



## Phase equilibria in Ni–Ti–Ta ternary system

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**Abstract:** Phase diagrams of two isothermal sections of the Ni–Ti–Ta ternary system at 1000 and 1200 °C in a full composition range were determined by X-ray diffraction and electron probe microanalysis. The experimental results indicated a ternary compound  $\tau$  phase with low solid solubility and composition ranges of (16.3–22.4) at.% Ta, (15.9–24.1) at.% Ti and (58.5–60.0) at.% Ni at 1000 °C. The two terminal solid solutions (bcc-(Ta) and  $\beta$ -Ti) formed a continuous solid solution at 1000 and 1200 °C. A certain amount of Ti can dissolve into Ni–Ta intermetallic compounds near the Ni–Ta side, with the highest value of 21.9 at.% observed in the  $\text{Ni}_2\text{Ta}$  compound at 1000 °C.

**Key words:** microstructure; Ni–Ti–Ta ternary system; isothermal section phase diagram; electron probe microanalysis

## 1 Introduction

Nickel-based superalloys have aerospace applications due to their excellent high-temperature properties and oxidation and corrosion resistance in extremely harsh environments [1,2]. However, to meet the high industrial requirements of structural materials used in high-temperature aviation applications, materials with greater mechanical strength and oxidation and corrosion resistances are required. To improve the properties of these alloys, an excellent method is to add alloy refractory elements [3–6]. For example, Ta addition can significantly improve the oxidation and hot-corrosion resistances of nickel-based alloys by forming a stable oxidation layer at elevated temperatures [4,7–9]. Meanwhile, as Ta is a solid-solution strengthening element, alloying with it can also improve the hot-corrosion and oxidation resistances [10,11]. However, topologically close-

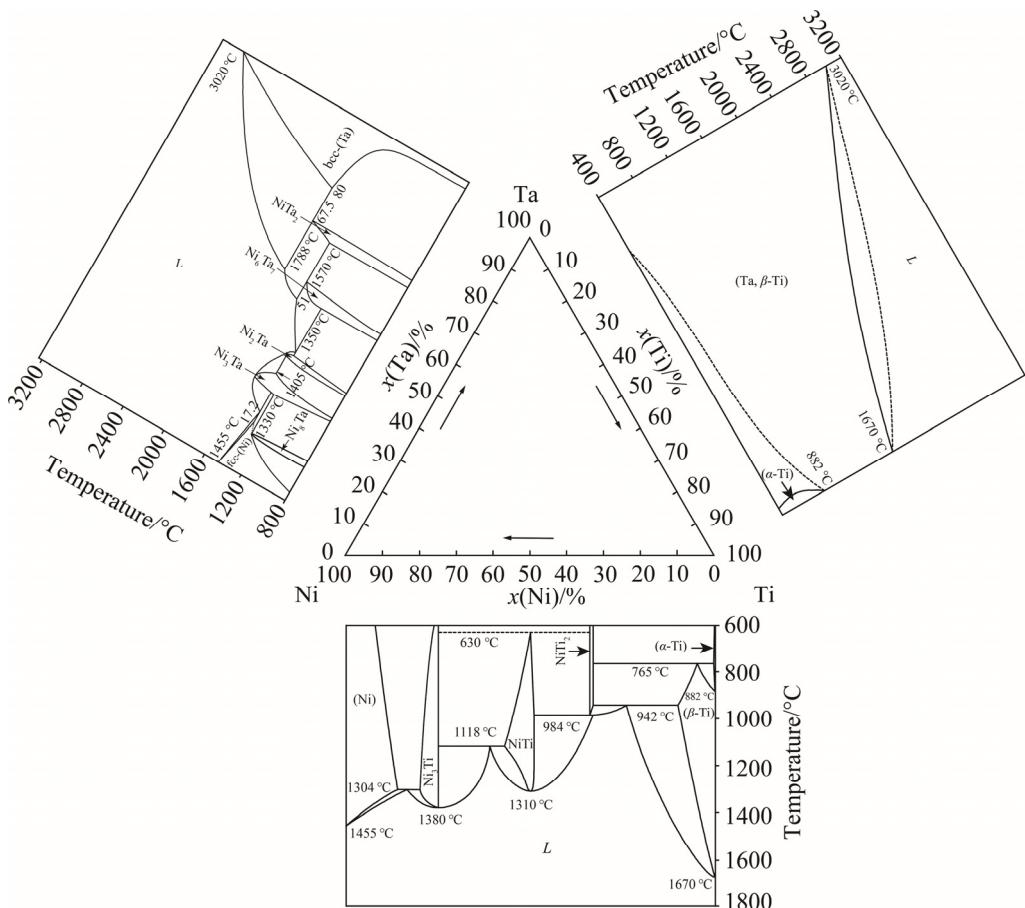
packed (TCP) phases will degrade the mechanical properties of superalloys with excessive additions of Ti and Ta elements [12,13]. Therefore, it is necessary to investigate a phase diagram of the ternary Ni–Ti–Ta system, not only for future thermodynamics assessment but also for enhancing its potential practical applications.

The Ni–Ti–Ta ternary system consists of three binary subsystems: Ni–Ti, Ni–Ta and Ti–Ta, as illustrated in Fig. 1 [14–16]. The Ni–Ta binary system has been investigated by many researchers [14,17–23]. In 2018, ZHOU et al [14] reviewed the Ni–Ta system, where only four intermetallic compounds of  $\text{Ni}_2\text{Ta}$ ,  $\text{Ni}_3\text{Ta}$ ,  $\text{NiTa}$  and  $\text{NiTa}_2$ , and two terminal solid solutions of fcc-(Ni) and bcc-(Ta) coexist. The  $\text{Ni}_8\text{Ta}$  phase was confirmed to be a metastable phase. Additionally, there are two eutectic reactions, three peritectic reactions, a peritectoid reaction and a congruent transformation in this system. Two eutectic reactions:  $L \rightleftharpoons \text{Ni}_3\text{Ta} + \text{fcc}$  and  $L \rightleftharpoons \text{Ni}_2\text{Ta} + \text{Ni}_6\text{Ta}_7$  occur at 1330 and

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**Fig. 1** Binary phase diagrams constituting Ni–Ti–Ta ternary system [14–16]

1350 °C, respectively. The  $\text{Ni}_6\text{Ta}_7$  and  $\text{NiTa}_2$  phases form from two peritectic reactions. At present, several investigators have assessed the thermodynamic database of this binary system with the CALPHAD method and first-principles calculations [17–19].

Ni–Ti binary phase diagrams have been reported by many researchers [24–29]. In 2009, KEYZER et al [15] reassessed the Ni–Ti binary phase diagrams and found them to be in good agreement with experimental results. The Ni–Ti binary system contains three intermetallic compounds of  $\text{Ni}_3\text{Ti}$ ,  $\text{NiTi}$  and  $\text{NiTi}_2$ , and three solid solutions of fcc-(Ni),  $\alpha$ -Ti and  $\beta$ -Ti. There are three eutectic reactions, one eutectoid reaction and one peritectic reaction in this system. The eutectic transformation of liquid phase (*L*) occurs at 1304 °C to form fcc-(Ni) and  $\text{Ni}_3\text{Ti}$ ; the eutectic transformation of *L* phase to  $\text{NiTi}$  and  $\text{Ni}_3\text{Ti}$  occurs at 1118 °C; the eutectic transformation of *L* phase to  $\text{NiTi}_2$  and  $\beta$ -Ti occurs at 942 °C. In this study, results of KEYZER et al [15] will be used. The Ti–Ta binary system is homogeneous at high

temperatures and a solid solution of  $\alpha$ -Ti appears at low temperature [16]. As for the Ni–Ti–Ta ternary system, GUPTA [29] summarized the existing research in 1991, but no information about the phase equilibrium of the Ni–Ti–Ta ternary system was found. In 2007, DU et al [30] investigated the phase equilibria in the Ni–Ti–Ta ternary system at 927 °C. The stable phases in the ternary Ni–Ti–Ta system are listed in Table 1.

In the compositional design and microstructural analysis of nickel-based superalloys, conducting phase equilibrium experiments with the Ni–Ti–Ta ternary system at high temperatures is essential. In the present work, we investigated the phase equilibria of Ni–Ti–Ta systems at 1000 and 1200 °C via electron probe microanalysis (EPMA) and X-ray diffraction (XRD).

## 2 Experimental

High-purity metals were used as raw materials to prepare the alloys: nickel (99.9 wt.%), titanium (99.9 wt.%) and tantalum (99.9 wt.%). All the

**Table 1** Stable solid phases in Ni–Ti–Ta ternary system

System	Phase	Pearson symbol	Protote	Space group	Strukturbericht	Ref.
	fcc-(Ni)	<i>cF</i> 4	Cu	<i>Fm</i> $\bar{3}m$	A1	[14]
	bcc-(Ta)	<i>cI</i> 2	W	<i>Im</i> $\bar{3}m$	A2	[14]
	Ni <sub>8</sub> Ta	<i>tI</i> 36	Ni <sub>8</sub> Nb	<i>I4/mmm</i>	–	[14]
	Ni <sub>3</sub> Ta	<i>tI</i> 8	TiAl <sub>3</sub>	<i>I4/mmm</i>	D0 <sub>22</sub>	[14]
Ni–Ta	Ni <sub>3</sub> Ta	<i>mP</i> 16	NbPt <sub>3</sub>	<i>P2</i> <sub>1</sub> / <i>m</i>	–	[14]
	Ni <sub>3</sub> Ta	<i>oP</i> 8	Cu <sub>3</sub> Ti	<i>Pmmm</i>	D0 <sub>a</sub>	[14]
	Ni <sub>2</sub> Ta	<i>tI</i> 6	MoSi <sub>2</sub>	<i>I4/mmm</i>	C11 <sub>b</sub>	[14]
	Ni <sub>6</sub> Ta <sub>7</sub>	<i>hP</i> 13	Fe <sub>7</sub> W <sub>6</sub>	<i>R</i> $\bar{3}m$	D8 <sub>5</sub>	[14]
	NiTa <sub>2</sub>	<i>tI</i> 12	Al <sub>2</sub> Cu	<i>I4/mcm</i>	C16	[14]
Ni–Ti	(Ni)	<i>cF</i> 4	Cu	<i>Fm</i> $\bar{3}m$	A1	[15]
	$\alpha$ -Ti	<i>hP</i> 2	Mg	<i>P</i> 63/ <i>mmc</i>	A3	[15]
	$\beta$ -Ti	<i>cI</i> 2	W	<i>Im</i> $\bar{3}m$	A2	[15]
	Ni <sub>3</sub> Ti	<i>hP</i> 16	Ni <sub>3</sub> Ti	<i>P</i> 63/ <i>mmc</i>	D0 <sub>24</sub>	[15]
	NiTi	<i>cP</i> 2	CsCl	<i>Pm</i> $\bar{3}m$	B2	[15]
	NiTi <sub>2</sub>	<i>cF</i> 96	NiTi <sub>2</sub>	<i>Fd</i> $\bar{3}m$	E9 <sub>3</sub>	[15]
Ti–Ta	(Ta, $\beta$ -Ti)	<i>cI</i> 2	W	<i>Im</i> $\bar{3}m$	A2	[16]

metals were well cleared to avoid surface oxidation impurities before melting. All alloys were prepared in the form of molar fractions (at.%). Ingots of around 20 g in mass were re-melted at least five times to obtain uniform alloys with mass loss less than 5% using an arc furnace in a high-purity argon atmosphere with a non-consumable tungsten electrode on a water-cooled copper platform. Then, the samples, except for the liquid phase that precipitated during heat treatment, were individually sealed in high-purity argon quartz tubes and annealed at 1000 and 1200 °C for 30 d. In order to prevent oxidation, some pure yttrium filings were placed in the quartz capsules. Additionally, alloys that precipitated liquid phase during heat treatment were placed separately in an alumina crucible to avoid contact with quartz, and then sealed in a quartz tube with high-purity argon gas. They were annealed at 1000 and 1200 °C for a much shorter duration of 3 h.

All the alloys were water-quenched after heat treatment and well prepared for metallographic analysis. The equilibrium compositions of all phases in the specimens were determined by electron probe microanalysis (EPMA) at an accelerating voltage of 20 kV and a probe current of  $1.0 \times 10^{-8}$  A. The crystal structure was identified by

its X-ray diffraction (XRD) pattern using a diffractometer (Phillips Panalytical X-pet) with Cu K $\alpha$  radiation at 40 kV and 40 mA. The results were measured in the  $2\theta$  range from 20° to 90° at intervals of 0.015308° and a count time of 0.3 s per step.

### 3 Results and discussion

#### 3.1 Microstructure

The phase relationship of the Ni–Ti–Ta ternary system at 1000 °C was established based on an analysis of 29 alloys that were annealed for 45 d. The nominal compositions and phase equilibrium compositions of the alloys are displayed in Table 2. Meanwhile, the microstructures and XRD patterns of typical annealed alloys are presented in Figs. 2 and 3, respectively.

Figure 2(a) shows the three-phase equilibrium of NiTi (black) + NiTa<sub>2</sub> (grey) + (Ta,  $\beta$ -Ti) (white), in the microstructure of the Ni<sub>36</sub>Ti<sub>32</sub>Ta<sub>32</sub> alloy. The corresponding XRD pattern is displayed in Fig. 3(a). Figure 2(b) shows the microstructure of the Ni<sub>57</sub>Ti<sub>21</sub>Ta<sub>22</sub> alloy, which contained three phases:  $\tau$  (grey) + Ni<sub>6</sub>Ta<sub>7</sub> (white) + NiTi (black). In the Ni<sub>74</sub>Ti<sub>10</sub>Ta<sub>16</sub> alloy, a three-phase region of Ni<sub>3</sub>Ta (grey) + Ni<sub>2</sub>Ta (white) + Ni<sub>3</sub>Ti (black) is confirmed

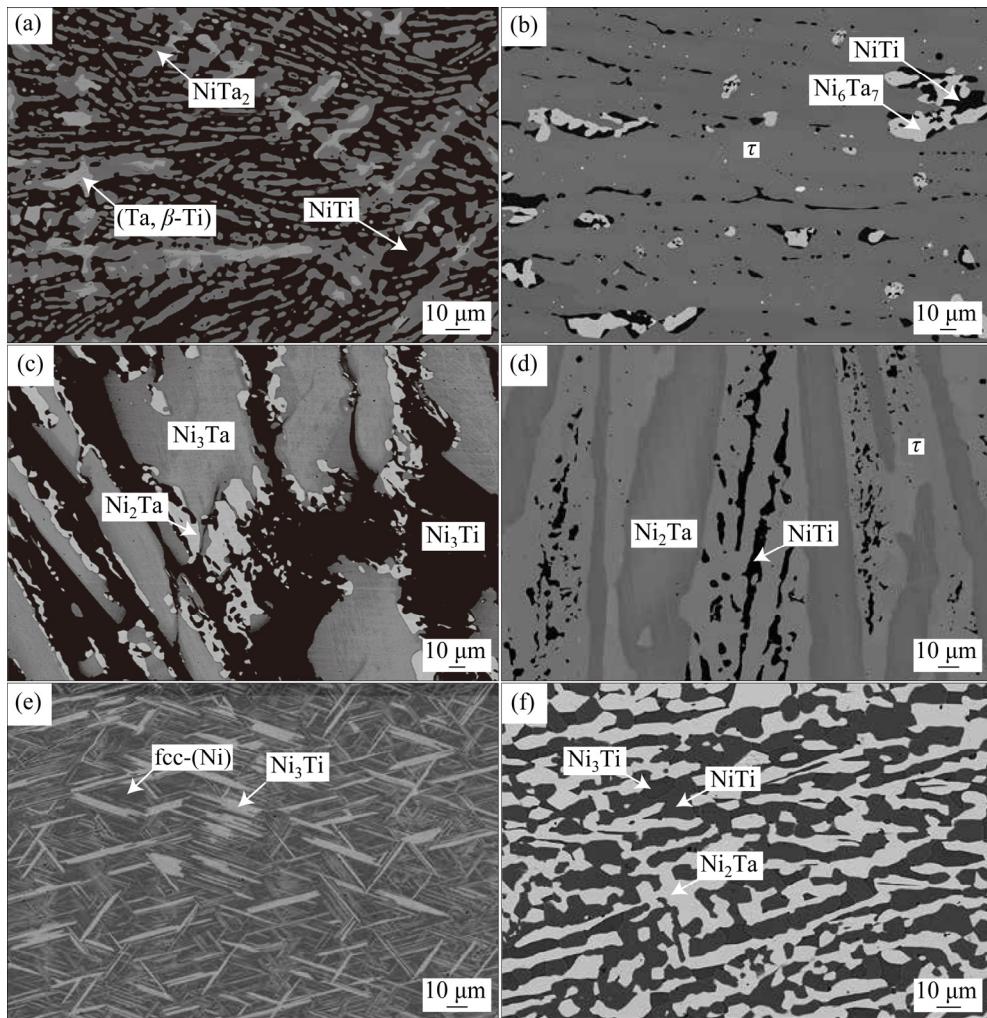
**Table 2** Phase equilibrium compositions of phases in Ni–Ti–Ta ternary alloys annealed at 1000 °C

Nominal alloy/at.%	Phase equilibrium			Composition/at.%						Annealing time/d	
	Phase 1	Phase 2	Phase 3	Phase 1		Phase 2		Phase 3			
				Ti	Ta	Ti	Ta	Ti	Ta		
Ni <sub>17</sub> Ti <sub>23</sub> Ta <sub>60</sub>	Ta, $\beta$ -Ti	NiTi		9.3	88.7	47.3	4.3			45	
Ni <sub>39</sub> Ti <sub>14</sub> Ta <sub>47</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	NiTi	NiTa <sub>2</sub>	10.7	45.6	41.5	8.8	7.1	57.8	45	
Ni <sub>56</sub> Ti <sub>12</sub> Ta <sub>32</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	Ni <sub>2</sub> Ta		8.9	40.6	11.7	20.9			45	
Ni <sub>36</sub> Ti <sub>32</sub> Ta <sub>32</sub>	NiTa <sub>2</sub>	NiTi	Ta, $\beta$ -Ti	13.2	51.2	46.4	4.3	8.2	88.2	45	
Ni <sub>19</sub> Ti <sub>46</sub> Ta <sub>35</sub>	<i>L</i>	Ta, $\beta$ -Ti		61.6	5.7	31.5	65.5			3/24	
Ni <sub>57</sub> Ti <sub>21</sub> Ta <sub>22</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	NiTi	$\tau$	14	36.5	37.8	9.7	20.6	20.1	45	
Ni <sub>51</sub> Ti <sub>26</sub> Ta <sub>23</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	NiTi		14.6	37.1	39.3	9.3			45	
Ni <sub>26</sub> Ti <sub>53</sub> Ta <sub>21</sub>	<i>L</i>	Ta, $\beta$ -Ti	NiTi	60.2	6.7	30	65.8	53.9	3.2	3/24	
Ni <sub>81</sub> Ti <sub>6</sub> Ta <sub>13</sub>	Ni <sub>3</sub> Ta	fcc-(Ni)	Ni <sub>3</sub> Ti	5	17.4	3.8	8.2	6.3	11.2	45	
Ni <sub>74</sub> Ti <sub>10</sub> Ta <sub>16</sub>	Ni <sub>3</sub> Ta	Ni <sub>2</sub> Ta	Ni <sub>3</sub> Ti	6.1	17.8	8.7	22.9	14.3	10.3	45	
Ni <sub>61</sub> Ti <sub>22</sub> Ta <sub>17</sub>	Ni <sub>2</sub> Ta	$\tau$	NiTi	16.4	16.3	24.1	16.4	39.4	7	45	
Ni <sub>55</sub> Ti <sub>36</sub> Ta <sub>9</sub>	Ni <sub>2</sub> Ta	NiTi		18.3	14.9	40.8	6.1			45	
Ni <sub>86</sub> Ti <sub>8</sub> Ta <sub>6</sub>	fcc-(Ni)	Ni <sub>3</sub> Ti		6.9	5.1	12	7			45	
Ni <sub>67</sub> Ti <sub>26</sub> Ta <sub>7</sub>	Ni <sub>2</sub> Ta	Ni <sub>3</sub> Ti	NiTi	21.9	11.3	23.5	2.5	41.8	3.5	45	
Ni <sub>15</sub> Ti <sub>71</sub> Ta <sub>14</sub>	<i>L</i>	Ta, $\beta$ -Ti		68.5	3.9	73.3	20.4			3/24	
Ni <sub>91</sub> Ti <sub>1</sub> Ta <sub>8</sub>	fcc-(Ni)			1.1	7.7					45	
Ni <sub>85.5</sub> Ti <sub>9.5</sub> Ta <sub>5</sub>	fcc-(Ni)	Ni <sub>3</sub> Ti		7.5	4	14.5	6.1			45	
Ni <sub>68</sub> Ti <sub>25</sub> Ta <sub>7</sub>	Ni <sub>2</sub> Ta	Ni <sub>3</sub> Ti	NiTi	22.2	11	23.2	2.5	42	3.4	45	
Ni <sub>61</sub> Ti <sub>14</sub> Ta <sub>25</sub>	Ni <sub>2</sub> Ta	$\tau$	Ni <sub>6</sub> Ta <sub>7</sub>	10.3	22.4	15.9	24.7	9.9	40.1	45	
Ni <sub>37</sub> Ti <sub>50</sub> Ta <sub>13</sub>	<i>L</i>	Ta, $\beta$ -Ti	NiTi	60.5	6.5	30.1	66	54.1	3.3	3/24	
Ni <sub>24</sub> Ti <sub>63</sub> Ta <sub>13</sub>	<i>L</i>	Ta, $\beta$ -Ti		65.5	5.3	54.4	43.1			3/24	
Ni <sub>44</sub> Ti <sub>19</sub> Ta <sub>37</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	NiTi		13.8	41.7	41.7	8.1			45	
Ni <sub>44</sub> Ti <sub>16</sub> Ta <sub>40</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	NiTi	NiTa <sub>2</sub>	14.5	41.3	41.2	9.1	7.5	57.4	45	
Ni <sub>60</sub> Ti <sub>13</sub> Ta <sub>27</sub>	Ni <sub>2</sub> Ta	$\tau$	Ni <sub>6</sub> Ta <sub>7</sub>	9.5	24	15.9	26.3	9.2	40.5	45	
Ni <sub>84</sub> Ti <sub>14</sub> Ta <sub>2</sub>	fcc-(Ni)	Ni <sub>3</sub> Ti		10.2	1.1	16.9	3.1			45	
Ni <sub>63</sub> Ti <sub>24</sub> Ta <sub>13</sub>	Ni <sub>2</sub> Ta	NiTi		18.5	14.5	41.7	5.8			45	
Ni <sub>56</sub> Ti <sub>31</sub> Ta <sub>13</sub>	$\tau$	NiTi		23.5	18	41	7.4			45	
Ni <sub>41</sub> Ti <sub>39</sub> Ta <sub>20</sub>	Ta, $\beta$ -Ti	NiTi		9.3	88.7	47.3	4.3			45	
Ni <sub>32</sub> Ti <sub>18</sub> Ta <sub>50</sub>	NiTa <sub>2</sub>	NiTi	Ta, $\beta$ -Ti	13.5	50.8	46.1	4.5	8.5	88	45	

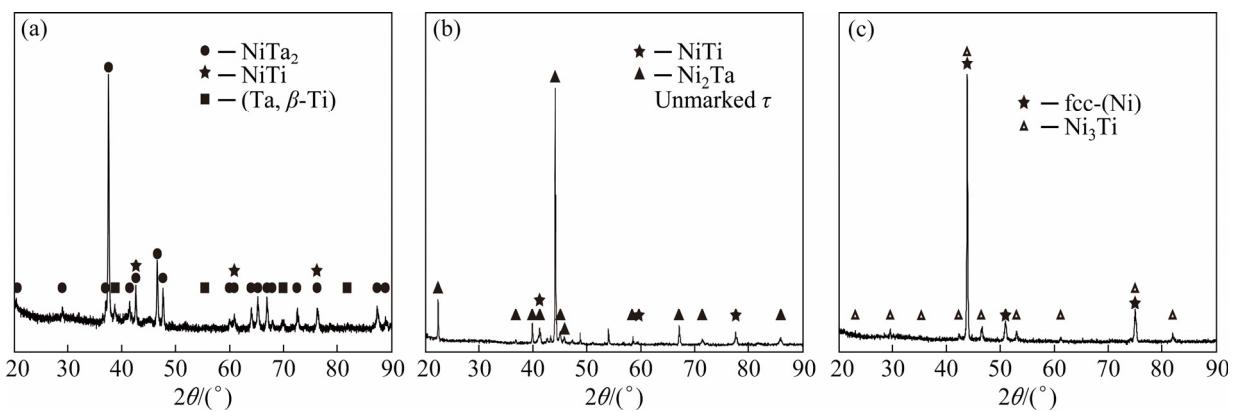
in Fig. 2(c). Figure 2(d) features the microstructure of the Ni<sub>61</sub>Ti<sub>22</sub>Ta<sub>17</sub> alloy, which consisted of Ni<sub>2</sub>Ta (grey) + NiTi (black) +  $\tau$  (white), and the XRD analysis in Fig. 3(b) confirms the three-phase microstructure. As described in Fig. 2(e), a two-phase equilibrium of fcc-(Ni) (black) + Ni<sub>3</sub>Ti (white) was identified in the Ni<sub>86</sub>Ti<sub>8</sub>Ta<sub>6</sub> alloy. The corresponding phase relationship was identified by the XRD results, in which the characteristic peaks

of the fcc-(Ni) and Ni<sub>3</sub>Ti phases are labelled with different symbols in Fig. 3(c). A three-phase region of Ni<sub>3</sub>Ti (black) + NiTi (dark-grey) + Ni<sub>2</sub>Ta (white) was found in the Ni<sub>68</sub>Ti<sub>25</sub>Ta<sub>7</sub> alloy, as shown in Fig. 2(f).

Twenty-three alloys were designed to investigate the phase relation of the Ni–Ti–Ta system at 1200 °C. Table 3 lists the nominal compositions and phase equilibrium compositions



**Fig. 2** Microstructures of typical alloys in Ni-Ti-Ta system annealed at 1000 °C for 45 d: (a) Ni<sub>36</sub>Ti<sub>32</sub>Ta<sub>32</sub>; (b) Ni<sub>57</sub>Ti<sub>21</sub>Ta<sub>22</sub>; (c) Ni<sub>74</sub>Ti<sub>10</sub>Ta<sub>16</sub>; (d) Ni<sub>61</sub>Ti<sub>22</sub>Ta<sub>17</sub>; (e) Ni<sub>86</sub>Ti<sub>8</sub>Ta<sub>6</sub>; (f) Ni<sub>68</sub>Ti<sub>25</sub>Ta<sub>7</sub>



**Fig. 3** XRD patterns of typical alloys in Ni-Ti-Ta system annealed at 1000 °C for 45 d: (a) Ni<sub>36</sub>Ti<sub>32</sub>Ta<sub>32</sub>; (b) Ni<sub>61</sub>Ti<sub>22</sub>Ta<sub>17</sub>; (c) Ni<sub>86</sub>Ti<sub>8</sub>Ta<sub>6</sub>

of the alloys. Additionally, the microstructures and XRD patterns of typical alloys are presented in Figs. 4 and 5, respectively. As presented in Fig. 4(a), the microstructure of a three-phase region of NiTa<sub>2</sub>

(grey) + Ni<sub>6</sub>Ta<sub>7</sub> (white) + L (black) was identified in the Ni<sub>36</sub>Ti<sub>32</sub>Ta<sub>32</sub> alloy and the corresponding XRD pattern is displayed in Fig. 5(a). Figure 4(b) shows the two-phase microstructure of L (black) +

**Table 3** Phase equilibrium compositions of phases in Ni–Ti–Ta ternary alloys annealed at 1200 °C

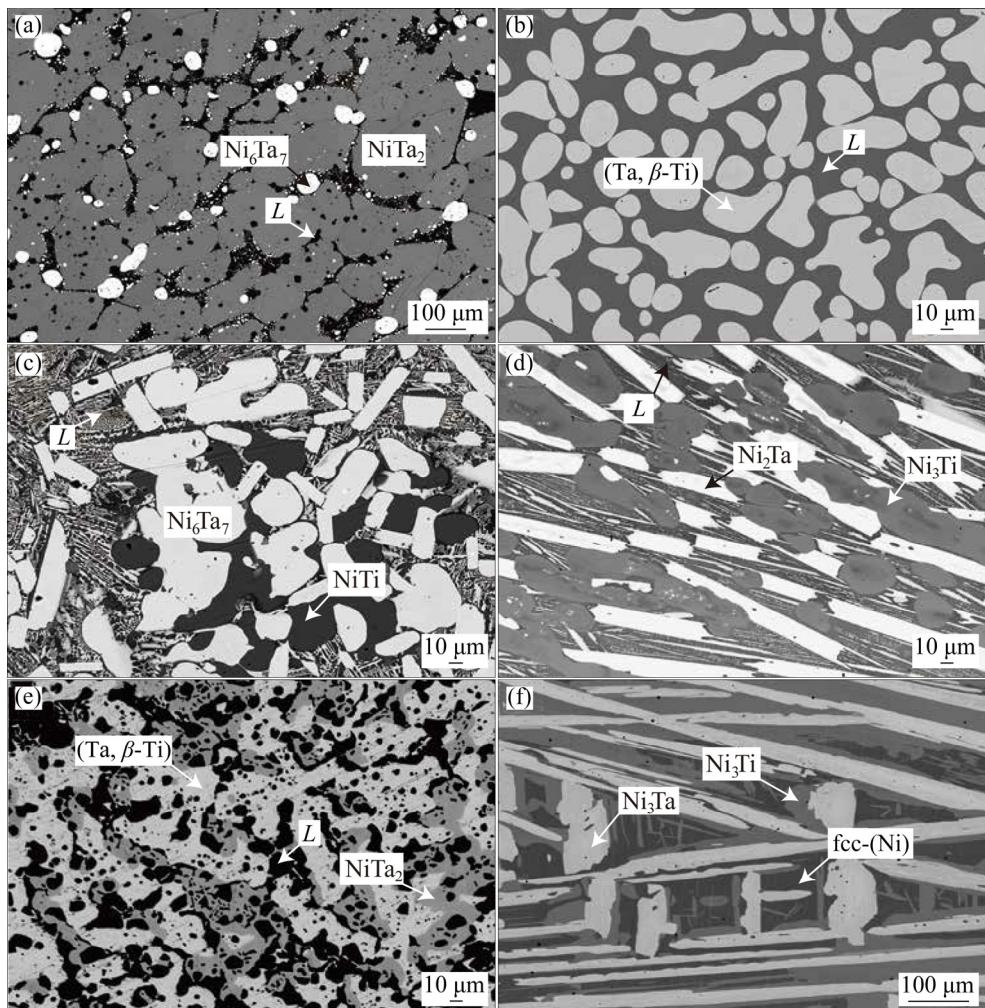
Nominal alloy/at.%	Phase equilibrium			Composition/at.%						Annealing time/d	
	Phase 1	Phase 2	Phase 3	Phase 1		Phase 2		Phase 3			
				Ti	Ta	Ti	Ta	Ti	Ta		
Ni <sub>17</sub> Ti <sub>23</sub> Ta <sub>60</sub>	Ta, $\beta$ -Ti	<i>L</i>	NiTa <sub>2</sub>	6.6	92.5	52.2	16.3	9.7	57.1	3/24	
Ni <sub>39</sub> Ti <sub>14</sub> Ta <sub>47</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	<i>L</i>	NiTa <sub>2</sub>	11	46.6	52.2	15.2	5.7	60.7	3/24	
Ni <sub>56</sub> Ti <sub>12</sub> Ta <sub>32</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	Ni <sub>2</sub> Ta		9.1	42.4	11.3	22.3			30	
Ni <sub>36</sub> Ti <sub>32</sub> Ta <sub>32</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	<i>L</i>	NiTa <sub>2</sub>	10.8	46.2	52	15.3	5.9	60.5	3/24	
Ni <sub>19</sub> Ti <sub>46</sub> Ta <sub>35</sub>	<i>L</i>	Ta, $\beta$ -Ti		58.8	7.4	29.3	69.3			3/24	
Ni <sub>57</sub> Ti <sub>21</sub> Ta <sub>22</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	$\tau$	<i>L</i>	9.3	40.8	18.4	22	31.8	13.6	3/24	
Ni <sub>51</sub> Ti <sub>26</sub> Ta <sub>23</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	<i>L</i>		13.2	40.2	37.3	13.1			3/24	
Ni <sub>26</sub> Ti <sub>53</sub> Ta <sub>21</sub>	<i>L</i>	Ta, $\beta$ -Ti		58.2	8.3	28.7	69.9			3/24	
Ni <sub>81</sub> Ti <sub>6</sub> Ta <sub>13</sub>	Ni <sub>3</sub> Ta	fcc-(Ni)	Ni <sub>3</sub> Ti	5.7	17.1	5.8	7.9	8.9	9.5	30	
Ni <sub>74</sub> Ti <sub>10</sub> Ta <sub>16</sub>	Ni <sub>3</sub> Ta	Ni <sub>2</sub> Ta	Ni <sub>3</sub> Ti	6.7	16.5	13.3	17.6	11	11.5	30	
Ni <sub>86</sub> Ti <sub>8</sub> Ta <sub>6</sub>	fcc-(Ni)	Ni <sub>3</sub> Ti		8.4	5.2	12.9	6.5			30	
Ni <sub>67</sub> Ti <sub>26</sub> Ta <sub>7</sub>	Ni <sub>2</sub> Ta	Ni <sub>3</sub> Ti	<i>L</i>	20.1	13.3	21.1	4.4	32.2	4.5	3/24	
Ni <sub>15</sub> Ti <sub>71</sub> Ta <sub>14</sub>	<i>L</i>	Ta, $\beta$ -Ti		68.5	3.9	73.3	20.4			3/24	
Ni <sub>91</sub> Ti <sub>1</sub> Ta <sub>8</sub>	fcc-(Ni)			1.1	7.8					30	
Ni <sub>85.5</sub> Ti <sub>9.5</sub> Ta <sub>5</sub>	fcc-(Ni)			9.1	4.6					30	
Ni <sub>68</sub> Ti <sub>25</sub> Ta <sub>7</sub>	Ni <sub>2</sub> Ta	Ni <sub>3</sub> Ti	<i>L</i>	20.3	13.1	21	4.6	32.1	4.7	3 h	
Ni <sub>61</sub> Ti <sub>14</sub> Ta <sub>25</sub>	Ni <sub>2</sub> Ta	Ni <sub>6</sub> Ta <sub>7</sub>		11.4	21.4	9.3	41.4			30	
Ni <sub>37</sub> Ti <sub>50</sub> Ta <sub>13</sub>	NiTi	<i>L</i>		47.3	4.9	52.3	9.9			3/24	
Ni <sub>24</sub> Ti <sub>63</sub> Ta <sub>13</sub>	<i>L</i>	Ta, $\beta$ -Ti		65.5	5.3	54.4	43.1			3/24	
Ni <sub>60</sub> Ti <sub>13</sub> Ta <sub>27</sub>	Ni <sub>2</sub> Ta	Ni <sub>6</sub> Ta <sub>7</sub>	$\tau$	11	22	9.5	42.4	14.9	24.3	30	
Ni <sub>84</sub> Ti <sub>14</sub> Ta <sub>2</sub>	fcc-(Ni)	Ni <sub>3</sub> Ti		11.9	2.2	18.7	3.4			30	
Ni <sub>63</sub> Ti <sub>24</sub> Ta <sub>13</sub>	Ni <sub>2</sub> Ta	<i>L</i>		18.2	16.3	31.9	10.1			3/24	
Ni <sub>41</sub> Ti <sub>39</sub> Ta <sub>20</sub>	Ni <sub>6</sub> Ta <sub>7</sub>	NiTi	<i>L</i>	19.1	39.8	46.1	6	51.8	12.3	3/24	

(Ta,  $\beta$ -Ti) (white) in the Ni<sub>19</sub>Ti<sub>46</sub>Ta<sub>35</sub> alloy. Figure 4(c) displays a three-phase section of *L* (grey) + Ni<sub>6</sub>Ta<sub>7</sub> (white) + NiTi (black) in the Ni<sub>41</sub>Ti<sub>39</sub>Ta<sub>20</sub> alloy. The grey area composed of black and white phases in Fig. 4(c) is a liquid phase region at high temperature. However, after quenching, the liquid phase may undergo a phase transition, resulting in a new white phase. As shown in Fig. 4(d), the microstructure of the Ni<sub>67</sub>Ti<sub>26</sub>Ta<sub>7</sub> alloy was determined as *L* (black) + Ni<sub>2</sub>Ta (white) + Ni<sub>3</sub>Ti (grey). In the Ni<sub>17</sub>Ti<sub>23</sub>Ta<sub>60</sub> alloy, a three-phase equilibrium of (Ta,  $\beta$ -Ti) (white) + *L* (black) + NiTi<sub>2</sub> (grey) was identified, as shown in Fig. 4(e). The XRD analysis in Fig. 5(b) supports the microstructure of this alloy. In the Ni<sub>81</sub>Ti<sub>6</sub>Ta<sub>13</sub> alloy, a three-phase region of Ni<sub>3</sub>Ta (white) + Ni<sub>3</sub>Ti

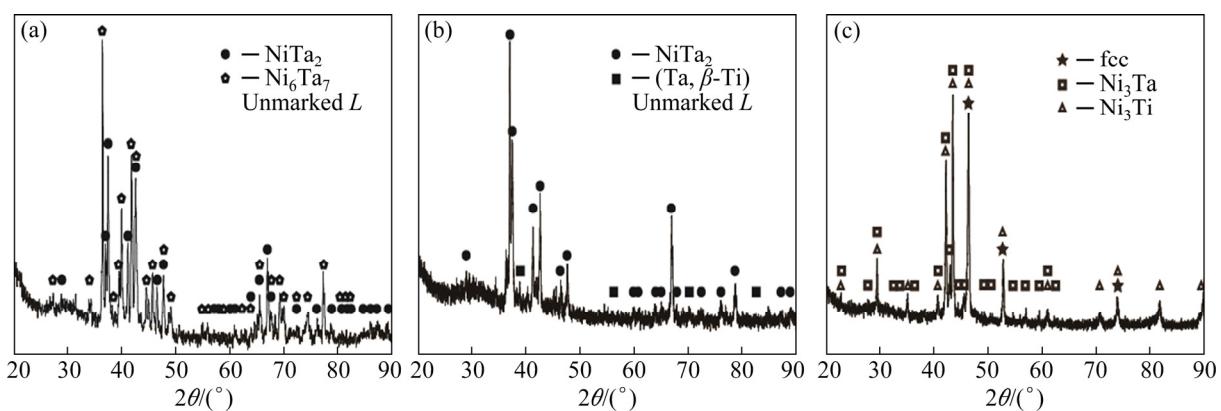
(grey) + fcc-(Ni) (black) was confirmed in Fig. 4(f) and the corresponding XRD pattern is displayed in Fig. 5(c).

### 3.2 Isothermal sections

According to the experimental results, the isothermal sections of the Ni–Ti–Ta ternary system at 1000 and 1200 °C were established and shown in Figs. 6 and 7, respectively. At 1000 °C, nine three-phase regions, ((Ta,  $\beta$ -Ti) + NiTa<sub>2</sub> + NiTi), (Ni<sub>6</sub>Ta<sub>7</sub> + NiTa<sub>2</sub> + NiTi), (Ni<sub>6</sub>Ta<sub>7</sub> + NiTa<sub>2</sub> + Ni<sub>3</sub>Ti), (Ni<sub>6</sub>Ta<sub>7</sub> + Ni<sub>2</sub>Ta +  $\tau$ ), (Ni<sub>3</sub>Ti + Ni<sub>2</sub>Ta + Ni<sub>3</sub>Ta), (Ni<sub>3</sub>Ti + fcc-(Ni) + Ni<sub>3</sub>Ta), (Ni<sub>3</sub>Ti + Ni<sub>2</sub>Ta + NiTi), ( $\tau$  + Ni<sub>2</sub>Ta + NiTi), (Ni<sub>6</sub>Ta<sub>7</sub> + NiTi +  $\tau$ ) and ((Ta,  $\beta$ -Ti) + *L* + NiTi), were experimentally determined, which are marked as triangles with solid lines. There is a three-phase region (Ni<sub>8</sub>Ta +



**Fig. 4** Microstructures of typical alloys in Ni-Ta-Ti ternary system annealed at 1200 °C for 3 h (a–e) and 30 d (f): (a) Ni<sub>36</sub>Ti<sub>32</sub>Ta<sub>32</sub>; (b) Ni<sub>19</sub>Ti<sub>46</sub>Ta<sub>35</sub>; (c) Ni<sub>41</sub>Ti<sub>39</sub>Ta<sub>20</sub>; (d) Ni<sub>67</sub>Ti<sub>26</sub>Ta<sub>7</sub>; (e) Ni<sub>17</sub>Ti<sub>23</sub>Ta<sub>60</sub>; (f) Ni<sub>81</sub>Ti<sub>6</sub>Ta<sub>13</sub>



**Fig. 5** XRD patterns of typical alloys in Ni-Ta-Ti system annealed at 1200 °C for 3 h (a, b) and 30 d (c): (a) Ni<sub>36</sub>Ti<sub>32</sub>Ta<sub>32</sub>; (b) Ni<sub>17</sub>Ti<sub>23</sub>Ta<sub>60</sub>; (c) Ni<sub>81</sub>Ti<sub>6</sub>Ta<sub>13</sub>

fcc-(Ni) + Ni<sub>3</sub>Ta) that has not been measured experimentally and is represented by a dotted triangle. The corresponding results show that a ternary compound  $\tau$  phase was detected with a

small composition range of (58.5–60.0) at.% Ni, (15.9–24.1) at.% Ti and (16.3–22.4) at.% Ta at 1000 °C. Two body centered cubic terminal solid solutions (Ta) and  $\beta$ -Ti formed a continuous phase

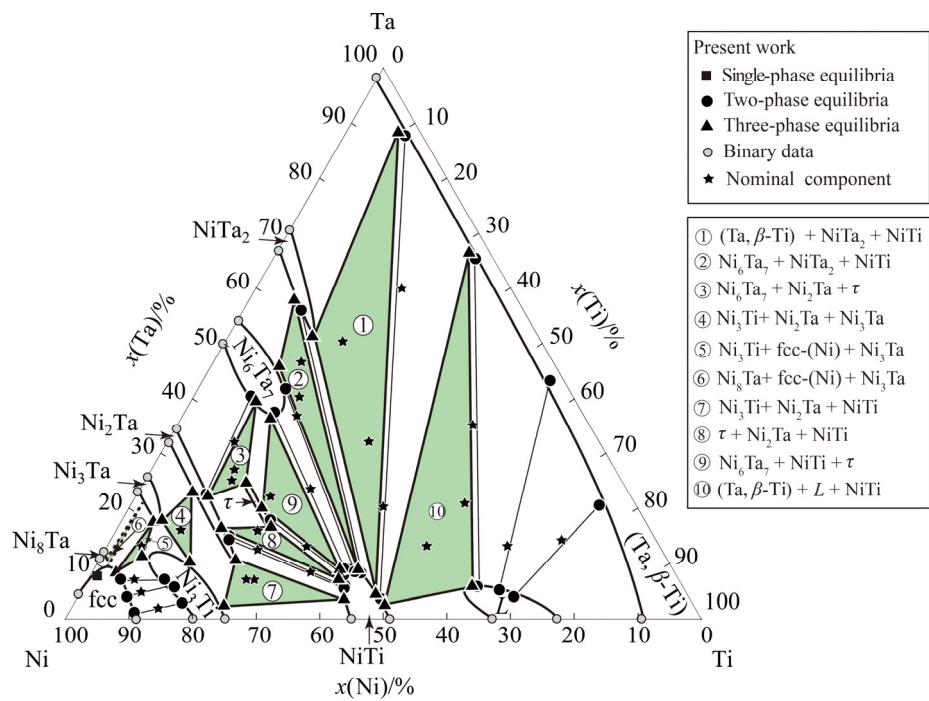


Fig. 6 Experimentally determined isothermal section of Ni–Ti–Ta system at 1000 °C

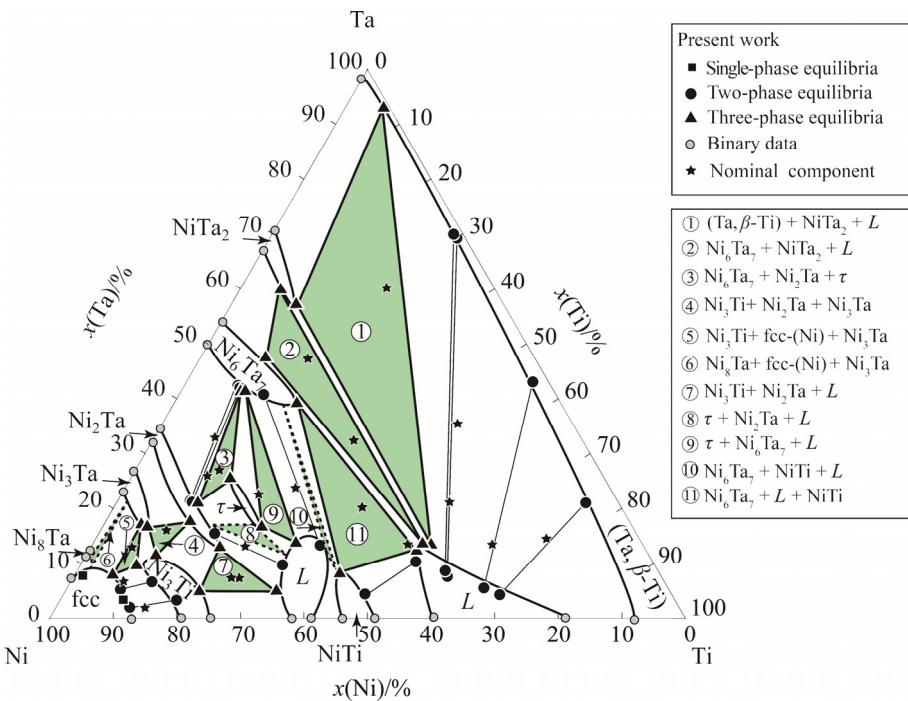


Fig. 7 Experimentally determined isothermal section of Ni–Ti–Ta system at 1200 °C

(Ta,  $\beta$ -Ti). The solubilities of Ta in the fcc-(Ni),  $\text{Ni}_3\text{Ti}$  and  $\text{NiTi}$  phases were about 9.0 at.%, 12.0 at.% and 9.7 at.%, respectively. Meanwhile, the  $\text{Ni}_3\text{Ta}$ ,  $\text{Ni}_2\text{Ta}$ ,  $\text{Ni}_6\text{Ta}_7$  and  $\text{NiTa}_2$  were dissolved at about 6.1 at.%, 21.9 at.%, 14.6 at.% and 13.5 at.% Ti, respectively.

Eight three-phase regions,  $(\text{Ta}, \beta\text{-Ti}) + \text{NiTa}_2 +$

$L$ ),  $(\text{Ni}_6\text{Ta}_7 + \text{NiTa}_2 + L)$ ,  $(\text{Ni}_6\text{Ta}_7 + \text{Ni}_2\text{Ta} + \tau)$ ,  $(\text{Ni}_3\text{Ti} + \text{Ni}_2\text{Ta} + \text{Ni}_3\text{Ta})$ ,  $(\text{Ni}_3\text{Ti} + \text{fcc-(Ni)} + \text{Ni}_3\text{Ta})$ ,  $(\text{Ni}_3\text{Ti} + \text{Ni}_2\text{Ta} + L)$ ,  $(\tau + \text{Ni}_6\text{Ta}_7 + L)$  and  $(\text{Ni}_6\text{Ta}_7 + \text{NiTi} + L)$ , were determined in the isothermal section of the Ni–Ti–Ta ternary system at 1200 °C. However, the remaining three three-phase equilibria of  $(\text{Ni}_8\text{Ta} + \text{fcc-(Ni)} + \text{Ni}_3\text{Ta})$ ,  $(\tau + \text{Ni}_2\text{Ta} + L)$

and  $(\text{Ni}_6\text{Ta}_7 + \text{NiTi} + L)$  were inferred. The experimental results show that the ternary compound  $\tau$  phase with almost the same composition range still existed at 1200 °C. Besides, with increasing temperature, a liquid phase presented within (38–41) at.% Ti.

## 4 Conclusions

(1) The isothermal sections of the Ni–Ti–Ta ternary system at 1000 and 1200 °C in the full composition range were experimentally established. Ten and eleven three-phase equilibria exist in this ternary system at 1000 and 1200 °C, respectively.

(2) A ternary compound  $\tau$  phase with almost the same small solubility was determined at both temperatures. The composition ranges of  $\tau$  phase were (58.5–60.0) at.% Ni, (15.9–24.1) at.% Ti and (16.3–22.4) at.% Ta at 1000 °C.

(3) The homogeneity ranges of binary compounds were measured and the phase relations among them were determined. The solid solubilities of Ti in Ni–Ta binary compounds and of Ta in Ni–Ti binary compounds were not large, but  $\text{Ni}_2\text{Ta}$  dissolved at 21.9 at.% Ti at 1000 °C.

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## Ni–Ti–Ta 三元体系的相平衡关系

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**摘要:** 采用 X 射线衍射和电子探针显微分析方法, 获得 Ni–Ti–Ta 三元体系在 1000 和 1200 °C 的全成分范围内等温截面相图。实验结果显示, Ni–Ti–Ta 三元体系的 1000 和 1200 °C 等温截面相图中均存在一个固溶度较小的三元化合物  $\tau$  相, 且通过相平衡可知, 该相在 1000 °C 的成分为(16.3~22.4)% Ta, (15.9~24.1)% Ti 和(58.5~60.0)% Ni(摩尔分数)。此外, 在这两个等温截面相图中, bcc-(Ta) 和  $\beta$ -Ti 均形成连续的固溶体。Ti 在 NiTa 二元化合物中均有一定的固溶度, 其中, 1000 °C 时 Ti 在 Ni<sub>2</sub>Ta 中的固溶度达 21.9%(摩尔分数)。

**关键词:** 显微组织; Ni–Ti–Ta 三元体系; 等温截面相图; 电子探针显微分析

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