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Phase equilibria of the Ti-Al-Nb system at 1400 °C

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ABSTRACT

Phase equilibria in the Ti-Al-Nb ternary system at 1400 °C was established, using scanning electron microscopy (SEM), electron backscattered diffraction (EBSD) and electron probe micro-analysis (EPMA). No ternary compound was observed in this work. Compared with the calculated results, the $\alpha + \beta + \gamma$ three-phase region moves to higher Nb and Al contents and the $\sigma + \beta + \gamma$ three-phase region moves to a lower Ti content in our experimental results. The tie-triangle of $\sigma + \delta + \beta$ moves to lower Ti and higher Nb contents. As a result, the homogeneity range of single α phase hugely expands to higher Nb and Al contents and the homogeneity range of single β phase region hugely expands to a higher Al content. The phase regions are also different with the previous reported experimental results.

1. Introduction

TiAl alloys are promising candidates to replace heavy Ni-based superalloys in the turbine blades of aircraft engines as well as in turbocharger wheels for advanced automobile engines due to their low density and high strength at elevated temperatures [1–6]. The γ-TiAl alloys with high Nb contents have higher strengths at elevated temperatures and excellent oxidation resistance [7,8]. The phase diagram of the Ti-Al-Nb ternary system is very important for alloy design and processing.

As most hot-working and heat treatments are carried out at temperatures between 1300 °C and 1400 °C [5,9], understanding the phase equilibria of the Ti-Al-Nb system at 1300 °C and 1400 °C is essential to alloy design and heat treatment processing. However, most research about the Ti-Al-Nb phase diagrams were focused on the temperatures lower than 1200 °C [10–30] or transformations for some special alloys [31–38], with very limited data for temperatures at 1400 °C, which leads to an incomplete understanding of the phase diagrams of the Ti-Al-Nb system. To the best of the authors' knowledge, there is only one systematic study at 1400 °C by Chen et al. in reference [20]. However Chen's work is not confirmed by other researchers and there are many differences between our present work and their work.

There has been some literature regarding the calculation of ternary or binary phase diagrams of Ti-Al-Nb system using the CALPHAD (CALculation of PHAse Diagrams) method [39–51]. The binary system Nb-Ti was modelled in Ref. [39]. At elevated temperatures, Nb and Ti form a continuous solid solution with the body-centered cubic crystal structure in the complete composition range, according to reference [39]. The Al-Nb binary system was re-assessed by Witusiewicz et al. in 2009 [50], and three compounds, i.e., δ-Nb_3Al, σ-Nb_2Al, and ε-$NbAl_3$, were considered. The Ti-Al system is complicated and crucial. Witusiewicz et al. re-assessed the Ti-Al binary system in 2008 [40], and the calculated phase diagrams are in good agreement with reported experimental results. Witusiewicz et al. [50] and Cupid et al. [51] independently modelled the Gibbs free energies of the phases in the Ti-Al-Nb system using the CALPHAD method in 2009. Fig. 1 shows the 1400 °C isothermal sections of the phase diagram calculated by Witusiewicz et al. [50] and Cupid et al. [51]. According to Witzsiewicz's results, the $\sigma + \delta + \beta$ three-phase field moves to higher Ti and lower Nb compositions than those in Cupid's results. The

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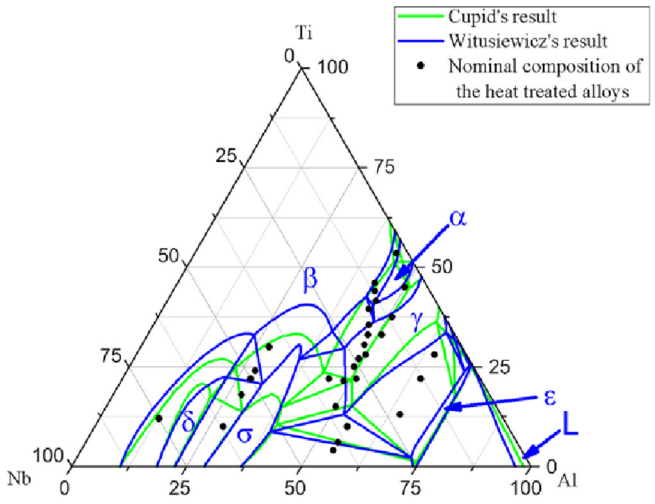

Fig. 1. 1400 °C isothermal sections of the Ti-Al-Nb system: calculated results of Witusiewicz et al. [50] and Cupid et al. [51], with the nominal compositions of the investigated alloys in the present work.

α + β + γ three-phase field from Witusiewicz et al. is located at a higher Nb content. The σ + β+ γ three-phase field from Witusiewicz et al. moves to higher Ti and lower Nb contents compared with Cupid's results. As a result, the σ and δ single phase region expands to lower Nb and higher Ti contents and the α single phase region expands to a higher Nb content compared with Cupid's results. Coincidentally, there is a good agreement in the location and compositions of the σ + γ + ε three-phase field from the both works. Those differences are caused by the limited experimental data at this temperature. Table 1 lists the phase designations most often used in the literature for the Al-Nb-Ti system, along with crystal structure data, based on reference [50].

In this paper, we present our experimental studies, using SEM, EBSD and EPMA, of phase equilibria in the Ti-Al-Nb system over the range of 0–70 at.% Al content at a temperature of 1400 °C. This research work is needed as a scientific base for optimizing the development of TiAl-based alloys and determining temperatures for heat treatment or hot working. The present results can also be used to re-optimize the Gibbs free energy descriptions of the phases in the Ti-Al-Nb system.

2. Experimental

Thirty ternary alloys, each with a mass of approximately 25 g, were prepared by non-consumable arc melting (tungsten electrode) of the high purity elements, i.e., Ti (99.99 wt.%), Al (99.99 wt.%), and Nb (99.99 wt.%), using a water cooled copper hearth in a gettered ultra-high purity argon atmosphere. The buttons were melted, turned over, and re-melted more than ten times to ensure compositional homogeneity. Only those samples with less than 1 wt% weight losses were used for phase analysis. Nominal compositions of these alloys were marked in Fig. 1 and listed in Table 2. Some 8 × 8 × 9 mm specimens were cut by electro-discharge machining (EDM) from the ingots and then sealed into different evacuated quartz capsules back-filled with Ar (99.999 V%) and with Ti (99.99 wt.%) fillings as getters for the removal of residual oxygen from the tube. All specimens were homogenized at 1400 °C for 15 h (see Table 2) in order to reach a good homogenization and then quenched in water. All heat treatments were performed in a laboratory muffle furnace with molybdenum disilicide heating elements ($MoSi_2$).

The surfaces of heat-treated sample ingots were removed more than 1 mm to avoid the reaction with the quartz capsule or oxidation during quenching, and then the samples were cut into halves. One of the halves was ground using a series of SiC papers ranging from #800, #1000, #1500, #2000 and #3000, followed by electrolytic polishing. The microstructures of the heat-treated specimens were examined using a ZEISS SUPRA 55 scanning electron microscope (SEM) in back-scattered electron (BSE) mode. The compositions of the heat-treated specimens were analyzed using a JXA-8230 electron probe microanalysis (EPMA). Phase constituents of each alloy could be speculated based on the compositions, which were characterized by the EPMA results. Electron backscatter diffraction (EBSD, which is also equipped on the SEM ZEISS SUPRA 55) analysis was performed to identify the phases of some alloys. Oxford Instruments HKL Channel 5 software was utilized to acquire and index the EBSD patterns. The phase identification in this work was based mainly on SEM, EPMA and EBSD results. The nominal compositions of the alloys investigated in this work and compositions of the individual phases in equilibrium at 1400 °C as determined by EPMA are given in Table 2. The composition of a given phase was established on the basis of at least ten EPMA measurement points, and the phase compositions given in this table are their average values.

Table 1
Phase designations most often used in the literature for the Al-Nb-Ti system, along with crystal structure data [50].

Phase (designation)	Pearson symbol	Space group	Strukturbericht designation	Prototype
(Al)(αAl), fcc_A1	cF4	Fm-3 *m*	A1	Cu
α, (αTi), hcp_A3	hp2	$P6_3/mmc$	A3	Mg
α_2, Ti_3Al	hP8	$P6_3/mmc$	$D0_{19}$	Ni_3Sn
β, (βTi), bcc_A2	cI2	Im-3 m	A2	W
β_0, bcc_B2	cI2	*Pm*-3 m	B2	CsCl
γ, γTiAl, TiAl	tP4	*P4/mmm*	$L1_0$	AuCu
δ, Nb_3Al	cP8	*Pm*-3n	A15	Cr_3Si
ε, $(Ti_{1-x}Nb_x)Al_3$, $TiAl_3$(h), $NbAl_3$	tI8	*I4/mmm*	$D0_{22}$	$TiAl_3$(h)
ε(l), $TiAl_3$(l)	tI32	*I4/mmm*	–	$TiAl_3$(l)
ζ, $Ti_{2+x}Al_{5-x}$	tP28	*P4/mmm*	–	Ti_2Al_5
η, $TiAl_2$	tI24	$I4_1/amd$	–	$HfGa_2$
σ, Nb_2Al	tP30	$P4_2/mnm$	$D8_b$	σCrFe
Ti_3Al_5	tP32	*P4/mbm*	–	Ti_3Al_5
O_1, O, O_1(h), Ti_2NbAl	oC16	*Cmcm*	–	NaHg
O_2, O_2(r), Ti_2NbAl	oC16	*Cmcm*	–	NaHg
τ, Ti4$NbAl_3$	hP6	$P6_3/mmc$	$B8_2$	Ni_2In
γ_1-$Ti_4Nb_3Al_9$	tP16	*P4/mmm*	–	γ_1-$Ti_4Nb_3Al_9$