



Article Phase Equilibria in the Ni-V-Ta Ternary System

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Abstract: Two isothermal sections of the Ni-V-Ta ternary system at 1200 °C and 1000 °C have been experimentally established using X-ray diffraction, electron probe microanalysis, energy dispersive spectroscopy, and differential scanning calorimeter with equilibrated key alloys. The equilibrium composition of each phase is measured by electron probe microanalysis and energy dispersive spectroscopy. The results indicate that: (1) nine three-phase and eight three-phase regions were confirmed in the isothermal section at 1200 °C and 1000 °C, respectively; (2) the C14 phase with a large solubility of about 51.2 at. % Ni was observed at 1200 °C, while it forms a single-phase region at 1000 °C; (3) a small liquid region was confirmed at 1200 °C, but disappeared at 1000 °C.

Keywords: Ni-V-Ta system; phase equilibrium; liquid section; electron probe microanalysis

1. Introduction

Nickel-based superalloys have become unique materials used in gas turbine engines, due to their excellent mechanical properties, oxidation, and creep resistance at elevated temperatures [1]. With increasing industry requirements for better high-temperature strength materials, there is an urgent need to improve the high-temperature properties of these superalloys. Many studies have been carried out to confirm the fact that a small addition of refractory elements (e.g., Re, Ru and Ta) can improve the high-temperature strength [1-3]. Besides, in 1981, Yang [4] found that a trace addition of 1 at. % Ta in complex nickel-based alloys improved the oxidation resistance significantly. Recently, Park et al. [5] have found that Ta exerted a beneficial effect on oxidation resistance in nickel-based superalloys. Additionally, the Ni_3V ($D0_{22}$) phase is a critical phase in the dual two-phase intermetallic alloys composed of Ni₃Al and Ni₃V, which possessed outstanding mechanical and chemical properties [6–8]. However, there is limited information on the Ni-V-Ta ternary system [9,10]. In 1971, Moreen et al. [9] investigated the ordering reactions of Ni_8X (X: V, Ta, Nb) by electrical resistivity and electron diffraction techniques. Then, Gupta [10] reviewed the experimental data of the ternary system. Unfortunately, no information of experimental phase diagram and thermodynamic database was found for this ternary system. Therefore, it is essential to establish the phase relation of Ni-V-Ta ternary system for developing thermodynamic database and practical application.

The Ni-V-Ta ternary system consists of three sub-systems: Ni-Ta, Ni-V, and V-Ta binary system, as shown in Figure 1. All the stable compounds and solid solutions are summarized in Table 1.

The Ni-Ta system was systematically investigated by several researchers [11–23]. Solid solution face-centered cubic (fcc), body-centered cubic (bcc-(Ta)), and five intermetallic compounds (Ni₈Ta, Ni₃Ta, Ni₂Ta, Ni₆Ta₇ and 6 NiTa₂) coexist in this binary system according to Nash et al. [16] and

Pan et al. [20]. There are three peritectic reactions and two eutectic reactions in the binary system. The NiTa₂ and Ni₆Ta₇ phases form from the peritectic reaction L + bcc-(Ta) \leftrightarrow NiTa₂ at 1788 °C and L + NiTa₂ \leftrightarrow Ni₆Ta₇ at 1570 °C, respectively. At 1330 °C, the eutectic reaction L \leftrightarrow Ni₃Ta + fcc occur. Meanwhile, the thermodynamic information of this binary system was assessed by several investigators [16–22] with the Computer Coupling of Phase Diagrams and Thermochemistry (CALPHAD) approach and first-principles calculation. The Ni-Ta phase diagram adopted in present work was assessed by Nash et al. for its better agreement with the experimental results.

The Ni-V phase diagram was well studied by many investigators [24–27]. There are two terminal solid solutions (fcc, bcc-(V)) and five intermetallic compounds (σ' , Ni₈V, Ni₃V, Ni₂V and NiV₃) in this system according to Smith et al. [24]. Two eutectoids, a eutectic, a peritectic, a peritectoid, and three congruent transformations exist in the system. The Ni₃V phase forms from a congruent transformation at 1045 °C. The information of the Ni-V system was thermodynamically analyzed by Watson et al. [26] and Kabanova et al. [27], which were in agreement with the experimental results.

As for the V-Ta binary system, a continuous solid solution bcc-(V, Ta) at elevated temperatures and an intermediate phase (V₂Ta) at lower temperatures were identified according to the previous work [28–32]. However, the focus lay on the phase transition of Laves phase at low temperatures. In 1972, Savieky et al. [28] confirmed a C15-type (MgCu₂) phase and C14-type (MgZn₂) phase two-phase field at the temperature range of 1125 °C to 1280 °C. Later, Danon et al. [30] and Pavlů et al. [32] evaluated the V-Ta system by CALPHAD approach and first-principles calculation.

Thus, because of the lacking information of the Ni-V-Ta system, the goal of this work was to establish the isothermal sections of the Ni-V-Ta ternary system at 1000 $^{\circ}$ C and 1200 $^{\circ}$ C.



Figure 1. Binary phase diagrams constituting the Ni-V-Ta ternary system. Data from [16,24,30].

System	Phase	Pearson Symbol	Prototye	Space Group	Strukturbericht	References	
Ni-Ta	fcc	cF4	Cu	Fm-3m	A1	[16]	
	Ni ₈ Ta	tI36	Ni ₈ Nb	I4/mmm		[16]	
	Ni ₃ Ta	tI8	TiAl ₃	I4/mmm	D0 ₂₂	[16]	
		mP16	NbPt ₃	$P2_1/m$	[
		oP8	Cu ₃ Ti	Pmmn	Pmmn D0 _a		
	Ni ₂ Ta	tI6	MoSi ₂	I4/mmm	C11 _b	[16]	
	Ni ₆ Ta ₇	hR39	Fe ₇ W ₆	R-3mh	$D8_5$	[16]	
	NiTa ₂	tI12	Al ₂ Cu	I4/mcm	C16	[16]	
	bcc-(Ta)	cI2	W	Im-3m	A2	[16]	
Ni-V	fcc	cF4	Cu	Fm-3m	A1	[24]	
	Ni ₈ V	tI18	Ni ₈ Nb	I4/mmm		[24]	
	Ni ₂ V	oI6	MoPt ₂	Immm		[24]	
	Ni ₃ V	tI8	Al ₃ Ti	I4/mmm	D0 ₂₂	[24]	
	σ'	tP30	σ	$P4_2/mnm$		[24]	
	NiV ₃	cP8	Cr ₃ Si	Pm3n		[24]	
	bcc-(V)	cI2	W	Im-3m	A2	[24]	
V-Ta	bcc-(V, Ta)	cI2	W	Im-3m	A2	[30]	
	C14	hP12	MgZn ₂	<i>P63/mmc</i>	C14	[30]	
	C15	cF4	MgCu ₂	Fd-3m	C15	[30]	

Table 1. The stable solid phases in the three binary systems. Data from [16,24,30].

2. Experimental Details

The raw materials were the following high purity metals: nickel (99.9 wt. %), vanadium (99.9 wt. %) and tantalum (99.9 wt. %). All alloys were prepared in the form of atomic ratios (at. %). Bulk alloys with nominal composition were prepared by arc furnace melting in a high purity argon atmosphere with a non-consumable tungsten electrode on a water-cooled copper platform. The ingots, about 20 g, were re-melted at least five times to obtain homogeneous alloys with less than 0.5 wt. % weight loss.

All plate-shaped specimens were individually sealed in silica capsules backfilled with high purity argon, and annealed at 1000 °C for 55 days and 1200 °C for 40 days, respectively. Moreover, pure yttrium fillings were filled into the quartz capsules to prevent oxidation, and the alloys with liquid phase were wrapped in the pure tantalum slice to prevent contact reaction with quartz.

After heat treatment and metallographic preparation, the equilibrium compositions of all phases in specimens were determined by electron probe microanalysis (EPMA) with 20 kV accelerating voltage and 1.0×10^{-8} A probe current. The measured values were calibrated by ZAF (Z: atomic number effect, A: absorption effect, F: fluorescence effect) correction, with pure elements were used as standard samples. Energy-dispersive spectrometry (EDS) was used to measure the alloys' nominal composition and liquid phase composition. In order to identify the crystal structure, we used powder X-ray diffraction (XRD), with a Phillips Panalytical X-pet diffractometer using Cu-K α radiation at 40 kV and 40 mA. The data were collected in the range of 20 from 20 to 90 °C with a step interval of 0.015308° and a count time of 0.4 s per step. The melting points of the alloys were determined by differential scanning calorimeter (DSC) with a heating and cooling rate of 10 °C/min.

3. Results and Discussion

3.1. Phase Equilibria at 1000 °C

For the construction of isothermal section of the Ni-V-Ta ternary system at 1000 $^{\circ}$ C, 38 alloys were prepared and the equilibrium composition of each phase was summarized in Table 2.

Table	2. Equilibrium	compositions of	t the phase in t	he Ni-V-Ta terr	hary alloys ar	nnealed at 1000 °	^o C for
55 day	/S.						

	Phase Equilibrium		Co	mposit	osition (at. %)				
Nominal Alloys (at. %)	Dhasa 1 /Dhasa 2 /Dhasa 2	Phase 1		Phase 2		Phase 3			
	Phase 1/Phase 2/Phase 3	V	Та	V	Та	V	Та		
Ni ₂₄ V ₆₇ Ta ₉	bcc-(V)/σ'/C14	90	3.1	70.6	5.2	47.1	27.1		
Ni ₁₃ V ₆₅ Ta ₂₂	bcc-(V)/C14	90.5	5.3	49.8	30.1				
$Ni_{41}V_{49}Ta_{10}$	σ'/C14	54.2	4.4	31.8	21.8				
$Ni_9V_{41}Ta_{50}$	bcc-(Ta)/C14	37	58.5	47.3	36.2				
Ni ₆₁ V ₃₀ Ta ₉	fcc/Ni ₃ Ta/σ'	36.4	3.2	13.2	15	51.6	2.7		
Ni ₅₉ V ₂₄ Ta ₁₇	σ′/C14/Ni ₃ Ta	53	4.7	31	22.1	6.8	19.8		
Ni ₄₈ V ₁₈ Ta ₃₄	Ni ₆ Ta ₇ /C14/Ni ₃ Ta	17.9	47	23.5	29	2.5	24.1		
Ni ₇₇ V ₁₄ Ta ₉	fcc/Ni ₃ V/Ni ₃ Ta	13.1	4.6	17	6.7	12	12.4		
Ni ₁₉ V ₁₁ Ta ₇₀	bcc-(Ta)/Ni ₆ Ta ₇ /NiTa ₂	9.6	88.3	14.5	51.5	2.2	65.9		
Ni ₁₇ V ₆₁ Ta ₂₂	bcc-(V)/C14	90.3	4.8	46.5	29.5				
Ni ₅₂ V ₄₅ Ta ₃	fcc/ \sigma'	37.9	2.8	51.8	2.7				
$Ni_{26}V_{34}Ta_{40}$	Ni ₆ Ta ₇ /C14	26.1	45	37.3	37.9				
Ni ₆₈ V ₁₅ Ta ₁₇	Ni_3Ta/σ'	8.1	18.6	52.5	3.8				
Ni ₆₄ V ₅ Ta ₃₁	Ni ₂ Ta/Ni ₃ Ta/Ni ₆ Ta ₇	3.4	30.6	1.1	25.2	15.8	47.5		
Ni ₈₂ V ₇ Ta ₁₁	Ni ₈ Ta/Ni ₃ Ta	8.9	7.1	6.5	17				
Ni ₁₈ V ₂₃ Ta ₅₉	Ni ₆ Ta ₇ /bcc-(Ta)	23.1	49	22	74.9				
Ni ₈ V ₂₈ Ta ₆₄	Ni ₆ Ta ₇ /bcc-(Ta)	30	46.9	26.6	70				
Ni5V49Ta46	C15/bcc-(Ta)	55.5	35.3	41.1	56.3				
Ni ₆ V ₇₁ Ta ₂₃	C15/bcc-(V)	58.4	30.5	90.7	7.5				
$Ni_{35}V_{50}Ta_{15}$	σ'/C14	62.5	5.9	37.5	25				
Ni ₇₁ V ₁₉ Ta ₁₀	fcc/Ni ₃ Ta	33.7	2.5	17.1	11				
Ni ₇₉ V ₁₀ Ta ₁₁	fcc/Ni ₃ Ta	12.2	5.3	10.2	14.3				
$Ni_{50}V_6Ta_{44}$	Ni ₂ Ta/Ni ₆ Ta ₇	2.1	34	6.7	48.8				
Ni ₃₉ V ₄ Ta ₅₇	NiTa ₂ /Ni ₆ Ta ₇	0.6	64.1	6.5	52.9				
$Ni_9V_2Ta_{89}$	NiTa ₂ /bcc-(Ta)	1	69.1	3.1	94.5				
Ni ₇ V ₁₃ Ta ₈₀	Ni ₆ Ta ₇ /bcc-(Ta)	18	50.2	15.1	81.9				
Ni ₅₉ V ₁₇ Ta ₂₄	Ni ₃ Ta/C14	3.5	22.6	25.2	26.4				
Ni ₃₇ V ₃₅ Ta ₂₈	C14	35	28						
Ni ₃₄ V ₃₃ Ta ₃₃	C14	33	33						
Ni ₄₄ V ₂₆ Ta ₃₀	C14	26	30						
$Ni_4V_{68}Ta_{28}$	C15/bcc-(V)	61.2	31.5	90.1	8.5				
$Ni_5V_{54}Ta_{41}$	C15/bcc-(Ta)	56.5	37	39.8	48.9				
Ni ₁₃ V ₄₆ Ta ₄₁	bcc-(Ta)/C14	39.1	57.8	51	35				
Ni ₁₄ V ₃₆ Ta ₅₀	Ni ₆ Ta ₇ /C14/bcc-(Ta)	31.6	45.2	44.1	36.4	28.7	66.4		
Ni ₁₈ V ₅₅ Ta ₂₇	C14/bcc-(V)	50.2	31	90.2	5.4				
$Ni_{81}V_1Ta_{18}$	Ni ₈ Ta/Ni ₃ Ta	1.5	11.5	0.7	22.5				
Ni ₁₂ V ₆₂ Ta ₂₆	C14/bcc-(V)	52	31.3	91.3	4.8				
Ni ₃₃ V ₂₅ Ta ₄₂	Ni ₆ Ta ₇ /C14	21.4	45.9	29.8	36.1				

Microstructure and XRD results of the typical alloys after annealed at 1000 °C for 55 days are shown in Figures 2 and 3, respectively. Figure 2a–c shows the microstructure of the three-phase region of the alloys. In Figure 2a, the three-phase equilibrium of σ' (black) + Ni₃Ta (gray) + C14 (light gray) in the Ni₅₉V₂₄Ta₁₇ alloy was established. Figure 2b shows the microstructure of the Ni₆₁V₃₀Ta₉ alloy, in which the black phase σ' , the white phase Ni₃Ta and the gray matrix fcc were observed. As displayed in Figure 2c, three-phase region of NiTa₂ + Ni₆Ta₇ + bcc-(Ta) in the Ni₁₉V₁₁Ta₇₀ alloy was identified. The light gray phase NiTa₂ precipitated in the matrix Ni₆Ta₇, along with the white phase bcc-(Ta). Figure 2d–f presents the microstructure of the two-phase section of the alloys. Figure 2d shows the two-phase section of bcc-(Ta) (gray) + C15 (black) in the Ni₅V₄₉Ta₄₆ alloy and the corresponding XRD result is shown in Figure 3a. Figure 2e is the microstructure of Ni₆Ta₇ (gray) + C14 (black) in the Ni₃₃V₂₅Ta₄₂ alloy. In the Ni₁₃V₄₆Ta₄₁ alloy annealed at 1000 °C for 55 days, two-phase equilibrium of C14 + bcc-(Ta) was confirmed in Figure 2f. The corresponding crystal structure was identified by XRD

pattern in Figure 3b. In order to better identify the crystal structure of the C14 phase, we obtained single-phase alloys and the XRD result is presented in Figure 3c.



Figure 2. Microstructure of the typical alloys in the Ni-V-Ta system annealed at 1000 °C for 55 days. (a) $Ni_{59}V_{24}Ta_{17}$; (b) $Ni_{61}V_{30}Ta_9$; (c) $Ni_{19}V_{11}Ta_{70}$; (d) $Ni_5V_{49}Ta_{46}$; (e) $Ni_{33}V_{25}Ta_{42}$; (f) $Ni_{13}V_{46}Ta_{41}$.



Figure 3. The XRD patterns of typical alloys in the Ni-V-Ta system annealed at 1000 $^{\circ}$ C for 55 days. (a) Ni₅V₄₉Ta₄₆; (b) Ni₁₃V₄₆Ta₄₁; (c) Ni₃₄V₃₃Ta₃₃.

According to the microstructure and XRD results, the isothermal section of the Ni-V-Ta system at 1000 °C is shown in Figure 4. Eight three-phase sections, $\sigma' + bcc-(V) + C14$, $fcc + \sigma' + Ni_3Ta$, $C14 + \sigma' + Ni_3Ta$, $fcc + Ni_3Ta + Ni_3V$, $C14 + Ni_6Ta_7 + Ni_3Ta$, $Ni_2Ta + Ni_6Ta_7 + Ni_3Ta$, $NiTa_2 + Ni_6Ta_7 + bcc-(Ta)$, and $C14 + Ni_6Ta_7 + bcc-(Ta)$, were confirmed and marked by triangle with solid lines. Unfortunately, four three-phase equilibriums, $fcc + Ni_8Ta + Ni_3Ta$, $fcc + Ni_3Ta + Ni_3V$, C14 + C15 + bcc-(Ta) and C14 + C15 + bcc-(V) were not established and marked by triangle with dash lines. The determined two-phase region was marked in black fine lines and single phase with small squares. The experimental data indicate that: (1) a single-phase region C14 with a large solubility of three elements, which was measured to be 23.3–52 at. % V, 22.4–38.1 at. % Ta and 13.1–49 at. % Ni, exists at 1000 °C; (2) although the C14 phase is not found in V-Ta binary system at 1000 °C, it is stabilized by addition of Ni confirmed by the presence of a single-phase C14; (3) the Ni₃V phase was confirmed at 1000 °C near the Ni-V side, which did not form a continuous solid solution with the Ni₃Ta phase; (4) the phases fcc, Ni₃V and σ'

dissolve up to 5.8 at. %, 7.1 at. % and 5.9 at. % Ta, respectively; (5) the solubility of V in the phases



Figure 4. Experimentally determined isothermal section of the Ni-V-Ta system at 1000 °C.

3.2. Phase Equilibria at 1200 °C

Based on the experimental results, eight intermetallic compounds (σ' , Ni₈Ta, Ni₃Ta, Ni₂Ta, Ni₆Ta₇, NiTa₂, C14, C15) and two solid solutions—fcc and bcc—were confirmed at 1200 °C, as shown in Table 1. The isothermal section of the Ni-V-Ta ternary system at 1200 °C was established by 38 alloys summarized in Table 3.

	Phase Equilibrium		Co	mposit	ion (at.	. %)					
Alloys (at. %)	Phase 1/Phase 2/Phase 3	Pha	Phase 1		Phase 2		Phase 3				
	Thase 1/Thase 2/Thase 3	V	Ta	V	Ta	V	Та				
Ni ₂₄ V ₆₇ Ta ₉	bcc-(V)/σ'/C14	85.1	4.1	66.2	6.5	43.6	24.6				
Ni ₁₃ V ₆₅ Ta ₂₂	bcc-(V)/C14	88.8	7.5	49.5	29						
Ni ₄₁ V ₄₉ Ta ₁₀ *	σ′/L/C14	55.6	5.5	42.2	11.1	34.5	22.2				
Ni ₉ V ₄₁ Ta ₅₀	bcc-(Ta)/C14	33.2	66.4	45.6	37.3						
Ni ₆₁ V ₃₀ Ta ₉ *	fcc/Ni ₃ Ta/L	34.2	4	9.1	27.5	37.3	11.8				
Ni ₇₅ V ₁₈ Ta ₇	fcc/Ni ₃ Ta	20.9	6.5	16.8	9.1						
Ni59V24Ta17 *	Ni ₃ Ta/L/C14	6.4	20.2	37.5	12.2	26.3	23.7				
Ni ₄₈ V ₁₈ Ta ₃₄	Ni ₃ Ta/Ni ₆ Ta ₇ /C14	2.3	23.1	18.6	46	21	28.6				
Ni ₇₇ V ₁₄ Ta ₉	fcc/Ni ₃ Ta	15.1	6.2	10.8	14.1						
Ni ₁₉ V ₁₁ Ta ₇₀	bcc-(Ta)/Ni ₆ Ta ₇ /NiTa ₂	9.3	89.1	12.5	52.7	0.9	66.1				
Ni ₁₇ V ₆₁ Ta ₂₂	bcc-(V)/C14	88.3	6.6	48.3	28.1						
Ni52V45Ta3 *	fcc/σ//L	41.6	2	53.8	3	45.9	5				
Ni ₂₆ V ₃₄ Ta ₄₀	$Ni_6Ta_7/C14$	28.3	44.9	38	36.8						
Ni ₆₈ V ₁₅ Ta ₁₇ *	Ni ₃ Ta/L	7.9	18.7	37.3	12						
Ni ₆₄ V ₅ Ta ₃₁	Ni ₂ Ta/Ni ₃ Ta/Ni ₆ Ta ₇	3.8	29.9	1.7	24.5	16	46.5				
$Ni_{82}V_7Ta_{11}$	Ni ₈ Ta/Ni ₃ Ta	8	7.9	5.5	18.2						
Ni ₁₈ V ₂₃ Ta ₅₉	Ni ₆ Ta ₇ /bcc-(Ta)	23.8	50	20.9	78.9						
Ni ₈ V ₂₈ Ta ₆₄	Ni ₆ Ta ₇ /bcc-(Ta)	31.5	47.5	24.8	75						
Ni ₅ V ₄₉ Ta ₄₆	C14/bcc-(Ta)	55	36.2	44.3	55.6						
$Ni_{6}V_{71}Ta_{23}$	C14/bcc-(V)	56.5	30.6	89.8	9.5						
Ni35V50Ta15	σ′/C14	60.5	7.2	39.4	23.1						
Ni ₇₁ V ₁₉ Ta ₁₀	fcc/Ni ₃ Ta	26.8	5.1	14.7	12.1						
Ni ₇₉ V ₁₀ Ta ₁₁	fcc/Ni ₃ Ta	10.8	7.5	6.6	17.2						
$Ni_{50}V_6Ta_{44}$	Ni ₂ Ta/Ni ₆ Ta ₇	2.1	32.5	8.1	48.5						
Ni ₃₉ V ₄ Ta ₅₇	$NiTa_2/Ni_6Ta_7$	0.6	64.8	5.7	53.5						
Ni ₉ V ₂ Ta ₈₉	NiTa ₂ /bcc-(Ta)	0.6	66.9	2.6	97.1						
Ni ₇ V ₁₃ Ta ₈₀	Ni ₆ Ta ₇ /bcc-(Ta)	18	51.5	12.4	87.5						
Ni ₅₉ V ₁₇ Ta ₂₄	Ni ₃ Ta/C14	4.7	21.5	24.4	25.1						
Ni37V35Ta28	C14	35	28								
Ni ₃₄ V ₃₃ Ta ₃₃	C14	33	33								
Ni ₄₄ V ₂₆ Ta ₃₀	C14	26	30								
Ni ₃ V ₅₇ Ta ₄₀	C14/C15	47	35.9	46.8	42.1						
Ni ₁₃ V ₄₆ Ta ₄₁	C14/bcc-(Ta)	48.8	36.8	41.5	58						
Ni14V36Ta50	Ni ₆ Ta ₇ /C14/bcc-(Ta)	34	44.5	40.9	37	30.1	68.8				
Ni ₁₈ V ₅₅ Ta ₂₇	C14/bcc-(V)	49.6	30.5	89.1	8.2						
Ni ₈₁ V ₁ Ta ₁₈	Ni ₈ Ta/Ni ₃ Ta	1.5	12.1	0.9	22.1						
Ni ₁₂ V ₆₂ Ta ₂₆	C14/bcc-(V)		30.4	89.3	9.1						
Ni33V25Ta42	$Ni_6Ta_7/C14$	22.5	45	30.2	34.7						

Table 3. Equilibrium compositions of the phase in the Ni-V-Ta ternary alloys annealed at 1200 °C for 40 days except the starred ones.

* The alloys indicated with (*) were annealed at 1200 °C for 3 h.

The microstructure and the corresponding XRD analysis are presented in the Figures 5a–f and 6a–c, respectively. Figure 5a shows the three-phase equilibrium of C14 + σ' + bcc-(V) in the Ni₂₄V₆₇Ta₉ alloy annealed at 1200 °C for 40 days. Figure 5b features the microstructure of the Ni₄₈V₁₈Ta₃₄ alloy. The small black phase Ni₃Ta and the white phase Ni₆Ta₇ were evenly distributed in the gray matrix phase C14. The corresponding phase relationship was identified by XRD result, in which the characteristic peaks of the C14, Ni₃Ta and Ni₆Ta₇ phases were labelled by different symbols, as presented in Figure 6a. In the Ni₁₄V₃₆Ta₅₀ alloy quenched from 1200 °C, the three-phase region of C14 (dark gray) + Ni₆Ta₇ (light gray) + bcc-(Ta) (white) was confirmed in Figure 5c and the corresponding XRD pattern is displayed in Figure 6b. Figure 5d features the microstructure of the Ni₁₉V₁₁Ta₇₀ alloy. The small light gray phase NiTa₂ and white block phase bcc-(Ta), along with the dark gray matrix phase Ni₆Ta₇ were discovered. As presented in Figure 5e, a three-phase section of

 Ni_2Ta (light gray) + Ni_6Ta_7 (white) + Ni_3Ta (black) was identified in the $Ni_{64}V_5Ta_{31}$ alloy annealed at 1200 °C for 40 days, and its XRD result is shown in Figure 6c. Figure 5f is the microstructure of three-phase region of fcc + Ni_3Ta + L in the $Ni_{61}V_{30}Ta_9$ alloy annealed at 1200 °C for 3 h.



Figure 5. Microstructure of the typical alloys in the Ni-V-Ta system annealed at 1200 °C for 40 days. (a) $Ni_{24}V_{67}Ta_9$; (b) $Ni_{48}V_{18}Ta_{34}$; (c) $Ni_{14}V_{36}Ta_{50}$; (d) $Ni_{19}V_{11}Ta_{70}$; (e) $Ni_{64}V_5Ta_{31}$; and (f) the $Ni_{61}V_{30}Ta_9$ alloy annealed at 1200 °C for 3 h.



Figure 6. The X-ray diffraction (XRD) patterns of typical alloys in the Ni-V-Ta system annealed at 1200 °C for 40 days. (**a**) Ni₄₈V₁₈Ta₃₄; (**b**) Ni₁₄V₃₆Ta₅₀; (**c**) Ni₆₄V₅Ta₃₁.

Based on the experimental data mentioned above, the isothermal section of the Ni-V-Ta system at 1200 °C was established, as shown in Figure 7. Nine three-phase sections, $\sigma' + bcc-(V) + C14$, $\sigma' + C14 + L$, $\sigma' + fcc + L$, $fcc + L + Ni_3Ta$, $C14 + L + Ni_3Ta$, $C14 + Ni_6Ta_7 + Ni_3Ta$, $Ni_2Ta + Ni_6Ta_7 + Ni_3Ta$, $NiTa_2 + Ni_6Ta_7 + bcc-(Ta)$ and $C14 + Ni_6Ta_7 + bcc-(Ta)$, were experimentally determined and presented as a triangle with solid lines. The other two three-phase equilibriums C15 + C14 + bcc-(Ta) and $fcc + Ni_3Ta + Ni_8Ta$ were not confirmed and were displayed as a triangle with dashed lines. The corresponding results indicate that: (1) the C14 phase extends from the V-Ta side to the center and dissolves more Ni at 1200 °C, while the solubility of Ni in the C15 phase decreases than that at 1000 °C; (2) due to the congruent transformation fcc $\leftrightarrow Ni_3V$ at 1045 °C, the Ni_3V phase disappeared at 1200 °C; (3) a small liquid region was detected; (4) the solubility of V in the phases Ni_3Ta, Ni_2Ta and Ni_6Ta_7 was measured to be around 17.2 at. %, 3.9 at. % and 33.8 at. %, respectively, which increases slightly as the temperature increases from 1000 to 1200 °C; (5) the phases fcc, σ' and bcc-(V) dissolve about 6.2 at. %, 7.3 at. % and 10.1 at. % Ta, respectively.



Figure 7. Experimentally determined isothermal section of the Ni-V-Ta system at 1200 °C.

3.3. Liquid Region

It is noticeable that a small liquid section was confirmed near the Ni-V side. As shown in Figure 2f, the bar-shaped white phase Ni₃Ta and the black phase fcc uniformly distribute in the liquid matrix. According to the eutectic reaction, $L \leftrightarrow fcc + \sigma'$ at 1202 °C in the Ni-V binary system, the eutectic microstructure is suggested to be consistent with the fcc and σ' phase. In order to identify the temperature of phase transition, we conducted DSC analysis. With the DSC result shown in Figure 8, we observed that the σ' phase transformed to the liquid phase when the temperature increased from 1100 to 1200 °C in the Ni₅₉V₂₄Ta₁₇ alloy. The melting point of the σ' phase in the alloy was confirmed

to be about 1137 °C. The corresponding results indicate that a third element addition of Ta in Ni-V alloys reduces the melting point of the alloys.



Figure 8. Heating curve of the Ni₅₉V₂₄Ta₁₇ alloy. The microstructure of the Ni₅₉V₂₄Ta₁₇ alloy annealed at (**a**) 1100 °C for 30 days; (**b**) 1200 °C for 3 h.

4. Conclusions

The results are shown as follows: (1) two isothermal sections of the Ni-V-Ta ternary system at 1000 °C and 1200 °C were experimentally established by the means of equilibrated alloy method; (2) the C15 phase dissolves less Ni, while the solubility of Ni in the C14 phase increases when the temperature raises from 1000 to 1200 °C; (3) the C14 phase was stabilized by addition of Ni against low temperature confirmed by the presence of a single-phase region at 1000 °C; (4) a small liquid phase was determined, while the Ni₃V phase just disappeared at 1200 °C; (5) the solubility of Ta in the phases fcc and σ' increases as the temperature increases from 1000 to 1200 °C, while the solubility of Ni in the bcc-(Ta) phase shows the opposite tend. This research is of great significance to support the thermodynamic assessment of the Ni-V-Ta system.

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