

# NANOCRYSTAL, PHASE TRANSFORMATION AND MICROSTRUCTURE OF NI50TI50 SHAPE MEMORY ALLOY

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## **ABSTRACT:**

The nanocrystal, phase transformation and microstructure behavior of  $Ni_{50}Ti_{50}$  shape memory alloy was investigated by scanning electronic microscope, X-ray diffraction and differential scanning calorimetry. The results showed that the microstructure of Ni-Ti binary alloy consists of the  $NiTi_2$  phase and the NiTi matrix phase. One-step phase transformation was observed alloy.

**KEYWORDS:** Shape memory effect; Super-elasticity, Martensitic transformation;

## **INTRODUCTION**

Ni-Ti based shape memory alloys (SMAs) is a very important material because it owns unique shape memory effect and super-elasticity behaviors [1]. Today this kind of material has been 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 has included

# MATERIALS AND METHODS

The Ni<sub>50</sub>Ti<sub>50</sub> alloy were prepared by melting each 10g of raw materials with different nominal compositions (99.9 mass% sponge Ti, 99.7 mass% electrolytic Ni) in a nonconsumable arc-melting furnace using a water-cooled copper crucible. The alloy in denoted Ni<sub>50</sub>Ti<sub>50</sub> alloy, respectively. Are-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

Fe, Nb, Hf, Zr, Pd, Pt, etc. Among, Fe and Nb have been added to Ni-Ti binary alloys, which decreased the martensitic transformation temperature. But, Hf, Zr, Pd and Pt addition can increase the martensitic transformation temperature of Ni-Ti alloys. Moreover, the nanocrystal, 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).

mechanically and lightly polished to obtain a plain surface. The phase transformation temperature of  $Ni_{50}Ti_{50}$  alloy were 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.

## **RESULTS AND DISCUSSION**

#### Microstructure of Ni<sub>50</sub>Ti<sub>50</sub> alloy

Fig.1 depicts the back-scattering SEM images of  $Ni_{50}Ti_{50}$  alloy. There are two different morphologies, namely, black phase and matrix can be identified in the SEM image. The black phase is in irregular shape and distributed randomly in the matrix. To analyze the chemical composition Ni-Ti alloy, EDS measurement was conducted during SEM and the

results 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 concluded to be NiTi phase. The Ti:Ni ratio in the black phase of Ni-Ti alloy is measured to be near 2:1. By XRD analysis, there is a NiTi<sub>2</sub> phase in Ni<sub>50</sub>Ti<sub>50</sub>. Thus, the black phase can be concluded to be NiTi<sub>2</sub>.



Figure 1. Back-scattering SEM image Ni<sub>50</sub>Ti<sub>50</sub>

Table 1.

Composition of Ni-Ti alloy									
Alloy	Phase	Ti (at. %)	Ni (at. %)						
Ni50Ti50	matrix	50.64	49.36						
	black	66.99	33.01						
	phase								

## XRD analysis of Ni<sub>50</sub>Ti<sub>50</sub> alloy

Fig.2 depicts the XRD curve of  $Ni_{50}Ti_{50}$  alloy at room temperature. The diffraction peaks are identified to be from NiTi B19' martensite phase and NiTi<sub>2</sub> phase alloy after comparing with JCPDF cards (numbers 65-0145 and 72-0442). 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 martensite phase fraction and NiTi<sub>2</sub> phase fraction. In this paper, the letter denotes the NiTi B19' martensite phase and the denotes the NiTi<sub>2</sub> phase. This perspective will be confirmed in the following DSC analysis. The lattice parameters of alloy can be also calculated by peaks position in XRD curve and shows in Table.2 It is shown clearly that cell volume V expand for either Ni-Ti binary alloy. The observation can also be confirmed in the following composition analysis.



Figure 2. XRD curve of Ni<sub>50</sub>Ti<sub>5</sub>

Table 2.

Lattice parameters of Ni-Ti alloy										
Alloy	Phase	<i>a</i> (nm)	<i>b</i> (nm)	<i>c</i> (nm)	$\beta(^{\circ})$	$V(nm^3)$	Source			
Ni50Ti50	М	0.2898	0.4121	0.4619	97.86	0.05465	This work			
NiTi	М	0.2898	0.4108	0.4646	97.78	0.05480	JCPDF card No.65-0145			

#### 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>50</sub> alloy. Each DSC curve of Ni<sub>50</sub>Ti<sub>50</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. It is well known that quenched Ni-Ti binary alloys show one-step B2↔B19' transformation and the transformation temperatures are strongly dependent on Ni concentration. 0.1 at. % increase in Ni concentration can lower the  $M_s$  of Ni-Ti binary alloys by more than 10 °C. For example, Liu *et al* 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 60min. T.A. Tabish et al measured the M<sub>s</sub> to be -22.12 °C for Ni<sub>50</sub>Ti<sub>50</sub> alloy after annealing at 1000 °C for 120min [8]. R.J.Wasilewski et al measured the  $M_s$  to be 65 °C for Ni<sub>49.8</sub>Ti<sub>50.2</sub> alloy [9]. In this work, the composition of the matrix is measured to be Ni<sub>49,36</sub>Ti<sub>50,64</sub>, which is Ti-rich. So, a high  $M_s$  of Ti-Ni binary alloy is reasonable. Again, the martensite transformations finish temperature  $M_f$ in Ni-Ti alloy is higher than room temperature of 20 °C. Thus, the martensite transformations have finished at room temperature and the Ni-Ti alloy should be in total martensite phase, which is in agreement with the XRD results.



Figure. 3 DSC curve of Ni<sub>50</sub>Ti<sub>50</sub>

## **CONCLUSIONS**

the microstructure In summary, and phase transformation behavior was investigated by XRD, SEM and DSC. The microstructure of the Ni<sub>50</sub>Ti<sub>50</sub> alloys consists of NiTi<sub>2</sub> alloy and NiTi matrix. The

## REFERENCES

- [1] K. Otsuka, and X. Ren, "Recent developments in the research of shape memory alloys," Intermetallics, vol. 7, no. 5, pp. 511-528, 1999.
- [2] J. Frenzel, J. Pfetzing, K. Neuking, et al, "On the

lattice parameters of NiTi matrix is a=0.2898nm, b=0.4121nm, c=0.4619nm. The Ni-Ti alloy has a one-step martensitic transformation.

influence of thermomechanical treatments on the microstructure and phase transformation behavior of Ni-Ti-Fe shape memory alloys," Mat. Sci. Eng. A, vol. 482-484, pp. 635-638,

2008.

- [3] X.L. Meng, W. Cai, Y.D Fu, et al, "Shape memory behaviors in an aged Ni-rich TiNiHf high temperature shape-memory alloy," Intermetallics, vol. 16, no. 5, pp. 698-705, 2008.
- [4] Y. Liu, M. Kohl, K. Okutsu, et al, "TiNiPd thin film microvalve for high temperature applications," Mat. Sci. Eng. A, vol. 378, no. 1-2, pp. 205-209, 2004.
- [5] K. Otsuka, X. Ren, "Physical metallurgy of Ti-Ni-based shape memory alloys," Prog. Mater. Sci., vol. 50, no. 5, pp.511-678, 2005.
- [6] W. Cai, A.L. Liu, J.H. Sui, L.C. Zhao, "Effects of Cerium Addition on Martensitic Transformation and Microstructure of Ti<sub>49.3</sub>Ni<sub>50.7</sub>

Alloy," Mater. Trans., vol.47, no. 3, pp. 716-719, 2006.

- [7] J.W. Xu, A.L. Liu, W. Cai, "Crystal structure and formation mechanism of rare earth rich phases in Ti-Ni-Ce alloys," J. Funct. Mater., vol.39, no.4, pp. 600-602, 2008.(in Chinese)
- [8] T.A. Tabish, S. Atiq, M. Ali, A.N. Ch, Z.U. Reham, T.Z. Butt, "Development and characterization of Ni<sub>50</sub>Ti<sub>50</sub> shape memory alloy used for biomedical applications," Int. J. Chem Mater. Sci. Eng., vol. 7, no. 12, pp. 92-93, 2013.
- [9] R.J. Wasilewski, S.R. Butler, J.E. Hanlon, "On the Martensitic Transformation in TiNi," Met. Sci. J., vol. 1, pp. 104-110, 1967.