

Short Communications

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Die Überstruktur der γ -Hochtemperaturphase im System Kupfer–Zinn. Von H. HENDUS und H. KNÖDLER, *Institut für Metallforschung, Saarbrücken 15, Deutschland*

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Bisher bestand keine Klarheit darüber, auf welche Weise sich im System Cu–Sn die raumzentrierte β -Phase (Existenzbereich z. B. bei 700° C. von 13,9 bis 15,3 At. % Sn)* und die raumzentrierte γ -Hochtemperaturphase (bei 700° C. von 16,2 bis 25,5 At. % Sn) strukturell voneinander unterscheiden, und ob die von einigen Autoren† ausgesprochene Vermutung, dass die γ -Phase eine Überstruktur besitze, zutrifft.

Im Zusammenhang mit noch laufenden Untersuchungen in anderen Phasenbereichen des Systems Cu–Sn wurde dieser Frage anhand von Röntgenaufnahmen nachgegangen. Die Auswertung der Debye–Scherrer-Diagramme, aufgenommen an 15 Legierungsproben im Bereich von 11,81 bis 25,00% bei 700° C. mit Cu $K\alpha$ -Strahlung (λ (Cu $K\alpha_1$) = 1,54050 Å), ergab einen stetigen Verlauf der Gitterkonstanten vom Beginn der β -Phase mit $a_0 = 3,018_3$ Å‡, über das zwischen 15,3 und 16,2 At. % Sn gelegene β/γ -Mischgebiet hinweg, bis 25,00% mit $a_0 = 3,058_3$ Å. Bis etwa 15% verläuft die Gitterkonstante anscheinend linear mit der Konzentration, darüber mit zunehmender negativer Krümmung.

Auf den Röntgenaufnahmen treten bei 15% schwach die ersten Überstrukturlinien auf; ab 17% werden sie deutlich und vollständig. Am ausgeprägtesten sind die

Zusatzlinien im Röntgendiagramm der Legierung mit 25,00 At. % Sn; Lage und Intensität aller Linien stimmen überein mit denen der berechneten Interferenzen (700° C). für das kubische Gitter der Verbindung Cu₃Sn mit der Gitterkonstanten:

$$a_\gamma = 2a_0 = 6,116_6 \text{ \AA};$$

16 Atomen (12 Cu, 4 Sn) je Zelle auf den Gitterplätzen:

$$4 \text{ Cu: } 0, 0, 0; \frac{1}{2}, 0, 0; \bar{1}, 0, 0;$$

$$4 \text{ Cu: } \frac{1}{2}, \frac{1}{2}, \frac{1}{2}; \frac{1}{2}, \frac{1}{2}, 0; \bar{1}, 0, 0;$$

$$4 \text{ Cu: } \frac{3}{4}, \frac{3}{4}, \frac{3}{4}; \frac{3}{4}, \frac{1}{4}, \frac{1}{4}; \bar{1}, 0, 0;$$

$$4 \text{ Sn: } \frac{1}{4}, \frac{1}{4}, \frac{1}{4}; \frac{3}{4}, \frac{3}{4}, \frac{1}{4}; \bar{1}, 0, 0;$$

Nach den vorliegenden Ergebnissen ist die γ -Hochtemperaturphase im System Cu–Sn analog zur Überstruktur im Fe₃Al-Gitter geordnet.

Über diese Strukturbestimmung und damit in Zusammenhang stehende Untersuchungen wird später eingehend berichtet werden.

Literatur

HANSEN, M. (1936). *Der Aufbau der Zweistofflegierungen*. Berlin: Springer.

RAYNOR, G. V. (1944). *Annotated Equilibrium Diagram Series*, No. 2. London: Institute of Metals.

SMITH, C. V. (1948). *Metals Handbook*, S. 1204. Cleveland: American Society of Metals.

* Alle Phasengrenzkonzentrationen sind dem von Raynor (1944) dargestellten Diagramm Cu–Sn entnommen.

† Vergl. die Zusammenfassung der Arbeiten über Cu–Sn bei Hansen (1936) und Smith (1948).

‡ a_0 = Gitterkonstante bei statistischer Atomverteilung auf die Punktlagen des kubisch raumzentrierten Gitters.

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The structure of thorium–magnesium intermetallic compounds.* By D. T. PETERSON, P. F. DILJAK and C. L. VOLD, *Ames Laboratory, Institute for Atomic Research, Iowa State College, Ames, Iowa, U. S. A.*

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In the course of a preliminary investigation of the thorium–magnesium phase system, two intermetallic compounds were observed. The samples were prepared by heating magnesium sealed in a thorium metal crucible. The resulting samples were often not homogeneous, but

large primary crystals of an intermetallic compound were formed. These crystals were separated mechanically from the matrix, analysed, and examined by X-ray diffraction. Samples heated at 800° C. and above exhibited, for the most part, dendrites with a pyramidal form. Samples heated at 700° C. and below were found to contain large primary crystals of a tabular form. Analysis of a massive sample of the compound formed at 800° C. found a

* Contribution No. 457. Work was performed in the Ames Laboratory of the Atomic Energy Commission.

thorium content of 83.6 weight%. The compound formed at 700° C. and below could be isolated only by mechanical separation of the dendritic crystals. Analyses of these samples found 78 weight% thorium. Small amounts of the magnesium-rich matrix could easily have adhered to the samples of the low-temperature form. Accurate density determinations could not be made because of the small sample size. The analytical results indicated that the compounds were very close to ThMg₂.

Crystals of both compounds were examined by X-ray diffraction with a Weissenberg camera. The high-temperature form was found to belong to the space group *Fd3m* and have a lattice constant of 8.570 Å. The compound has the Cu₂Mg, *C15* structure and is therefore a Laves phase. The analysed composition of the phase and the qualitative agreement between calculated and observed intensities leave no doubt as to the structure. There are no parameters involved in the location of the atoms.

The low-temperature form was found to belong to the space group *C6/mmc* and to have the Ni₂Mg, *C36* structure. The lattice constants are: $a_0 = 6.086$ Å, $c_0 = 19.64$ Å. The axial ratio c/a is 3.23. This structure involves several parameters. The parameters reported in *Strukturbericht* for Ni₂Mg were used for a trial structure. The calculated intensities agreed very well with the observed intensities

for Weissenberg patterns through the fourth layer and for 00*l* precession patterns. No attempt was made to refine the atomic parameters because the magnesium atoms could not be located precisely, owing to their small contribution to the intensities. The calculation of the position of the thorium atoms must be quite accurate in order to have given the excellent qualitative agreement of the calculated and observed intensities.

These compounds seem to belong to the family of Laves phases and are additional examples of the formation of one type at high temperature and another at a lower temperature. The cubic, high-temperature form does not transform readily into the hexagonal modification. Samples were observed to contain crystals of both types even after heating at 700° C. for 48 hr.

The radius ratio for magnesium to thorium radii is 0.889. Closest bond distances in the two structures are:

	Th-Th	Th-Mg	Mg-Mg
Cubic	3.711 Å	3.553 Å	3.029 Å
Hexagonal	3.69	3.53	3.04

The bond distances for the hexagonal compound are not as accurate as those for the cubic compound. The distances are very nearly the same in both forms, as would be expected from the structural similarity.

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A graphical aid for the rapid evaluation of absorption corrections by Albrecht's method.

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The labour of estimating absorption corrections by Albrecht's (1939) method can be considerably reduced with the aid of the following simple procedure.

An outline of the cross-section of the crystal is drawn on tracing paper to a scale such that dimensions of length x correspond to true lengths, x' , by the relation

$$x = \mu x',$$

where μ is the linear absorption coefficient of the crystal, and m is a constant (see below). This is pinned down permanently in the correct orientation over the corresponding reciprocal lattice (see the bold outline *EFGH* in Fig. 1). Over this again are placed two transparent sheets marked in their overlapping parts with uniformly spaced parallel lines. They are pinned to the board at O , the origin of the reciprocal net, and are rotated for each reflexion so that one set of rulings, I , is parallel to the incident rays; the other, D , to the diffracted rays. This is greatly facilitated if both sheets carry a Bernal circle to match the underlying reciprocal lattice: both circles are arranged to intersect at S , the reciprocal-lattice point.* The absorption suffered by a reflexion could be

estimated by listing $(l_1 + l_2)$ (Fig. 1) for as many (N) points of intersection of the ray rulings as are necessary, and then subsequently evaluating

$$A = N^{-1} \sum \exp [-(l_1 + l_2)/m].$$

We have, however, found it especially convenient to use a transparent rule, *RR'* (Fig. 1), graduated to read $\exp[-x/m]$, where x is in cm. This is laid along the incident ray with its origin at the point of entry into the crystal. Using a pair of dividers, one may find the point Q on the scale corresponding to $(l_1 + l_2)$ from the origin and so read off the attenuation directly. This is quickly repeated for $P_1 - P_7$, and, after a shift of the ruler, for P_8 etc. The factors are accumulated in a desk adder. The graduations of *RR'* are most conveniently copied from the *C*(10:1) or *D*(100:1) scales of any 10 in. or 25 cm. slide rule. The corresponding values of m are 11.03 (10 in.) and 10.86 (25 cm.) for the *C* scale, and half these for the *D* scale. It is advisable to mark the ruler with the appropriate factor.

Heavily absorbing crystals are represented by large sections and so have many points of intersection of the ray rulings. If the rulings are coloured alternately red and black, one can, by using either red-red (or black-black), red-black, or all the intersections, sample at uniformly spaced points representing either one quarter, one half, or all of the available intersections.

If, for weakly absorbing crystals, the cross-section is fairly regular, the variation of A throughout most of

* Incidentally, when used in this way, these sheets are convenient for the recognition and interpretation of cases of 'Umweganregung', the Renninger (1937) effect. This will occur if another lattice point T also falls on the *D*-circle, and if both reflexions S , T are strong. The simulated reflexion corresponds to a point U on the *I*-circle.