <i>g</i>	$x1/41/4$		Ce $x = 0.2482$		
24 <i>g</i>	$x1/41/4$	Tl (Occ. = 0.167) $x = 0.6710$	Pt $x = 0.6219$		
16 <i>e</i>	xxx		Ge $x = 0.0857$		Tm $x = 0.1214$
16 <i>e</i>	xxx		Pt $x = 0.1639$	0.51Sb + 0.49Cu $x = 0.1659$	
16 <i>e</i>	xxx	Te $x = 0.3570$	Pt $x = 0.3287$	0.58Sb + 0.42Cu $x = 0.3368$	Ni $x = 0.3112$
16 <i>e</i>	xxx		Ge $x = 0.4172$		
16 <i>e</i>	xxx		Pt $x = 0.5598$		
16 <i>e</i>	xxx		0.20Pt + 0.80Ge $x = 0.6462$		Tm $x = 0.6256$
16 <i>e</i>	xxx	Te $x = 0.1396$	Ge $x = 0.8575$	Cu $x = 0.8199$	Ni $x = 0.8154$
16 <i>e</i>	xxx		Pt $x = 0.9427$		
4 <i>c</i>	$1/41/41/4$	Tl			
4 <i>b</i>	$1/21/21/2$	Te		Sb	Tm
4 <i>a</i>	000	Te		Sb	Tm

$\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$. They differ by the atoms distributions in the layers allocated on the diagonals of the cube lattices (Fig. 4). The hexagons of similar shape form plane ribbons in the structures of BaHg_{11} and $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$. The similar fragments in the $\text{Ce}_3\text{Pt}_{23}\text{Si}_{11}$ structure type are flattened and connected trough one corner forming chains.

The most related to the investigated structure $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ (Wyckoff sequence h^4ge^3ba) are structure types with the same space group ($F\bar{4}3m$) and similar lattice parameters: $\text{Tl}_4\text{Ag}_{18}\text{Te}_{11}$ ($h^4g^2e^2cba$) [14], $\text{Ce}_3\text{Pt}_{23}\text{Ge}_{11}$ ($h^2g^3e^8$) [3] and TmNi_2 (h^2ge^4ba) [15] (Table 3). In the structures of $\text{Tl}_4\text{Ag}_{18}\text{Te}_{11}$ and $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ atoms of transition metals and *p*-elements occupy four Wyckoff positions 48*h*. Positions 24*g* are occupied by one atom of dysprosium or two atoms of thallium, in the $\text{Tl}_4\text{Ag}_{18}\text{Te}_{11}$ one of these sites is populated only on 16.7%. In both structure types,

$\text{Tl}_4\text{Ag}_{18}\text{Te}_{11}$ and $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$, positions 4*a* and 4*b* are populated by atoms of *p*-elements. In the structure of $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ three sites 16*e* are occupied by Cu and Sb atoms, in the structure of $\text{Tl}_4\text{Ag}_{18}\text{Te}_{11}$ only two of such positions are populated by Te atoms. Another Te atom is situated in 4*c* site. The atoms distributions on Wyckoff positions in the structures of $\text{Ce}_3\text{Pt}_{23}\text{Ge}_{11}$ and TmNi_2 are more complicated. The structure type TmNi_2 differ from other above-mentioned structures by number of atoms in the unit cell (192) while the numbers of atoms in the $\text{Ce}_3\text{Pt}_{23}\text{Ge}_{11}$, $\text{Tl}_4\text{Ag}_{18}\text{Te}_{11}$ and $\text{Dy}_3\text{Cu}_{20+x}\text{Sb}_{11-x}$ are 296, 284 and 272, respectively.

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