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Experimental phase diagram of the Ti-Si-Sn ternary system at 473 K

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1. Introduction

Titanium and its alloys as important structural materials are widely used in many fields such as aerospace, automobile, and medical appliances, due to their low density, high specific strength and modulus, and corrosion resistance. As a new titanium-casting alloy, the Ti-Si eutectic alloy has a strengthening mechanism that greatly differs from the traditional titanium alloys. Si is important constituent of multicomponent Ti-alloys [1,2], because it can essentially increase the corrosion and creep resistance and hightemperature strength of both titanium and Ti-Al alloys. As a conventional alloving addition for Ti alloys, Sn can improve the quenchability and viscosity of β -Ti [3]. Despite this, multicomponent phase diagrams on the basis of Ti, Si and Sn are mostly unknown. Moreover, phase equilibria in the boundary Ti-Si-Sn ternary were reported only in Refs. [4,5], however, in a narrow concentration interval. Therefore, it is essential to investigate further the phase relationship of the Ti-Si-Sn ternary system for developing polynary cast titanium alloys with high performance.

According to Ref. [6], five compounds, i.e. Ti_3Si , Ti_5Si_3 , Ti_5Si_4 , TiSi and TiSi₂, were found in the Ti–Si binary system. In Ref. [7], the Si–Sn phase diagram was reported without compounds. The Ti–Sn binary phase diagram [8] shows four intermediate phases of Ti₃Sn, Ti₂Sn, Ti₅Sn₃ and Ti₆Sn₅. Yet, a new phase Ti₂Sn₃ has

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ABSTRACT

The phase diagram of the ternary system Ti–Si–Sn system at 473 K was investigated by means of powder X-ray diffraction (XRD), differential thermal analysis (DTA) and scanning electron microscope (SEM) with energy dispersive analysis (EDX). The isothermal section consists of 14 single phase regions, 26 binary phase regions and 13 ternary phase regions. The 10 binary compounds, namely Ti₃Si, Ti₅Si₃, Ti₅Si₄, TiSi, TiSi₂, Ti₃Sn, Ti₂Sn₃, Ti₆Sn₅, Ti₂Sn₃, have been confirmed at 473 K. Moreover, a ternary phase with the crystal structure of tetragonal W_5Si_3 structure type and I4/mcm space group is confirmed in the Ti–Si–Sn system. The combined results of both EDX and XRD show that the composition of this ternary phase is 60.25–61.03 at.% Ti, 15.01–21.77 at.% Si and balance Sn.

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been confirmed in the isothermal section of the ternary La–Ti–Sn and Cu–Ti–Sn systems at 473 K [9,10]. This binary compound was revealed for the first time by Kuper et al. [11] and its crystal has been identified by Künnen et al. [12]. Structural data for the intermetallic compounds in the three binary systems are given in Table 1.

The purpose of the present work is to investigate experimentally the Ti–Si–Sn phase diagram, mainly by assemble an isothermal section at 473 K, so as to provide essential information for the design and fabrication of new-type titanium alloys with high performance.

2. Experimental details

The present investigation was carried out with 100 samples having weight of 2 g. The purities of the starting materials, namely, titanium, silicon and tin were 99.8 wt.%, 99.99 wt.% and 99.99 wt.%, respectively. The alloy buttons were produced by arc melting on a water-cooled copper crucible with a non-consumable tungsten electrode under pure argon atmosphere. Each as-arc-cast button was melted three times and turned around after melting for better homogeneity. Since the alloys contain tin, the electric current was as low as possible so as to minimize the weight loss by volatilization of tin. For most alloys, the weight loss was less than 1% after melting.

The as-cast samples were sealed in an evacuated quartz tube for homogenization treatment and then annealed at different temperatures in order to attain good homogenization. The heat treatment temperature was determined on the basis of the binary alloy phase diagrams of the Ti–Si, Ti–Sn and Si–Sn systems [8–10] and the results of differential thermal analysis (DTA) of some typical samples. The binary samples that contain more than 75% Sn in the Si–Sn system or 45.5% Sn in the Ti–Sn system, as well as the ternary samples with Sn content higher than 60%, were initially annealed at 773 K for 720 h and then cooled down to 473 K at a rate of 0.15 K/min, then kept at 473 K for 240 h. Other samples were homogenised initially at 1073 K for 720 h, then cooled down to 473 K at a rate of 0.15 K/min, and finally kept at 473 K for 240 h. Finally, all these annealed buttons were quenched in liquid nitrogen.

X-ray powder diffraction (XRD) and scanning electron microscopy (SEM) with energy dispersive analysis (EDX) were used in the present investigation. Samples for

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|---------------|--|
| Binary crysta | l structure data in the Ti-Si-Sn system. |

| Compound | Space group | Lattice parameters (nm) | | | Reference |
|---------------------------------|----------------------|-------------------------|-----------|-----------|-----------|
| | | а | b | С | |
| Ti₃Si | P4 ₂ /n | 1.0196 | - | 0.5097 | [13] |
| Ti ₅ Si ₃ | P6 ₃ /mcm | 0.7461 | _ | 0.5151 | [13] |
| Ti ₅ Si ₄ | P41212 | 0.7133 | - | 1.2977 | [13] |
| TiSi | Pnma | 0.657 | 0.364 | 0.503 | [13] |
| TiSi ₂ | Fddd | 0.8036(6) | 0.4773(4) | 0.8523(6) | [13] |
| Ti₃Sn | P6 ₃ /mmc | 0.5916 | _ | 0.4764 | [14] |
| Ti ₂ Sn | P6 ₃ /mmc | 0.4653 | - | 0.569 | [14] |
| Ti ₅ Sn ₃ | P6 ₃ /mcm | 0.8049(2) | - | 0.5454(2) | [14] |
| Ti ₆ Sn ₅ | P6 ₃ /mmc | 0.922 | - | 0.569 | [14] |
| Ti ₂ Sn ₃ | Cmca | 0.596 | 1.994 | 0.702 | [12] |



Fig. 1. The XRD pattern of the sample with 54 at.% Ti, 6 at.% Si and 40 at.% Sn containing Ti_2Sn_3, Ti_6Sn_5 and $Ti_5Sn_3.$

XRD analysis were initially powdered and then measured with the help of Rigaku D/Max 2500V diffractometer with Cu K α radiation and graphite monochromator operated at 40 kV, 200 mA. The Materials Data Inc. software Jade 5.0 [15] and powder diffraction file (PDF release 2002) were used for phase identification. By all these means, the phases relation and the crystal structure of the Ti–Si–Sn ternary system were determined.

3. Results and discussions

3.1. Boundary binary systems

At 473 K, the binary compounds in the Ti–Si, Ti–Sn and Si–Sn binary systems have been confirmed. In the Ti–Sn system, a new stable phase Ti_2Sn_3 has been confirmed in the isothermal section of the ternary La–Ti–Sn and Cu–Ti–Sn systems at 473 K in the authors' previous publications [9,10]. In the present work, the XRD pattern of the equilibrated sample with (54 at.% Ti, 6 at.% Si and 40 at.% Sn)



Fig. 2. The XRD pattern of the sample with 50 at.% Ti, 10 at.% Si and 40 at.% Sn containing $Ti_2Sn_3,$ Sn and $Ti_5Sn_3.$



Fig. 3. The XRD pattern of the sample with 30 at.% Ti, 40 at.% Si and 30 at.% Sn containing $\rm TiSi_2, \rm TiSi$ and Sn.

composition also clearly indicates the existence of Ti_2Sn_3 besides Ti_6Sn_5 and Ti_5Sn_3 phases, as illustrated in Fig. 1. In addition, the XRD pattern of the equilibrated sample with 50 at.% Ti, 10 at.% Si and 40 at.% Sn clearly indicates the existence of three phases namely Ti_2Sn_3 . Sn and Ti_5Sn_3 in Fig. 2. Based on this experimental result, five binary compounds Ti_3Sn , Ti_2Sn , Ti_5Sn_3 , Ti_6Sn_5 and Ti_2Sn_3 have been observed in Ti–Sn system.

In the Ti–Si system, five phases i.e. Ti_3Si , Ti_5Si_3 , Ti_5Si_4 , TiSi and $TiSi_2$ have been confirmed. It agrees well with the results of Ref. [13]. For example, Fig. 3 illustrates the XRD pattern of the equilibrated sample (30 at.% Ti, 40 at.% Si and 30 at.% Sn) containing three phases of TiSi₂, TiSi and Sn, which clearly proves that the TiSi₂ and TiSi binary compounds exist in the present isothermal section.

According to the present work, no binary compound exists in the Si–Sn system, which is confirmed by the XRD results of some



Fig. 4. The XRD pattern of the sample with 10 at.% Ti, 60 at.% Si and 30 at.% Sn containing Sn, Si and TiSi_2.

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