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Changes in published type structures.

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Abstract

The structure of $KSbSe_2$ was first determined [Kaniščeva, Palkina, Kuznecov, Lazarev & Tarasova (1976). *Izv. Akad. Nauk SSSR Neorg. Mater.* **12**, 537–538] in $P1$. Later it was independently determined in $P\bar{1}$ [Dittmar & Schäfer (1977). *Z. Naturforsch. Teil B*, **32**, 1346–1348]. The $P1$ description can be made equivalent to the $P\bar{1}$ structure within reasonable limits and it is concluded that there is only one form with space group $P\bar{1}$. The structure of $OsGe_2$ was determined [Weitz, Born & Hellner (1960). *Z. Metallkd.* **51**, 238–243] in $C2/m$. Later the structures of $NbAs_2$ and $NbSb_2$ were determined in $C2$ [Furuseth & Kjekshus (1964). *Nature (London)*, **203**, 512] and were regarded as a new structure type. Structures assigned to this type can be described within reasonable experimental error limits as being of the $OsGe_2$ type. Thus the $OsGe_2$ structure in $C2/m$ is to be preferred for classification purposes.

While preparing the structural indices for a new edition of the Metals and Alloys subfile (Powder Diffraction File, 1991) it was found that some type structures for intermetallic phases could be described with higher symmetry. The structure of $KSbSe_2$ was determined by Kaniščeva, Palkina, Kuznecov, Lazarev & Tarasova (1976) in $P1$ and refined to $R = 0.149$ for 1400 independent reflections based on diffractometer measurements with Mo radiation. Later Dittmar & Schäfer (1977) independently determined a structure for $KSbSe_2$ in $P\bar{1}$ and refined it to $R = 0.049$ for 1721 reflections based on diffractometer measurements with monochromatized Mo radiation. The descriptions of these two structures are very similar (*Structure Reports*, 1976, Vol. 42A, pp. 20–21; 1978, Vol. 44A, pp. 14–15) and furthermore the powder patterns calculated (Powder Diffraction File, 1990) from the coordinates given (Villars & Calvert, 1985) are virtually identical. Therefore these two reports were compared by running the program *CREDUC* (Le Page, 1982). This revealed the possibility of monoclinic symmetry for both unit cells (Table 1). However when the program *MISSYM* (Le Page, 1987, 1988) was run on the coordinates, triclinic solutions for both structures were calculated but for the $P1$ set an inversion centre was revealed at -0.242, -0.389, -0.186 (referred to the original cell). The discrepancies between the coordinates of pairs of related atoms in the $P1$ set, before averaging, had a range of 0 to 0.194 Å, with $\sigma = 0.065$ Å. The $P1$ coordinates, after averaging, are compared in Table 2 to the $P\bar{1}$ set. The differences have a range of 0.001 to 0.034 Å with $\sigma = 0.009$ Å and mean of 0.015 Å. It seems reasonable to conclude that $KSbSe_2$ has only one triclinic form and that the correct space group is $P\bar{1}$.

Table 1. *Possible monoclinic unit cells derived from the reduced cells*

Ref.	Formula	a (Å)	b (Å)	c (Å)	α ( )	β ( )	γ ( )
D*	$KSbSe_2$	8.0132	10.1029	12.4810	89.898	105.164	89.954
K†	$KSbSe_2$	8.0132	10.1119	12.4820	90.138	105.197	90.0

* Dittmar & Schäfer (1977).
† Kaniščeva, Palkina, Kuznecov, Lazarev & Tarasova (1976).

The structure of $OsGe_2$ was determined in $C2/m$ (Weitz, Born & Hellner, 1960; *Structure Reports*, 1960, Vol. 24, pp. 144–145) and refined to $R = 0.19$ for 540 reflections based on densitometer measurements of photographic data corrected for absorption. In 1964 the structures of $NbSb_2$ and $NbAs_2$ were determined in $C2$ (Furuseth & Kjekshus, 1964; *Structure Reports*, 1964, Vol. 29, pp. 18–21; Furuseth & Kjekshus, 1965; *Structure Reports*, 1965, Vol. 30A, pp. 14–16) based on photographic data corrected for absorption and taken with Cu radiation; for $NbAs_2$, $R = 0.11$ but for $NbSb_2$ refinement could not be taken beyond $R = 0.20$. As has been emphasized (Schomaker & Marsh, 1979), refinement difficulties can arise when symmetry elements have been neglected or overlooked. For $NbSb_2$ the coordinates reported had strict $C2/m$ symmetry while for $NbAs_2$ the deviations from $C2/m$ symmetry were 0.04 and 0.23 Å for the As atoms. Another $NbAs_2$ type structure, $MoAs_2$, was refined on the basis of photographic intensities for Mo radiation but no R factor was recorded. (Brown, 1965; *Structure Reports*, 1965, Vol. 30A, p. 21). The deviations from $C2/m$ symmetry were 0.10 and 0.003 Å respectively

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Table 2. *The two sets of coordinates for* $KSbSe_2$

	Ref.	x	y	z
K1	D*	0·4132	0·0841	0·2736
	K†	0·414	0·084	0·278
K2	D	0·1536	0·5637	0·3241
	K	0·154	0·566	0·326
Sb1	D	0·6512	0·5792	0·1955
	K	0·6485	0·583	0·1985
Sb2	D	0·9140	0·0088	0·2241
	K	0·912	0·006	0·2235
Se1	D	0·4057	0·5631	0·2142
	K	0·4065	0·560	0·214
Se2	D	0·1354	0·0242	0·1827
	K	0·137	0·026	0·183
Se3	D	0·6762	0·9909	0·2526
	K	0·678	0·9945	0·254
Se4	D	0·8792	0·6036	0·2157
	K	0·8805	0·602	0·217

* Dittmar & Schäfer (1977); both sets of coordinates are in these authors' setting.

† Kaniščeva, Palkina, Kuznecov, Lazarev & Tarasova (1976). These coordinates were originally given to three decimal digits; the fourth is due to the averaging.

Table 3. *Comparison of coordinates for refined examples of the* $OsGe_2$ *and* $NbAs_2$ *structure types*

Coordinates for CrP_2 have had an origin shift of $\frac{1}{2},\frac{1}{2},\frac{1}{2}$ added. Coordinates for $NbAs_2$ and $MoAs_2$ have had an origin shift of $0,0,\frac{1}{2}$ added. The coordinate set given for $OsGe_2$ is taken as standard.

Phase	Atom	x	y	z
$OsGe_2$[a]	Os	0·356	$\frac{1}{2}$	0·800
CrP_2[b]	Cr	0·3426	$\frac{1}{2}$	0·7958
$NbAs_2$[c]	Nb	0·3444	$\frac{1}{2}$*	0·8044
$MoAs_2$[d]	Mo	0·3452	$\frac{1}{2}$*	0·7982
$OsGe_2$	Ge1	0·101	$\frac{1}{2}$	0·888
CrP_2	P1	0·1017	$\frac{1}{2}$	0·9005
$NbAs_2$	As1	0·0948	0·488	0·8928
MoA_2	As1	0·0977	0·4956	0·8933
$OsGe_2$	Ge2	0·143	0	0·531
CrP_2	P2	0·1437	0	0·5288
$NbAs_2$	As2	0·1399	0·067	0·5257
$MoAs_2$	As2	0·1440	0·0225	0·5312

Notes: (*a*) $OsGe_2$ from Weitz, Born & Hellner (1960); (*b*) CrP_2 from Jeitschko & Donohue (1973); (*c*) $NbAs_2$ from Furuseth & Kjekshus (1965); (*d*) $MoAs_2$ from Brown (1965).

* Fixed during refinement because $C2$ is polar.

for the As atoms. No other refinements for this structure type have been found. The $OsGe_2$-type structure was found for CrP_2 (Jeitschko & Donohue, 1973; *Structure Reports*, 1973, Vol. 39A, pp. 8–9) and refined to $R = 0{\cdot}03$ on diffractometer data for 253 reflections recorded with Mo radiation and corrected for dispersion and extinction. The space group $C2$ was specifically tested and rejected and the similarity to $NbAs_2$ was emphasized. A comparison of coordinates for refined examples of the $OsGe_2$ and $NbAs_2$ structure types is given in Table 3. Pearson (1972) gives $NbAs_2$ in $C2$ as a prototype but had suggested that $OsGe_2$ may be of the $NbAs_2$ type (Pearson, 1967). Villars & Calvert (1985) take $NbSb_2$ in $C2$ as a prototype and give $OsGe_2$ separately in $C2/m$. The relatively low accuracy of the photographic single-crystal refinements and the relatively small deviations from $C2/m$ symmetry for the light atoms do not provide a convincing proof of the $C2$ space group and the $NbAs_2$ type as a separate type. All the refinements in $C2$ are potentially $C2/m$ within the stated experimental deviations [*MISSYM* (Le Page, 1987, 1988)]. Also these $C2$ and $C2/m$ cells, when tested with the program *CREDUC* (Le Page, 1982), are strictly monoclinic. Thus in future editions of the Powder Diffraction File (1991) these structures in $C2$ will be classified in $C2/m$ with $OsGe_2$ taken as the prototype structure for reasons of priority.

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