

# Ф И З И К А P H Y S I C S

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## **Dovchinvanchig Maashaa**

*PhD, School of Engineering and Technology, Mongolia University of Life Sciences, Ulaanbaatar, Mongolia, e-mail: Dovchinvanchig@muls.edu.mn*

## **Tsendsuren Balgan**

*PhD, School of Engineering and Technology, Mongolia University of Life Sciences, Ulaanbaatar, Mongolia, e-mail: tsendsuren\_balgan@yahoo.com*

## **Zhao Chun Wang**

*PhD, College of Arts and Sciences, Shanghai Maritime University, Shanghai Shi, Shanghai, China*

## **Microstructure and martensitic transformation of $Ni_{50}Ti_{47}Nd_3$ shape memory alloy** (Peer-reviewed)

**Abstract.** *The phase transformation and microstructure behavior of  $Ni_{50}Ti_{47}Nd_3$  shape memory alloy was investigated by scanning electron microscope, X-ray diffraction and differential scanning calorimetry. The results showed that the microstructure of Ni-Ti-Nd ternary alloy consists of the NiNd phase and the NiTi matrix phase. One-step phase transformation was observed alloy.*

**Keywords:** *Ni-Ti-Nd shape memory alloy, microstructure, martensitic transformation.*

## **Довчинванчиг Маашаа**

*Кандидат наук, Школа инжиниринга и технологий, Монгольский университет наук о жизни, Улан-Батор, Монголия, e-mail: Dovchinvanchig@muls.edu.mn*

## **Цендсурен Балган**

*Кандидат наук, Школа инжиниринга и технологий, Монгольский университет наук о жизни, Улан-Батор, Монголия, e-mail: tsendsuren\_balgan@yahoo.com*

## **Чжао Чунь Ван**

*Кандидат наук, Колледж искусств и наук, Шанхайский морской университет, Шанхай Ши, Шанхай, Китай*

## **Микроструктура и мартенситное превращение сплава состава $Ni_{50}Ti_{47}Nd_3$ с памятью формы**

**Аннотация.** *Методами сканирующей электронной микроскопии, дифракции рентгеновских лучей и дифференциальной сканирующей калориметрии исследованы фазовое превращение и поведение микроструктуры сплава  $Ni_{50}Ti_{47}Nd_3$  с памятью. Результаты показали, что микроструктура троичного сплава Ni-Ti-Nd состоит из фазы NiNd и матричной фазы NiTi. Наблюдалось пошаговое фазовое превращение сплава.*

**Ключевые слова:** *сплав Ni-Ti-Nd, обладающий памятью формы, микроструктура, мартенситное превращение.*

## **Introduction**

Ni-Ti based shape memory alloys (SMAs) are very important materials because of unique shape memory effect and super-elasticity behavior. Today this kind of material is used in many different fields, especially in engineering and medical application. Current research interest on SMA are mainly controlling the martensitic transformation temperature and improving the shape memory effect for their applications. The effect of martensitic transformation, super-elasticity and shape memory effect have been studied widely by adding transitional elements to Ni-Ti binary alloys which include Fe, Nb, Hf, Zr, Pd, Pt etc. Among them, Fe and Nb have been added to Ni-Ti binary alloys, which decrease the martensitic transformation temperature. But Hf, Zr, Pd and Pt

addition can increase the martensitic transformation temperature of Ni-Ti alloys.

Moreover, the microstructure and martensitic transformation temperature of the Ni-Ti binary alloys have also been studied using scanning electron microscopy (SEM), energy dispersive spectrometry (EDS), X-ray diffraction (XRD), and differential scanning calorimetry (DSC). The Ni-Ti binary alloys were found to decrease and increase the phase transformation temperature and change the phase transformation sequence.

Rare-earth element Nd is also a widely used element, particularly in magnetic materials. However, only few studies have been conducted on Nd addition to a shape memory alloy. The only study found in the literature is that on Nd addition to Ni-Ti-Fe alloy, but the Nd fraction is less than 1%. The effect of Nd addition to Ni-Ti binary alloy on microstructure and phase transformation temperature remains unclear.

In this paper Nd content with atomic fraction of 3% added to Ni-Ti binary alloys, and the microstructure and martensitic transformation are studied experimentally.

## 1. Experimental methods

The  $Ni_{50}Ti_{47}Nd_3$  alloy were prepared by melting each 10g of raw materials with different nominal compositions (99,9 mass% sponge Ti, 99,7 mass% electrolytic and 99,95 mass% Nd) in a nonconsumable arc-melting furnace using a water-cooled copper crucible. The alloy is denoted  $Ni_{50}Ti_{47}Nd_3$  alloy. Melting was repeated four times to ensure the uniformity of composition. The specimens are spark-cut from the ingots and solution – treated at 850°C for an hour in a quartz tube furnace. Subsequently the specimens were quenched using water. Thereafter, the specimens are mechanically and lightly polished to obtain a plain surface.

The phase transformation temperature of  $Ni_{50}Ti_{47}Nd_3$  alloy was determined by DSC using a TA Q2000 calorimeter. The temperature range of heating and cooling was from -30°C to 155°C, and the scanning rate of heating and cooling was 10°C/min. SEM observations were conducted using a FEI Quanta 650 FEG equipped with EDS analysis systems made by Oxford. An XRD experiment was conducted using a D/MAX-2500PC X-ray diffractometer.

## 2. Results and discussion

### 2.1. Microstructure of $Ni_{50}Ti_{47}Nd_3$ alloy

Fig. 1 depicts the back-scattering SEM images of  $Ni_{50}Ti_{47}Nd_3$  alloy. There are two different morphologies, namely, white phase and matrix can be identified in the SEM image. The white phase is in irregular shape and distributed randomly in the matrix.

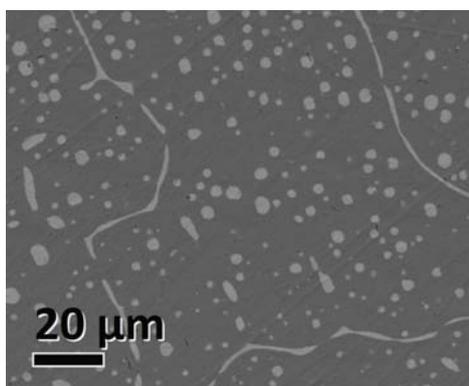


Fig. 1. Back-scattering SEM image  $Ni_{50}Ti_{47}Nd_3$

To identify the phase structure, EDS analysis was conducted in SEM. The compositions of Ni-Ti-Nd alloy are shown in Table 1. The Ti: Ni ratio in the matrix of all Ni-Ti alloy is measured to be near 1. Thus, the matrix can be concluded to be NiTi phase. The Nd: Ni ratio in the white phase of Ni-Nd alloy is measured to be near 1:1. It was found by XRD analysis, that there is a

NiTi phase. Thus, the white phase can be concluded to be NiNd.

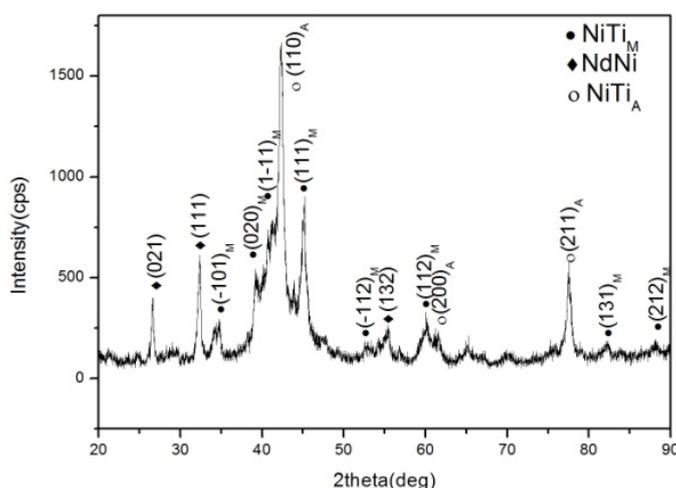
Table 1

Composition of Ni-Ti-Nd alloy

		Ti (at. %)	Ni (at. %)	Nd (at. %)
Ni <sub>50</sub> Ti <sub>47</sub> Nd <sub>3</sub>	Matrix	49,24	50,21	0,55
	White phase	2,97	49,84	47,19

### 2.2. XRD analysis of Ni<sub>50</sub>Ti<sub>47</sub>Nd<sub>3</sub> alloy

Fig. 2 depicts the XRD curve of Ni<sub>50</sub>Ti<sub>47</sub>Nd<sub>3</sub> alloy at room temperature. The diffraction peaks are identified to be from NiTi B19' phase and NiNd phase alloy after comparing with JCPDF cards (numbers 65-0145 and 19-0818). The detailed crystal plane indices are marked in Fig. 1 for the relative intensities of each XRD curve are quite different because of the differences in NiTi phase fraction and NiNd phase fraction. In this paper, the letter NiTi<sub>M</sub> denotes the NiTi B19' phase. This perspective will be confirmed in the following DSC analysis.

Fig. 2. XRD curve of Ni<sub>50</sub>Ti<sub>47</sub>Nd<sub>3</sub>

The lattice parameters of alloy can be also calculated by peaks position in XRD curve and in Table 2 it is shown clearly that cell volume  $V$  expand for either Ni-Ti-Nd ternary alloy. The observation can also be confirmed in the following composition analysis.

Table 2

Lattice parameters of Ni<sub>50</sub>Ti<sub>47</sub>Nd<sub>3</sub> alloy

Alloy	Phase	$a$ (nm)	$b$ (nm)	$c$ (nm)	$\beta$ (°)	$V$ (nm <sup>3</sup> )
Ni <sub>50</sub> Ti <sub>47</sub> Nd <sub>3</sub>	M	0,2939	0,4124	0,4639	98,39	0,05562

### 2.3. Phase transformation of Ni<sub>50</sub>Ti<sub>50</sub> alloy

Fig. 3 depicts the DSC curves of the Ni<sub>50</sub>Ti<sub>47</sub>Nd<sub>3</sub> alloy. Each DSC curve of Ni<sub>50</sub>Ti<sub>47</sub>Nd<sub>3</sub> shows only one peak during the heating and cooling process, which indicates a one-step B2 $\leftrightarrow$ B19' phase transformation. The effect of Ni-Ti concentration on martensitic transformation start temperature  $M_s$ . For Ni-Ti alloy, the  $M_s$  is measured to be 77,44°C. For example, Liu et al. [1] measured the  $M_s$  to be about -50°C for Ni<sub>50,7</sub>Ti<sub>49,3</sub> alloy after annealing at 900°C for 60 min. Tabish et al. [2] measured the  $M_s$  to be -22,12°C for Ni<sub>50</sub>Ti<sub>50</sub> alloy after annealing at 1000°C

for 120 min. Wasilewski et al. [3] measured the  $M_s$  to be  $65^\circ\text{C}$  for  $\text{Ni}_{49,8}\text{Ti}_{50,2}$  alloy. In this work, the composition of the matrix is measured to be  $\text{Ni}_{49,36}\text{Ti}_{50,64}$ , which is Ti-rich. So, a high  $M_s$  of Ti-Ni binary alloy is reasonable. Again, the martensitic transformation finish temperature  $M_f$  in NiTi alloy is higher than room temperature of  $20^\circ\text{C}$ . Thus, the martensitic transformations have been finished at room temperature and the Ni-Ti-Nd alloy should be in total martensitic phase, which is in agreement with the XRD results.

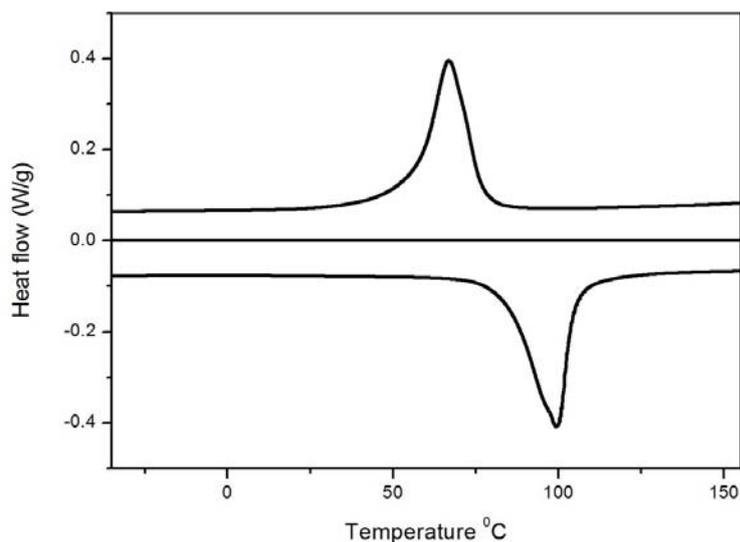


Fig. 3. DSC curve of  $\text{Ni}_{50}\text{Ti}_{47}\text{Nd}_3$

### 3. Conclusions

In summary, the microstructure and martensitic transformation behavior was investigated by XRD, SEM and DSC. The microstructure of the  $\text{Ni}_{50}\text{Ti}_{47}\text{Nd}_3$  alloy consists of NiNd alloy and NiTi matrix. The lattice parameters of  $\text{Ni}_{50}\text{Ti}_{47}\text{Nd}_3$  matrix are  $a=0,2939$  nm,  $b=0,4124$  nm,  $c=0,4639$  nm. The Ni-Ti-Nd alloy has a one-step phase transformation.

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