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On the Crystal Structures of the β and γ Phases in the System Indium–Tin

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Visually estimated intensities from powder photographs of the β and γ phases in the system indium–tin have been used to check the simple structures previously proposed for these two alloys. It is found that the tetragonal body-centred structure with two atoms per cell reported for the β phase is incorrect, although the correct structure has not yet been deduced. The γ phase is confirmed as having a structure with primitive hexagonal packing as Fink, Jette, Katz & Schnettler suggested. In this phase each atom is surrounded by six others at a distance of 3·21 Å and by two others at a distance of 2·99 Å.

Introduction

The system indium–tin has been investigated by Valentiner (1940), by Fink, Jette, Katz & Schnettler (1945), and by Rhines, Urquhart & Hoge (1947). This system is characterized by four phases with the following approximate composition limits in weight % (equals roughly atomic %) Sn at 20°C. (Rhines *et al.*, 1947):

$$\alpha\ 0\text{–}3,\quad \beta\ 14\text{–}27,\quad \gamma\ 75\text{–}88,\ \text{and}\ \delta\ 94\text{–}100.$$

The α phase has the structure of indium, body-centred tetragonal with two atoms per cell, and the δ phase the structure of white tin, body-centred tetragonal with four atoms per cell.

The β phase

The β phase was reported by Valentiner (1940) to have a tetragonal F lattice with four atoms per cell, and this was confirmed by Orlamünder in Klemm, Klemm, Hohmann, Volk, Orlamünder & Klein (1948). Fink *et al.* (1945) pointed out that a smaller I lattice with two atoms per cell could be chosen in place of Valentiner's larger F lattice, and confirmed the structure as being of this type. In none of these papers do the authors give intensity data to verify the one structure that is possible for a tetragonal I lattice with two atoms per cell.

Table 1. β-InSn: *X-ray powder data*

Indexed on the (incorrect) tetragonal I lattice, $Z = 2$, for composition 24·37 atomic % Sn; $a = 3{\cdot}463$ Å, $c = 4{\cdot}382$ Å (Fink *et al.*, 1945).

$I_{\text{obs.}}$	$2\theta°$ (Cu)	$d_{\text{meas.}}$ (Å)	hkl	$d_{\text{calc.}}$ (Å)
10	33·15	2·70	101	2·717
$3\frac{3}{4}$	36·85	2·44	110	2·449
5	41·35	2·18	002	2·191
$1\frac{3}{4}$	53·15	1·722	200	1·732
$6\frac{1}{2}$	56·40	1·629	112	1·633
$5\frac{1}{2}$	63·70	1·459	211	1·460
2	69·30	1·354	202	1·358
5	69·90	1·344	103	1·346
$\frac{1}{4}$	77·90	1·225	220	1·224
$\frac{3}{4}$	87·60	1·113	301	1·116
$1\frac{1}{2}$	89·70	1·092	004 310	1·096 1·095
4	92·80	1·064	222 213	1·069 1·063
2	100·90	0·999	114	1·000
2	103·80	0·979	312	0·9796
$1\frac{1}{2}$	110·70	0·936	321	0·9382
$1\frac{1}{2}$	112·60	0·926	204	0·9258
1	116·70	0·905	303	0·9057
3	130·20	0·849	105	0·8496
$3\frac{1}{2}$	137·60	0·826	411	0·8249
$3\frac{1}{2}$	141·40	0·816	224 330	0·8164 0·8162
$5\frac{1}{2}$	146·70	0·804	402 323	0·8052 0·8025
$6\alpha_1$	166·20	0·776	314 420	0·7745 0·7744

Note:

The intensity and spacing of only the α_1 component have been recorded for the last reflection for which α_1 and α_2 are clearly resolved.

We have taken a powder photograph using Cu $K\alpha$ radiation (Cu $K\alpha_1 = 1{\cdot}54050$ Å) of fine filings of the β phase with composition 24·37 atomic % (24·99 weight %) Sn, and we have estimated the intensities visually on a scale of 10. Table 1 gives these intensities along with the measured spacings, the Miller indices based on the tetragonal I lattice, and the spacings calculated on the cell dimensions of Fink *et al.* (1945) for this composition. If the structure is the simple one proposed by the previous authors, the sharp intensity changes from one powder reflection to the next can be due only to multiplicity. A brief examination of Table 1 shows that no simple relationship exists between the

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observed intensities and the multiplicity of this diatomic tetragonal *I* structure: the 002 reflection, for example, with multiplicity 2, has an observed intensity of 5, whereas the 200 reflection, with multiplicity 4, has an observed intensity of only $1\frac{3}{4}$. The conclusion is then that β-InSn does not have a tetragonal *I* lattice with two atoms per cell as previously proposed. Our attempts to find another structure for which the calculated intensities agree with the observed have not been successful, and the solution of this problem must await further investigation.

The γ phase

The γ phase was stated by Valentiner (1940) to have a tetragonal body-centred structure similar to that of white tin. Fink *et al.* (1945) showed that the observed powder reflections do not conform to the extinction conditions required by the tin structure, and that the agreement of Valentiner's calculated spacings with the measured was not satisfactory. They re-indexed the powder pattern in a fully satisfactory way on a primitive hexagonal lattice with one atom per cell. They pointed out that 'a simple hexagonal structure is unusual and the true structure of the γ phase may be one that is merely based on this'. They were unable to verify this simple structure by means of intensity measurements because of the 'spotty character of the lines'.

We have taken powder photographs with Cu $K\alpha$ radiation of fine filings of the γ phase of composition 81·25 atomic % (81·75 weight %) Sn, and have estimated the intensities visually on a scale of 10. One photograph exposed about ten times longer than normal showed no lines in addition to those which index well on the simple hexagonal cell of Fink *et al.* (1945); this must therefore be the correct unit cell of γ-InSn. Table 2 gives our observed intensities, our measured spacings compared with those calculated on the cell dimensions of Fink *et al.* for this composition, and the intensities calculated for a structure with one atom per cell in a simple hexagonal lattice. The satisfactory agreement between $I_{\text{calc.}}$ and $I_{\text{obs.}}$ is proof that the structure of γ-InSn is indeed one with primitive hexagonal packing, as the work of Fink *et al.* suggested.

In this simple structure each atom is surrounded by eight others at the corners of a hexagonal bipyramid, six in one plane at a distance of 3·21 Å, and two directly above and below at a distance of 2·99 Å. In comparison, each atom in white tin is surrounded by a distorted tetrahedron of four neighbours at a distance of 3·02 Å, and by two more at 3·17 Å; and each atom in indium, which has a distorted cubic close-packed structure, is surrounded by four others at 3·24 Å, and by eight more at 3·37 Å (Wyckoff, 1951). If the β phase had the tetragonal body-centred structure first assigned it, each atom would be surrounded by eight others at the corners of a right prism at about 3·28 Å, and by four others at the corners of a square at 3·46 Å.

Table 2. γ-InSn: *X-ray powder data*

Hexagonal, $P6/mmm$, $Z = 1$, for composition 81·25 atomic % Sn; $a = 3{\cdot}211$ Å, $c = 2{\cdot}992$ Å (Fink *et al.*, 1945).

$I_{\text{obs.}}$	$2\theta°$ (Cu)	$d_{\text{meas.}}$ (Å)	*hkil*	$d_{\text{calc.}}$ (Å)	$I_{\text{calc.}}$
$2\frac{1}{2}$	30·05	2·97	0001	2·992	4·0
10	32·35	2·77	$10\bar{1}0$	2·781	11·0
10	44·55	2·03	$10\bar{1}1$	2·037	12·1
$4\frac{1}{2}$	57·35	1·604	$11\bar{2}0$	1·606	4·0
$\frac{1}{2}$	62·20	1·490	0002	1·496	1·1
7	66·00	1·413	$11\bar{2}1$	1·415	6·0
$3\frac{1}{2}$	67·35	1·388	$20\bar{2}0$	1·390	2·9
5	71·70	1·314	$10\bar{1}2$	1·317	5·2
5	75·45	1·259	$20\bar{2}1$	1·261	4·7
4	89·55	1·094	$11\bar{2}2$	1·095	3·7
4	94·25	1·051	$21\bar{3}0$	1·051	3·5
4	98·35	1·018	$20\bar{2}2$	1·018	3·5
0	(101·20)	—	0003	0·9973	0·6
7	101·90	0·992	$21\bar{3}1$	0·9916	7·1
$3\frac{1}{2}$	110·20	0·939	$10\bar{1}3$	0·9388	3·7
$1\frac{1}{2}$	112·45	0·927	$30\bar{3}0$	0·9270	1·9
$4\frac{1}{2}$	120·60	0·887	$30\bar{3}1$	0·8854	4·1
$7\frac{1}{2}\alpha_1$	126·80	0·861	$21\bar{3}2$	0·8600	6·3
$5\alpha_1$	130·20	0·849	$11\bar{2}3$	0·8472	3·3
$5\alpha_1$	143·10	0·812	$20\bar{2}3$	0·8104	4·6
$2\alpha_1$	146·40	0·805	$22\bar{4}0$	0·8028	2·5
$6\alpha_1$	154·80	0·789	$30\bar{3}2$	0·7879	6·9
$6\alpha_1$	165·10	0·777	$22\bar{4}1$	0·7753	11·8

Notes:

(*a*) Where α_1 and α_2 are clearly resolved, the intensity and spacing of only the α_1 component have been recorded; such reflections are marked α_1. For these reflections the stated value of $I_{\text{calc.}}$ equals two-thirds that actually calculated, the assumption being that α_1 has twice the intensity of α_2.

(*b*) $I_{\text{calc.}}$ is given by

$$I_{\text{calc.}} = F^2 p \frac{1+\cos^2 2\theta}{\sin^2\theta\cos\theta} ATK,$$

where K is a constant and the other letters have their usual crystallographic meanings. The absorption factor A was calculated in the manner of Henry, Lipson & Wooster (1951). In the temperature factor $T = \exp\left[-B(\sin\theta/\lambda)^2\right]$, the variable B was chosen as 3 Å^2. In order to place $I_{\text{calc.}}$ on the same scale as $I_{\text{obs.}}$, and to give the best overall agreement between the two sets of values, K was arbitrarily chosen as 1/1340.

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