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Abstract

Full Text

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ON EQUILIBRIUM IN THE TITANIUM–TIN SYSTEM

(Presented by Academician I. I. Chernyaev on 9 V 1960)

A considerable number of studies have been devoted to the investigation of the chemical interaction of Ti with Sn (1–11).

Fig. 1. Variants of the phase diagram of the Ti—Sn system according to data of various authors:
a—(6), *b*—(7), *c*—(8), *d*—(9,10)

The data obtained by different authors differ sharply from one another, as a result of which four fundamentally different variants of equilibrium between α-Ti and the γ-phase have been proposed (Fig. 1, according to the data of (6–11)).

Analyzing the data of works (6–11) from the standpoint of the reliability of the experimental methods used, the purity of the starting materials, and the duration of homogenization of the alloys in order to bring them to an equilibrium state, one may conclude that, apparently, the alloy specimens closest to equilibrium were obtained by M. K. Macwillian (10), who annealed them at 880° for 5500 hours. In the other works the annealing time was considerably shorter. M. K. Macwillian considers that a continuous series of solid solutions is formed between α-Ti and Ti$_3$Sn.

It should be noted that α-Ti and Ti$_3$Sn possess similar crystal structures (4,5). The atomic radii of α-Ti and the γ-phase in the corresponding lattices for coordination number 12 differ by less than 8%, which satisfies the well-known rules of I. I. Kornilov (12) and Hume-Rothery (13).

The present work was undertaken for the final solution of the question concerning the nature of equilibrium in the Ti—Sn system between α-Ti and the γ-phase and for constructing the equilibrium phase diagram in the concentration range from 0 to 25 at.% Sn. In setting up the work, the experience and results of studies set forth in works (6–11) were taken into account.

Fig. 2. Dependence of electrical resistivity on temperature for Ti–Sn alloys.

Figure 2: Fig. 2. Dependence of electrical resistivity on temperature for Ti–Sn alloys.

A comparative evaluation of methods for preparing Ti-based alloys for the study of phase diagrams (14) shows that induction melting in the suspended state is the most favorable (15).

Therefore, this method was chosen for preparing the alloys.* Alloys were prepared containing (according to chemical-analysis data): 0.83, 2.12, 4.35, 5.28, 6.52, 7.78, 10.1, 14.9, 18.1, 22.0, and 25 at.% Sn.

* In all preceding studies, the alloys were prepared by melting the components in an arc furnace with a water-cooled copper hearth and a nonconsumable electrode.

Ti of 99.9% purity ($\sigma_{\mathrm{B}} = 37$ kg/mm^2) and Sn 99.99% were used as starting materials.

Microscopic analysis of the cast specimens showed that alloys with a content up to and including 14.9% Sn are single-phase. In the structure of alloys with 18.1 and 22.0% Sn, small interlayers of a second phase are present. To bring the alloys to equilibrium, after preliminary deformation by 10-20% they were annealed at 850° in a vacuum of 10^{-4} mm.

Microscopic analysis of the alloys after annealing for 1500 hours showed that the interlayers of the second phase in alloys with 18.1 and 22.0 at.% Sn had become

Fig. 2. Dependence of electrical resistivity on temperature for Ti–Sn alloys. Sn content in the alloy (in percent):
1 –pure Ti, 2 –0.82, 3 –2.12, 4 –4.35, 5 –5.28, 6 –6.50,
7 –7.78, 8 –10.1, 9 –12.0, 10 –14.9, 11 –18.1, 12 –22.0

considerably thinner. Alloys containing 4.35; 5.28; 6.52 and 7.78 at.% Sn had the structure α'. The remaining alloys had a polyhedral structure. In connection with this, all the alloys were again subjected to deformation by 10-20% and additionally annealed at 800° for 1200 hours.

The microstructure study carried out after this showed that all the alloys are single-phase and have a polyhedral $\alpha(\gamma)$ structure; this indicates the existence of a continuous series of solid solutions between α-Ti and the γ-phase. To determine the upper temperature boundary of existence of the $\alpha(\gamma)$-phase region, we investigated the temperature dependence of the specific electrical resistivity of the alloys ρ (16), which was measured by a contactless method (17) in a vacuum of 10^{-4} mm on the apparatus described in (18). In the interval 800-900°, measurements were made every 5-10°. The measurement results are given in Table 1.

For all alloys, ρ initially increases linearly with increasing temperature (for this temperature interval, Table 2 gives the values of α_t), and then the rate of increase of ρ with temperature slows. At the moment of the phase transformation, a sharp decrease in ρ is observed. The values of ρ at adjacent temperatures between which the phase transformation occurs are combined in Table 1 by brackets.

For alloys in which the transition $\alpha \rightarrow \beta$ occurs over a temperature interval, it is possible to indicate the temperature of transition from the $\alpha + \beta$ region into β, since $\rho(\beta)$ increases only weakly with increasing temperature, whereas $\rho(\alpha + \beta)$ drops sharply; this is evidently associated with the rate of the phase transformation, which in this case is large, since the temperature interval of existence of the $(\alpha + \beta)$ region in the alloys studied is very narrow.

Table 1

Electrical resistivity ρ of Ti–Sn alloys at various temperatures ($\mu\Omega \cdot$ cm)

T, °C	0	0,83	2,12	4,35	5,28	6,52	7,78	10,1	12,0	14,9	18,1	22,0	25,0
20	45,5	57,8	70,0	85,0	93,0	101,5	113,0	116,0	116,0	111,0	101,0	84,0	71,0
120	65,4	72,5	84,1	95,3	100,2	109,0	118,6	119,0	119,5	116,0	106,2	91,5	80,3
200	78,5	87,0	93,6	105,5	108,0	113,5	124,0	124,0	122,5	119,7	111,0	98,5	89,4
300	94,4	100,0	105,0	114,6	114,3	119,5	130,1	128,5	127,0	124,8	116,2	105,3	97,5
400	110,5	109,0	114,2	119,5	120,0	123,8	135,0	136,0	133,0	126,8	120,8	111,5	105,0
500	122,6	117,0	120,3	122,3	122,0	129,5	138,0	138,0	137,1	130,2	127,2	117,3	112,5
600	132,0	121,2	123,1	125,8	125,3	132,3	140,5	142,3	138,5	132,4	129,5	121,1	118,5
700	139,1	125,0	127,8	128,7	129,0	135,7	142,5	145,0	139,8	134,5	132,1	125,4	122,1
750	142,1	128,0	128,8	129,1	130,5	137,3	144,0	147,0	140,5	135,2	133,4	127,0	123,5
800	143,0	128,0	129,3	129,3	132,0	139,4	144,3	149,7	140,8	135,4	134,0	128,8	125,1
820	143,4	128,3	129,5	129,7	133,0	140,0	144,2	150,0	141,0	135,1	135,2	129,2	
830	143,2	128,1	129,5	129,7	133,5	140,0	144,5	150,3	141,2	135,6	135,4	129,5	
835	143,3	128,3	129,3	130,1	133,4	140,3	145,0	150,4	141,7	135,5	136,0	130,0	
840	143,2	128,0	129,2	130,3	134,0	140,5	145,1	150,5	142,0	135,3	136,1	130,3	
845	143,3	128,2	129,4	130,3	134,0	114,5	145,3	150,4	142,0	135,8	136,6	131,1	
850	143,5	128,2	129,7	115,1	115,0	114,3	125,5	151,0	142,3	136,0	137,0	131,4	128,3
860	143,5	129,0	117,5	110,8	108,5	114,6	117,5	140,1	142,8	136,6	137,2	131,0	
870	144,2	118,7	110,0	109,7	108,5	114,9	118,0	132,2	129,0	136,3	137,1	131,5	
880	144,3	112,5	108,0	109,5	108,8	114,8	119,0	127,0	124,0	136,4	137,5	131,5	
890	125,4	112,7	108,4	109,8	109,0	115,0	119,5	125,1	122,0	131,5	137,5	131,7	
900	125,5	113,0	108,5	110,2	109,3	115,2	120,1	125,2	120,3	127,2	137,8	132,0	
920	125,8	113,5	108,7	110,2	109,5	115,3	120,5	125,2	119,0	124,5	137,7	132,2	
950	125,6	114,0	109,1	111,4	110,0	116,0	121,3	125,5	119,0	120,8	126,1	132,1	
980	125,8	115,2	109,5	111,7	110,5	116,8	121,5	126,1	120,5	118,0	121,3	132,3	
1000	126,1	116,0	110,4	112,4	111,3	117,0	121,8	126,3	122,5	118,2	117,5	132,5	130,2
1050	126,3	116,9	111,0	113,0	112,0	118,1	122,3	127,0	123,1	119,0	117,0	118,0	

T, °C	0	0,83	2,12	4,35	5,28	6,52	7,78	10,1	12,0	14,9	18,1	22,0	25,0
1100	126,5	117,5	111,3	113,3	115,1	119,5	123,1	129,2	124,5	118,8	117,4	117,5	
1150	127,2	118,5	112,0	115,0	115,8	120,0	123,5	130,0	125,0	120,0	119,0	119,0	133,0

Table 2

Temperature coefficient of electrical resistivity and temperatures of phase transformations in Ti–Sn alloys

	0	0,83	2,12	4,35	5,28	6,52	7,78	10,1	12,0	14,9	18,1	22,0	25,0
Temperature interval, °C	20 – 400	20 – 300	20 – 300	20 – 250	20 – 250	20 – 200	20 – 200	20 – 200	20 – 200	20 – 200	20 – 200	20 – 200	20 – 200
α_t, $\mu\Omega\cdot$cm/deg	0,178	0,161	0,132	0,113	0,083	0,068	0,061	0,044	0,036	0,055	0,067	0,081	0,105
t of transition, °C: $\alpha \to \alpha+\beta$, $\alpha \to \beta$ or $\alpha \to \beta+\gamma$	882	865	855	847	845	842	845	850	860	880	925	1000	–

	0	0,83	2,12	4,35	5,28	6,52	7,78	10,1	12,0	14,9	18,1	22,0	25,0
t of transition, °C: $\alpha+\beta \to \beta$ or $\beta+\gamma \to \beta$	–	880	875	855	850	–	855	880	–	–	–	–	–

Figure 2 gives the dependences of ρ on t in the interval 800—1050°. From the sharp breaks in these curves, the temperatures of the phase transformations in the alloys studied were determined (see Table 2).

It is seen from Fig. 2 that, in alloys with 5.28 and 7.78 at.% Sn, the transition from the α region to β occurs over a temperature interval, whereas the alloy with 6.52 at.% Sn lying between them undergoes an isothermal transformation at 842°, or, in any case, the transformation interval is less than 5°, as evidenced by the abrupt change in ρ and the absence of intermediate points on the falling branch of the curve for the dependence of ρ on t.

Figure 3 gives the dependences of ρ at 20, 200, and 300° and of α_t on concentration. As is seen from Fig. 3, the dependences of ρ on composition are characterized by smooth curves with a maximum corresponding to $\sim$ 12% Sn. The concentration dependence of α_t is characterized by a smooth curve with a minimum, also corresponding to $\sim$ 12% Sn (Fig. 3b). In their character, the dependences of the indicated properties are typical for systems with a continuous series of solid solutions ([19]).

To refine the course of the boundaries between the phase regions β and $\beta+\gamma$, as well as $\beta+\gamma$ and γ, the alloys were annealed at 900, 1000, and 1100° for 200 h.

Microscopic analysis of samples quenched from the indicated temperatures showed that all alloys in the concentration range from 0.83 to 10.1% Sn have the structure α'; alloys with 12.0, 12.9, 18.1, and 22.0% Sn have the structure $\alpha'+\gamma$.

Determinations of the melting temperatures of the alloys, carried out using the method of G. A. Meerson (20), confirm the data of works (6–10).

On the basis of the experimental material obtained, an equilibrium version of

Fig. 3. Dependence of electrical resistivity (a) and the temperature coefficient of electrical resistivity (b) on the composition of Ti–Sn alloys

Figure 3: Fig. 3. Dependence of electrical resistivity (a) and the temperature coefficient of electrical resistivity (b) on the composition of Ti–Sn alloys

Fig. 4. Equilibrium version of the Ti–Sn phase diagram according to data obtained in the present work

Figure 4: Fig. 4. Equilibrium version of the Ti–Sn phase diagram according to data obtained in the present work

the Ti–Sn diagram was constructed, shown in Fig. 4. It follows from Fig. 4 that between α-Ti and the γ phase there exists a continuous series of solid solutions. Addition of Sn lowers the temperature of the allotropic transformation of Ti to a minimum at 842° and 6.52 at.% Sn, and then raises it again.

Fig. 3. Dependence of electrical resistivity (**a**) and the temperature coefficient of electrical resistivity (**b**) on the composition of Ti–Sn alloys

Fig. 4. Equilibrium version of the Ti–Sn phase diagram according to data obtained in the present work

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