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ARTICLE

# MICROSTRUCTURE AND PHASE TRANSFORMATION OF Ni-Ti-Fe-Nd QUATERNARY SHAPE MEMORY ALLOY

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***Abstract:*** *The effect of rare earth element neodymium (Nd) addition on the microstructure and phase transformation behavior of $Ni_{50}Ti_{47}Fe_2Nd_1$ shape memory alloy was investigated by scanning electronic microscope, X-ray diffraction and differential scanning calorimetry. The results show that the microstructure of Ni-Ti-Fe-Nd quaternary alloy consists of $NiNd_3$ phase, $NiTi_2$ and the NiTi matrix. A one-step martensitic transformation is observed in the alloys. The martensitic transformation start temperature Ms $54.11^0$C.*

***Keywords:*** *Ni-Ti-Fe-Nd quaternary shape memory alloy; Microstructure; Phase transformation;*

## INTRODUCTION

Ni-Ti based shape memory alloys are technologically important materials because of their unique shape memory effect and super elastic behavior and they have been used in various fields, particularly in engineering and medical application [1]. The current research interest on SMA is primarily on controlling the martensitic transformation temperature and improving the shape memory effect for their application.

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, 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.

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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 shape memory alloy. The only study found in the literature is that on Nd addition to $Ti_{50}Ni_{47}Fe_{2.5}Nd_{0.5}$ alloy [2]. The effect of Nd addition to Ni-Ti-Fe ternary alloy on microstructure and martensite transformation temperature remains unclear. In this paper, $Ni_{50}Ti_{47}Fe_{2}Nd_{1}$ quaternary alloys, and the microstructure and phase transformation were studied experimentally.

## MATERIALS AND METHODS

The $Ni_{50}Ti_{47}Fe_{2}Nd_{1}$ quaternary alloy was prepared by melting 50 g of raw materials with different nominal compositions (99.99 mass % sponge Ti, 99.99 mass % electrolytic Ni, 99.99 mass % Fe and 99.95 mass % Nd) in a non-consumable arc-melting furnace using a water-cooled copper crucible. Arc-melting was repeated four times to ensure the uniformity of the composition. The specimens are spark-cut from the ingots and solution-treated at 850° C for one hour in a quartz tube furnace. Subsequently, the specimens were quenched using water. Thereafter, the specimens were mechanically and lightly polished to obtain a plain surface.

The phase transformation temperatures of $Ni_{50}Ti_{47}Fe_{2}Nd_{1}$ alloys were determined by differential scanning calorimeter (DSC) using a Perkin-Elmer Diamond calorimeter. The temperature range of heating and cooling was from -30° C to 150° C, and the scanning rate of heating and cooling was 10° C/min. SEM observations were conducted using a S-3400N Hitachi 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 $Ni_{50}Ti_{47}Fe_{2}Nd_{1}$ alloy*

Figure 1 depicts the XRD pattern of $Ni_{50}Ti_{47}Fe_{2}Nd_{1}$ alloy at room temperature. The diffraction peak can be attributed to NiTi B2 parent phase, a $NiTi_2$ phase and a $NiNd_3$ phase after comparison with the corresponding JCPDF cards (Nos. 65-4572, 18-0898, and 19-0818). By combining the XRD pattern result with EDS data, the $Ni_{50}Ti_{47}Fe_{2}Nd_{1}$ alloy can be confirmed to consist of near-aquiatomic NiTi matrix, $NiTi_2$ precipitates and $NiNd_3$ precipitates.

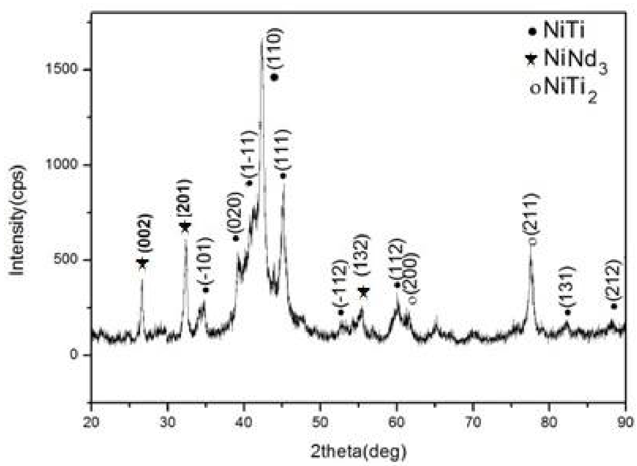


*Figure 1. XRD pattern of Ni50Ti47Fe2Nd1 alloys*

The lattice parameter of alloy can be also calculated [3] by peak position in XRD pattern which shown in Table 1.

*Table 1. Lattice parameters of Ni-Ti-Fe-Nd alloys*

| Alloy | Phase | *a*(nm) | *V*(nm$^3$) | Source |
|---|---|---|---|---|
| Ni-Ti-Fe-Nd | P | 0.3035 | 0.02795 | This work |
| NiTi | P | 0.3007 | 0.02719 | JCPDF card No.65-4572 |
| $NiTi_2$ | | 1.131 | 1.4503 | JCPDF card No.72-0442 |
| $NiNd_3$ | | 0.3803 | 0.17262 | JCPDF card No.19-0818 |

***Morphologies and compositions of $Ni_{50}Ti_{47}Fe_2Nd_1$ alloy***

Figure 2 depicts the back-scattering SEM images of $Ni_{50}Ti_{47}Fe_2Nd_1$ alloy. Three different morphologies, namely, white phase, black phase and matrix, can be identified in the SEM image (Figure 2). Some white particles that are nearly round-shape and up to 5 μm in diameter respectively with white curving areas can be found to be distributed randomly in the matrix. The black phase is distributed randomly in the matrix.

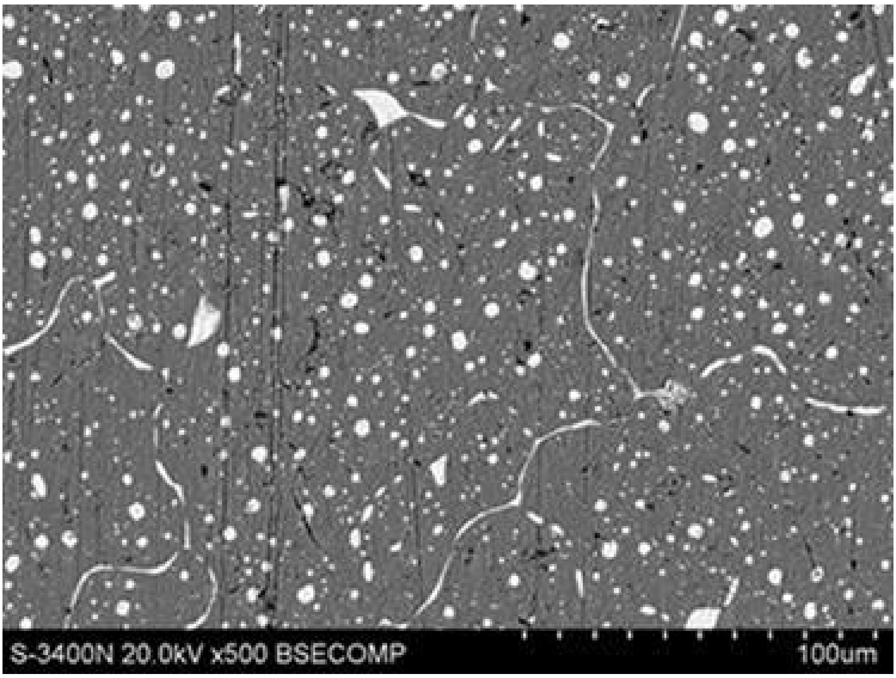


*Figure 2. Back-scattering SEM images of Ni-Ti-Fe-Nd alloys*

To identify the phase structure, EDS analysis was conducted in SEM. The compositions of $Ni_{50}Ti_{47}Fe_2Nd_1$ alloy are shown in Table 2. The Ti:Ni ratio in the matrix of Ni-Ti-Fe-Nd alloy is measured to be close to 1 and with a small amount of Fe solid solute. The Ti:Ni ratio in the white phase of Ni-Ti-Fe-Nd alloy is measured to be close to 2:1. By XRD analysis, there is an $NiTi_2$ phase . Thus, the black phase can be concluded to be $NiNd_3$ phase. According to the 773 K isothermal section of the ternary alloy phase diagram of the Ni-Ti-Nd, no intermetallic compounds can be found in the Ti-Nd binary system. However,

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Ni-Nd binary alloy phase diagram shows seven kinds of intermetallic compounds defined as $NdNi_5$, $Nd_2Ni_7$, $NdNi_3$, $NdNi_2$, NdNi, $Nd_7Ni_{13}$, and $Nd_3Ni$ [4]. The EDS results show that the Ni:Nd ratio in the white phase is close to 1:3 and can be regarded as the $NiNd_3$ intermetallic compound with a small amount of Ti and Fe solid solute.

*Table 2. Compositions of Ni-Ti-Fe-Nd alloys*

| Phase | Ni (at %) | Ti (at %) | Fe (at %) | Nd (at %) |
|---|---|---|---|---|
| matrix | 49.10 | 49.07 | 1.82 | 0.01 |
| white phase | 33.35 | 65.11 | 1.54 | 0 |
| Black phase | 24.58 | 1.32 | 0.89 | 73.21 |

***Phase transformation of $Ni_{50}Ti_{47}Fe_2Nd_1$ alloys***

Figure 3 depicts the DSC curve of the $Ni_{50}Ti_{47}Fe_2Nd_1$ alloy. Each DSC curve shown in one peak during the heating and cooling process, which indicates a one-step B2↔B19' phase transformation. Cooling process in martensitic start temperature Ms=54.110 C and martensitic finish temperatue $M_f$=3.50 C, heating process in austenitic start temperature As=27.050 C and austenitic finish temperature $A_f$=83.50 C.

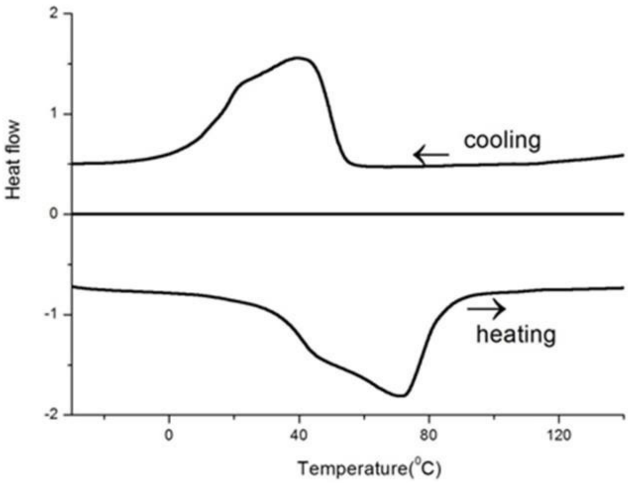


*Figure 3. DSC curve of Ni-Ti-Fe-Nd alloy*

## CONCLUSIONS

The microstructure and phase transformation Ni-Ti-Fe-Nd shape memory alloy is investigated. The following conclusions can be drawn. The $Ni_{50}Ti_{47}Fe_2Nd_1$ alloy exhibits one-step phase transformation on both heating and cooling process. The microstructure of the $Ni_{50}Ti_{47}Fe_2Nd_1$ alloy consists of NiTi B2 parent phase, $NiTi_2$ phase and $NiNd_3$ phase. The lattice of $Ni_{50}Ti_{47}Fe_2Nd_1$ is a=0.3035nm.

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