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Journal of Solid State Chemistry 174 (2003) 125–131

JOURNAL OF
SOLID STATE
CHEMISTRY

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A single crystal X-ray and HRTEM study of new series of compounds DyCu_x ($x = 4.5, 4$ and 3.5)

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Received 11 December 2002; received in revised form 21 March 2003; accepted 31 March 2003

Abstract

A series of monoclinic compounds DyCu_x ($x = 4.5, 4$ and 3.5) is described. It is constructed from structural blocks AB_5 (cubic AuBe₅ structure type) and AB_2 (cubic MgCu₂ structure type) by stacking $nAB_5 + AB_2$ and giving the compositions $A_2B_7, AB_4, A_4B_{17}, A_5B_{22}, A_6B_{27}, \dots, AB_5$. The resulting monoclinic superstructures can be derived from the cubic AuBe₅ type by introducing planar defects parallel to $\{hhh\}$ that lead to a nearly orthogonal $\approx(n+2) \times (n+2) \times (n+2-0.5)$ supercells. The present series is analogous to the monoclinic–hexagonal–trigonal–orthorhombic series $A_{n+1}B_{5n+2}$ obtained by the stacking $(n-1)AB_5 + A_2B_7$ where AB_5 is of the hexagonal CaCu₅ structure type and A_2B_7 is of the monoclinic Zr₂Ni₇ structure type.
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Keywords: Intermetallic compound; Crystal structure; DyCu_x; X-ray diffraction; HRTEM

1. Introduction

Binary phase diagrams in systems A –Cu (A =rare earth) are not very well understood around the composition 16–22 at% of A [1]. In some systems the phases with the composition A_2Cu_9 ($=ACu_{4.5}$) were reported: Gd–Cu [2], Dy–Cu [3] and Yb–Cu [4]. Structural model of YbCu_{4.5} was elaborated in Ref. [5]. Phases with other compositions were reported: ScCu₄ [6,7], A_2Cu_7 ($=ACu_{3.5}$) in the systems Nd–Cu, Gd–Cu, Dy–Cu and Yb–Cu [1]. However, no structural models were given. We will show here that using the structural model of YbCu_{4.5} it is possible to construct series of compounds AB_{5-x} ($0.5 \leq x \leq 1.5$) derived from the AuBe₅ structure type by the same building principle as in YbCu_{4.5}. As a model system for a crystallographic investigation of these series we have chosen the system Dy–Cu, because of easier synthesis conditions compared to the system Yb–Cu.

1.1. Structural model of YbCu_{4.5}

It was shown [5] that YbCu_{4.5} crystallize with a monoclinic superstructure derived from the cubic AuBe₅ type (with the lattice parameter $a \approx 7 \text{ \AA}$) by introducing planar defects parallel to $\{hhh\}$ which lead to a nearly orthogonal $\approx 7 \times 7 \times 6.5$ supercell having the cell parameters $a^s = 48.961(20)$, $b^s = 48.994(4)$, $c^s = 45.643(4) \text{ \AA}$, $\beta = 91.24(1)^\circ$. A 3-dimensional model of the superstructure was constructed in space group $C2$. Ordering of the planar defects in a $7 \times 7 \times 7$ supercell of the AuBe₅ structure type is schematically shown in Fig. 1. The planar defects are parallel to $\{hhh\}$. Half of the planar defects are copper deficient, which explains the deviation of the composition from the stoichiometry 1:5. The ordering of the planar defects as shown in Fig. 1 creates the superstructure. As described in Ref. [5] and as it can be seen in the Fig. 1 the planar defects delimit 3 regions (marked as I, I' and II) composed from blocks of the structure type AuBe₅. The block II, which is an orientation twin domain of blocks I and I', is shifted with respect to the block I in the direction $-a^s - b^s$ and the block I' with respect to the block II in the direction $-a^s + b^s$. Both shifts have the same length and lead to the monoclinic distortion. The decrease of the multiplicity of the

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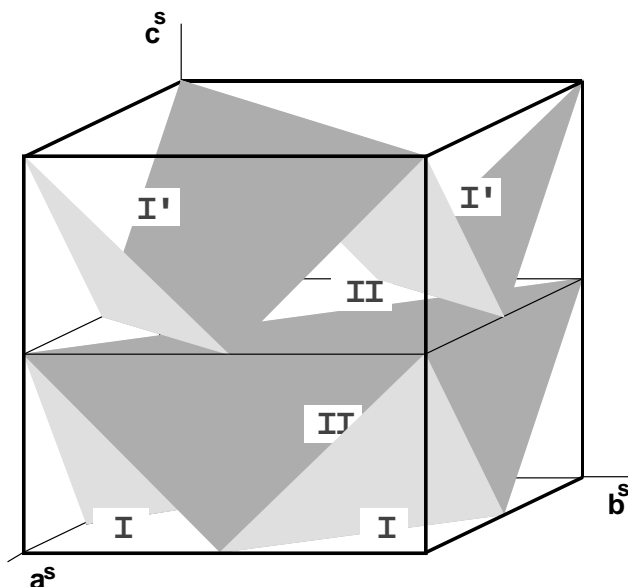


Fig. 1. Ordering of planar defects in $\text{YbCu}_{4.5}$ leading to a sevenfold monoclinic supercell of the cubic AuBe_5 -type subcell along all crystallographic directions [5]. Structural blocks marked as I, II and I' are of the AuBe_5 -type structure.

superstructure along the c -axis from 7 to about 6.5 is due to a third shift along that direction applied successively to individual blocks. The planar defects separate two mutually shifted orientation twin domains. Two types of the planar defects exist according to the apparent importance of either the twinning or the shear, and they occur respectively on (hhh) planes making mutually the angle of 70.53° in a cubic cell. We will further call them twin plane (erroneously called anti-phase boundary in Ref. [5]) and shear plane. In the projection of the structure along the direction $a^s + b^s$ the boundaries delimit triangles which are visible only in the upper part of the cell. In the lower part of the cell the projection is a superposition of two shifted blocks (I and II) of the AuBe_5 structure type. This structural model was confirmed by high resolution transmission electron microscopy (HRTEM).

2. Experimental

Several bulk samples of different composition DyCu_x were synthesized in an arc furnace ($x = 4$ and 4.45) or in an induction furnace ($x = 3.5$ and 4.5) from pure elements Dy (3N) and Cu (5N). Some alloys were investigated as cast ($x = 4.45$) whereas other were annealed in a sealed quartz ampoule under argon ($x = 4$; 14 days at 800°C , and $x = 3.5$ and 4.5 ; 7 days at 900°C). Several single crystals were extracted from the bulk samples. Attempts were made to synthesize a compound with the stoichiometric composition DyCu_5 , however, without any success to obtain the single crystal

with a sufficient quality for the analysis by X-ray diffraction.

The single crystals were all of the plate-like form, more or less rectangular. They have been examined by X-ray diffraction using an image plate diffractometer (Stoe IPDS, $\text{MoK}\alpha$). The extracted integrated intensities were corrected for absorption using indexed faces and the crystal size.

The sample preparation for the HRTEM analysis proved to be very difficult due to two main reasons. First, the crystals were very small ($\sim 20 \times 50 \times 60 \mu\text{m}$) so that conventional preparation techniques could not be used. In addition, this limited crystal size complicated the orientation of the crystals for subsequent preparation. The HRTEM samples were prepared by embedding oriented crystals between two silicon wafer pieces (sandwich technique). The “sandwich” was thinned by a Well diamond saw and trimmed to a 3 mm disk. Subsequently, the disk was thinned with a GATAN dimple grinder. Finally, the sample was followed up with a GATAN Precision Ion Polishing System (PIPS). The HRTEM investigation was carried out on a PHILIPS CM30ST (Super Twin) transmission electron microscope.

3. Results and discussion

The X-ray diffraction data of all single crystals can be indexed with a C -centered monoclinic super cells. The lattice parameters are given in Table 1.

Reciprocal lattice planes $hk0$, hhl and $h0l$ (the indices correspond to the monoclinic supercells) were reconstructed from the measured data. The images are given in Fig. 2. The HRTEM image and the corresponding selected area diffraction pattern of $\text{DyCu}_{3.5}$ along the direction $a^s + b^s$ are given in Fig. 3. Based on a close inspection of the X-ray diffraction reciprocal lattice planes in Fig. 2, it can be concluded that all three crystal structures are derived from the same hypothetical cubic structure of DyCu_5 (AuBe_5 type) by applying the building principle of $\text{YbCu}_{4.5}$ with a different multiplicity of the super cell and with a different monoclinic distortion. The reflections of the subcell can be seen on all reciprocal lattice planes $hk0$ and $h0l$ as main reflections. The satellite reflections, some of them are as strong as main reflections, are situated along the directions $a^{*s} + b^{*s}$ and $a^{*s} - b^{*s}$ and along the direction c^{*s} . The satellite reflections clearly show different

Table 1
Lattice parameters of $\text{DyCu}_{4.5}$, DyCu_4 and $\text{DyCu}_{3.5}$

	a^s (Å)	b^s (Å)	c^s (Å)	β (°)
$\text{DyCu}_{4.5}$	49.55(1)	49.56(1)	46.33(1)	97.5(1)
DyCu_4	28.370(1)	28.360(1)	25.658(1)	103.494(5)
$\text{DyCu}_{3.5}$	21.328(3)	21.292(3)	18.910(3)	108.26(2)

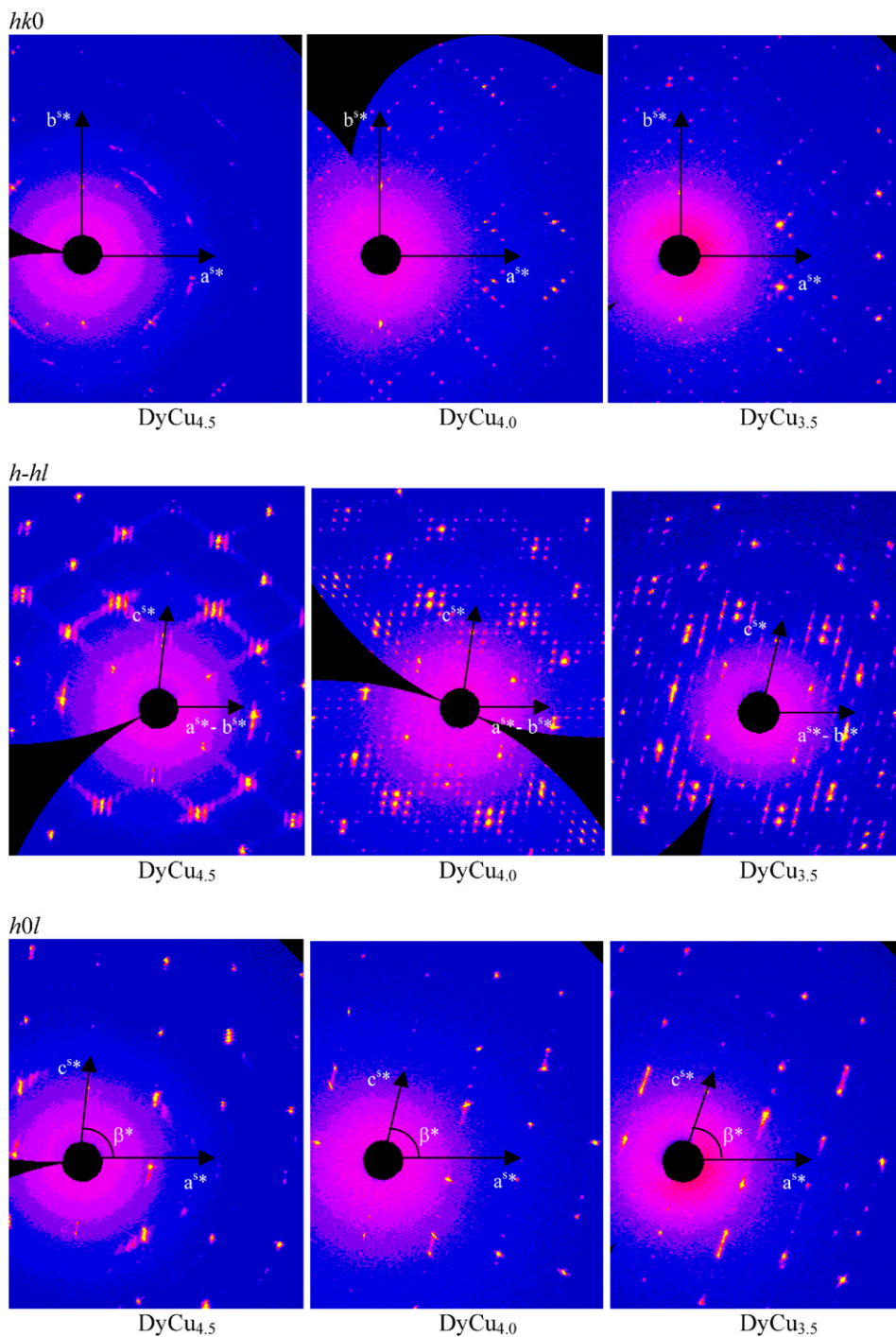


Fig. 2. Reciprocal lattice planes $hk0$, $h-hl$ and $h0l$ of $\text{DyCu}_{4.5}$, DyCu_4 and $\text{DyCu}_{3.5}$. Note that only the directions of the reciprocal axis are of relevance, not their length.

periodicities of the three structures, which correspond to three different multiplicities of the supercell: $7 \times 7 \times 6.5$ for $\text{DyCu}_{4.5}$, $4 \times 4 \times 3.5$ for DyCu_4 and $3 \times 3 \times 2.5$ for $\text{DyCu}_{3.5}$. From these results it can be concluded that the compound $\text{DyCu}_{4.5}$ is an analogue of $\text{YbCu}_{4.5}$. In addition the structures of two other compounds, $\text{DyCu}_{3.5}$ (already reported in the binary phase diagram [3]) and DyCu_4 (new compound), are both derived from

the AuBe_5 type by the same building principle as $\text{YbCu}_{4.5}$.

3.1. Structural model of the series of compounds AB_{5-x} ($0.5 \leq x \leq 1.5$), $A=\text{Dy}$, Yb , $B=\text{Cu}$

Using the structural model of the $\text{YbCu}_{4.5}$ and the experimental results from the DyCu_{5-x} crystals, we can

construct series of compounds AB_{5-x} ($0.5 \leq x \leq 1.5$) with the same building principle [5], which are based on the $AuBe_5$ type as well. The local structure of $YbCu_{4.5}$ between two planar defects is of the $AuBe_5$ type. The local structure around the shear planes is of the $MgCu_2$ type, a substitution derivative of the $AuBe_5$ type. The molar ratio between the $YbCu_5$ and $YbCu_2$ structural blocks is 5:1. Thus the overall composition $YbCu_{4.5}$ can be rationalized as $5YbCu_5 + 1YbCu_2 = 6YbCu_{4.5}$. The structural block $YbCu_2$ should be more correctly described as $1/2 Yb_2Cu_4$, if we want to compare the same volumes of both structural blocks. We prefer, however, to describe the second structural block as $YbCu_2$ to show clearly that it is of the $MgCu_2$ type.

If the ratio between the blocks AB_5 and AB_2 in a hypothetical compound AB_{5-x} is varied, the overall composition and the multiplicity of the supercell change too. A series of hypothetical compounds is presented in Fig. 4. The ratio 1:1 between the two blocks corresponds to the composition $AB_{3.5}$, to the smallest multiplicity ($3 \times 3 \times 2.5$) of the supercell and to the smallest size of the triangles in the structural projection along $a^s \pm b^s$. The ratio 2:1 between the two blocks corresponds to the

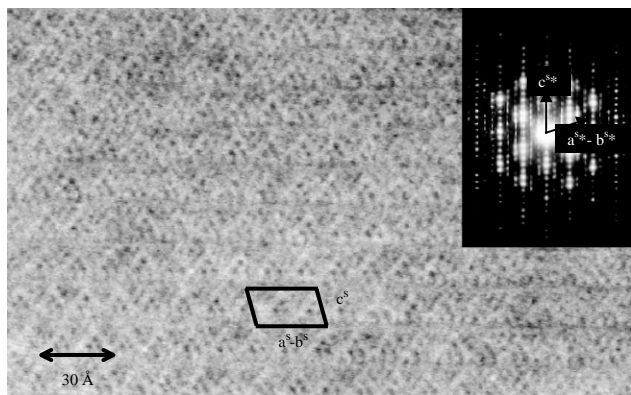


Fig. 3. A HRTEM image and the corresponding selected-area diffraction pattern of $DyCu_{3.5}$ along the direction $a^s + b^s$. The supercell is outlined in black. Note that only the directions of the reciprocal axis are of relevance, not their length.

composition AB_4 and to the multiplicity ($4 \times 4 \times 3.5$) of the supercell. A structural model of $DyCu_{3.5}$ and $DyCu_{4.0}$ was created in the same way as the model of $YbCu_{4.5}$, as described in Ref. [5]. The numerical values of the shifts applied to regions II and I' along the directions $-a^s - b^s$ ($-a^s + b^s$) and c^s (called hereafter s_1 and s_2 respectively) can be derived from the observed lattice parameters of the monoclinic supercell (c^s, β) and multiplicity (m) of the supercell based on the following model (see Fig. 5):

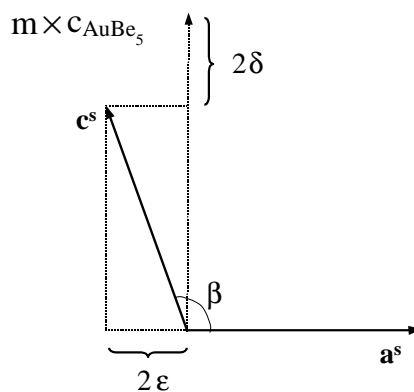
$$2\varepsilon = c^s \times \sin(\beta - 90),$$

$$2\delta = m \times c_{AuBe_5} - c^s \times \cos(\beta - 90),$$

$$s_1 = \varepsilon / a_{AuBe_5},$$

$$s_2 = \delta / c_{AuBe_5}.$$

The lattice parameters a_{AuBe_5} and c_{AuBe_5} correspond to a hypothetical cubic structure AB_5 of the $AuBe_5$ type. The shifts calculated by this model are given in Table 2.



$$s_1 = \varepsilon / a_{AuBe_5}$$

$$s_2 = \delta / c_{AuBe_5}$$

Fig. 5. Geometrical model of the shifts s_1 and s_2 between the structural blocks I, II and II, I' respectively used in the calculation of the structural models of compounds AB_{5-x} . Lattice parameters a_{AuBe_5} and c_{AuBe_5} correspond to a hypothetical structure AB_5 with the structure type $AuBe_5$.

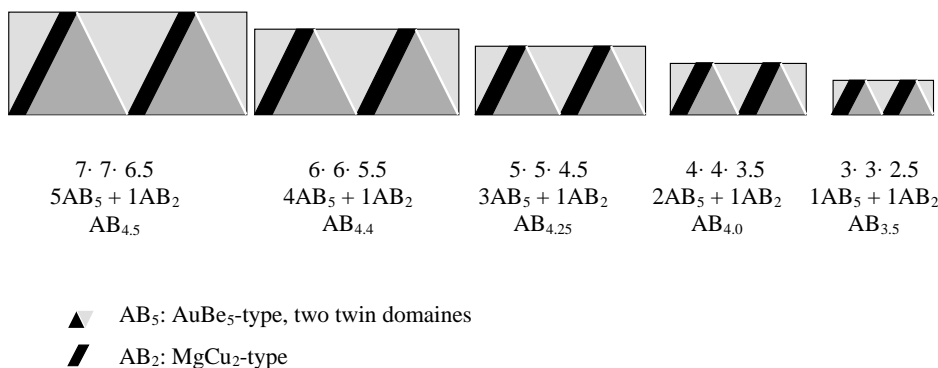


Fig. 4. Series of compounds AB_{5-x} derived from the structure type $AuBe_5$ by ordering of planar defects $\{hhh\}$.

The shifts s_1 and s_2 (for more details see Ref. [5]) of structural blocks AB_5 (AuBe₅ type) result in the AB_2 structure (MgCu₂ type) around the shear planes (black bands in Figs. 4 and 6) and two AB_5 (AuBe₅ type) orientation twin domains (light and dark gray triangles in Figs. 4 and 6).

The positions of 27 and 63 Dy atoms, and of 93 and 249 Cu atoms (all atoms in general position $4c$ of the space group $C2$) have been generated for DyCu_{3.5} and DyCu_{4.0}, respectively, yielding a total of 480 and 1248 atoms per cell and the compositions DyCu_{3.44} and DyCu_{3.95}, respectively. The observed main reflections and satellites along c^{s*} of DyCu_{3.5} and DyCu₄ were used to calculate the agreement factor $wR_F = 0.18$ and 0.17 , respectively, between observed and calculated structure factor amplitudes. The same reflections were used for YbCu_{4.5} in Ref. [5]. The other reflections were diffuse and could not be used for the calculations.

The projection of the structural model of DyCu_{3.5} along the $-a^s - b^s$ direction is shown in Fig. 6. The triangles, which are delimited by the planar defects, are visible only in the upper part of the cell, as in the case of

YbCu_{4.5}. They are smaller, than those in YbCu_{4.5} (compare Figs. 3 and 6 given here with Figs. 3 and 6 in Ref. [5]).

3.2. Description of the series AB_{5-x} ($0.5 \leq x \leq 1.5$), as series $nAB_5 + AB_2$

The structural model of the AB_{5-x} series presented here describes a new series of monoclinic structures (space group $C2$) that can be derived by a stacking of structural AB_5 blocks (cubic AuBe₅ type) and AB_2 blocks (cubic MgCu₂ type). Resulting monoclinic superstructures are derived from the cubic AuBe₅ type by introducing planar defects parallel to $\{hhh\}$ which lead to a nearly orthogonal supercell. Related series based on a stacking of AB_5 (hexagonal CaCu₅ type) and A_2B_4 (i.e., $2AB_2$, cubic MgCu₂ type) building blocks have been described in Refs. [8,9], and later generalized by including A_2B_7 building block [10]. Two sorts of stacking series occur: the rhombohedral–hexagonal $A_{n+1}B_{5n-1}$, as obtained by stacking $(n-1)AB_5 + A_2B_4$, giving the compounds $AB_2, AB_3, A_2B_7, A_5B_{19}, AB_4, \dots, AB_5$, and the monoclinic–hexagonal–trigonal–orthorhombic $A_{n+1}B_{5n+2}$, as obtained by stacking $(n-1)AB_5 + A_2B_7$, giving the compounds $A_2B_7, AB_4, A_4B_{17}, A_5B_{22}, A_6B_{27}, \dots, AB_5$. In all of these the stacking of the structural blocks occurs perpendicular to one crystallographic plane, i.e., hexagonal (001).

In the present series the stacking is more complicated, as it occurs perpendicular to all $\{hhh\}$ planes of a cubic structure, and involves a 3-dimensional array of AB_5 and AB_2 type structures. It can be considered as a special case of crystallographic shear structures [11]

Table 2

The shifts s_1 and s_2 between structural blocks I, II and II, I' respectively used in the calculation of the structural models of compounds AB_{5-x} . Lattice parameters a_{AuBe_5} and c_{AuBe_5} correspond to a hypothetical cubic structure AB_5 of the AuBe₅ type

Compound	s_1	s_2	$a_{AuBe_5} c_{AuBe_5}$ (Å)
DyCu _{4.5}	0.4258	0.2653	7.10
DyCu _{4.0}	0.4229	0.2401	7.10
DyCu _{3.5}	0.4174	0.2354	7.10
YbCu _{4.5}	0.0706	0.2405	7.00

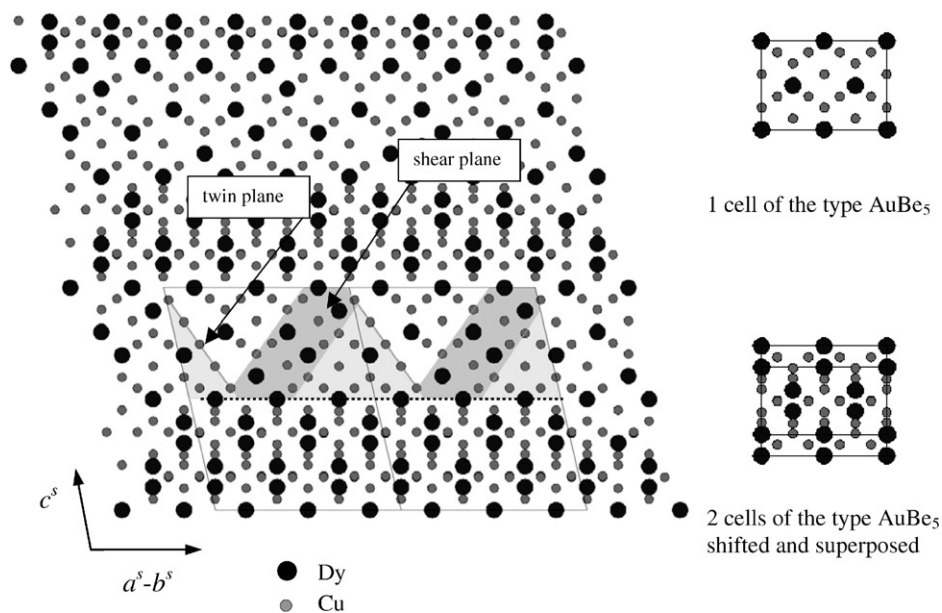


Fig. 6. Projection of the structural model of DyCu_{3.5} along $a^s + b^s$.

where the shear occurs on several non-coplanar planes. It is evident that our series is an analogue to the series $(n-1)AB_5 + A_2B_7$ in which hexagonal $CaCu_5$ type blocks are formally replaced by cubic $AuBe_5$ type blocks. As shown by [12] for Zr_2Ni_7 , which is a member of the $A_{n+1}B_{5n+2}$ series, A_2B_7 blocks can be obtained by stacking a cubic $AuBe_5$ type block and a half block of A_2B_4 of the cubic $MgCu_2$ type (i.e., one block AB_2) perpendicular to a $\{hhh\}$ plane in both cubic structures. The structure of Zr_2Ni_7 then appears as an analogue to the $DyCu_{3.5}$ structure presented here. For reasons of clarity and keeping with simple structure types we prefer, however, to describe the $A_{n+1}B_{5n+2}$ series presented here as a stacking $nAB_5 + AB_2$ rather than $(n-1)AB_5 + A_2B_7$.

The shifts s_1 that were used in the construction of structural models for different structures of the series (Table 2) are different for Dy-compounds and Yb-compounds.² However, they are clearly related by an operation $s_1 \rightarrow 1/2 - s_1$. Locally, around the plane defects $\{hhh\}$, the same structure is obtained when the shifts $(-s_1, -s_1, -s_2)$ or $(1/2 + s_1, 1/2 + s_1, -s_2)$ are used. In the resulting superstructure it corresponds to an interchange between the twin and shear planes, which can be concluded by a careful comparison of Fig. 6 in Ref. [5] and Fig. 6 presented here. However, the translation periodicity (monoclinic angle β and cell parameter c) of the whole superstructure changes. Whether this is related to the type of atom A (Yb or Dy) or whether for one type of atom A , different structures can occur for a given composition remains to be investigated.

The coordination of Dy atoms in the series presented here is the same as in $YbCu_{4.5}$.³ Three types of the coordination of atom A can be found in all compounds of the series:

- (i) Atoms A in the vicinity of shear planes have 13 B and 2 A neighbors. The $A-A$ distance for $A = Dy$ is $\approx 3.57 \text{ \AA}$, which is close to the distance in elemental dysprosium (3.59 \AA).
- (ii) Atoms A in the vicinity of twin planes have 15 B neighbors and 2 A neighbors. The $A-A$ distance for $A = Dy$ is $\approx 4.5-4.55 \text{ \AA}$.
- (iii) Atoms A in the $AuBe_5$ type regions have 16 B neighbors. The closest A atoms are 5.02 \AA (for $A = Dy$) apart.

More precise analysis of coordination polyhedrons near the plane defects cannot be done at present, because the atomic positions are not refined. However, in general, there occur three types of A atom sites

(coordinations i., ii. and iii.) in each structure of the series presented here. The molar ratio between these types in the structure, however, varies between the different members of the series: It is about 25%, 25% and 50% for $AB_{4.5}$, 40%, 30% and 30% for AB_4 and 50%, 35% and 15% for $AB_{3.5}$, respectively. Different fraction of Yb atoms of the (i)-type (metallic Yb–Yb bounds) in the $YbCu_{4.5}$ and $YbCu_{3.5}$ compounds of the $Yb_{n+1}Cu_{5n+2}$ series can be at the origin of different electronic properties of these compounds as observed in Ref. [13]. Interestingly, in all structures of the series presented here the coordination of the B atoms lying near the shear and on the twin planes are similar, i.e., they consist of 8 B and 4 A neighbors, as compared to 9 B and 3 A neighbors in the $AuBe_5$ type regions.

Based on the synthesis of the sample $DyCu_{3.5}$ we have obtained a crystal with the composition $Dy_{14}Cu_{51}$ ($DyCu_{\sim 3.6}$) and the structure of the type $Ce_{14}Cu_{51}$ [14] ($P6/m$, $a = 11.530$, $c = 8.665 \text{ \AA}$, disordered variant of the type $Gd_{14}Ag_{51}$). The results will be published elsewhere. It is interesting to note here that $DyCu_{3.5}$ and $DyCu_{\sim 3.6}$ have very similar composition but completely different crystal structures.

It is also interesting to note that we have recently found that a single crystal of the composition $ScCu_4$ (Kotur and Derkach, pers. comm.) has the diffraction pattern that is similar to that of the $DyCu_4$ and can be indexed with the C -centered monoclinic cell with the parameters $a = 27.644(3)$, $b = 27.636(3)$, $c = 25.055(4) \text{ \AA}$, $\beta = 103.82(2)^\circ$. However, due to diffuse diffraction pattern the structural model could not be verified.

4. Conclusions

We have described the series of monoclinic compounds $A_{n+1}B_{5n+2}$ constructed from structural blocks AB_5 (cubic structure type $AuBe_5$) and AB_2 (cubic structure type $MgCu_2$) by stacking $nAB_5 + AB_2$ and giving the compositions A_2B_7 , AB_4 , A_4B_{17} , A_5B_{22} , A_6B_{27} , ..., AB_5 . Resulting monoclinic superstructure is derived from the cubic $AuBe_5$ type by introducing planar defects parallel to $\{hhh\}$ which leads to a nearly orthogonal $\approx (n+2) \times (n+2) \times (n+2-0.5)$ supercell. The known representative are $YbCu_x$ ($x = 4.5$ and 3.5) and $DyCu_x$ ($x = 4.5, 4$ and 3.5). The series are the analogue of the monoclinic–hexagonal–trigonal–orthorhombic series $A_{n+1}B_{5n+2}$ obtained by stacking $(n-1)AB_5 + A_2B_7$ where AB_5 is of the hexagonal structure type $CaCu_5$ and A_2B_7 is of the monoclinic structure type Zr_2Ni_7 .

Acknowledgments

We thank K. Schenk and M. Bonin from the University of Lausanne for the help with the X-ray diffraction data

²Note that in Ref. [5] the shifts were applied as $(-s_1, 1/2 - s_1, 1/2 - s_2)$ which is (due to the F -lattice of the cubic AB_5 structure) equivalent to $(-s_1, -s_1, -s_2)$ as applied here.

³Note that the coordination of atoms A of the (i) and (ii) type was wrongly given as 14 B and 2 A atoms in Ref. [5].

collection. The discussion about different structure types of intermetallic compounds with O. Bodak from the University of L'viv is highly appreciated. We thank Christian Soltmann from the ETH Zürich for a careful reading of the manuscript. This work was supported by the Swiss National Science Foundation.

References

- [1] P.R. Subramanian, D.E. Laughlin, *Bull. Alloy Phase Diag.* 9 (3a) (1988) 316.
- [2] M.M. Carnasciali, S. Cirafici, E.A. Franceschi, *J. Less-Common Met.* 92 (1983) 143–147.
- [3] E. Franceschi, *J. Less-Common Met.* 87 (1982) 249–256.
- [4] A. Iandelli, A. Palenzona, *J. Less-Common Met.* 25 (1971) 333–335.
- [5] R. Černý, M. François, K. Yvon, D. Jaccard, E. Walker, V. Petříček, I. Čiřarová, H.-U. Nissen, R. Wessicken, *J. Phys.: Condens. Matter* 8 (1996) 4485–4493.
- [6] V.Ya. Markiv, I.S. Gavrilenko, V.V. Petkov, N.N. Belavina, *Metallofizika* 73 (1978) 39–45 (in Russian).
- [7] B.Ya. Kotur, V.O. Derkach, I.S. Dutsyak, A.Z. Pavlyshyn, *J. Alloys Compd.* 238 (1996) 81–85.
- [8] D.T. Cromer, A.C. Larson, *Acta Cryst.* 12 (1959) 855–859.
- [9] Y. Khan, *Acta Cryst. B* 30 (1974) 1533–1537.
- [10] E. Parthé, R. Lemaire, *Acta Cryst. B* 31 (1975) 1879–1889.
- [11] B.G. Hyde, S. Andersson, *Inorganic Crystal Structures*, Wiley-Interscience, New York, 1989.
- [12] F.R. Eshelman, J.F. Smith, *Acta Cryst. B* 28 (1972) 1594–1600.
- [13] D. Jaccard, A. Junod, J. Sierro, *Helv. Phys. Acta* 53 (1980) 583–589.
- [14] C. Allibert, W. Wong-NG, S.C. Nyburg, *Acta Cryst. C* 40 (1984) 211–214.