



# Influence of Fe addition on phase transformation behavior of NiTi shape memory alloy



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**Abstract:** Three different NiTi-based alloys, whose nominal compositions were Ni<sub>50</sub>Ti<sub>50</sub>, Ni<sub>49</sub>Ti<sub>49</sub>Fe<sub>2</sub>, Ni<sub>45</sub>Ti<sub>51.8</sub>Fe<sub>3.2</sub> (mole fraction, %), respectively, were used in the current research to understand the influence of Fe addition on phase transformation behavior in NiTi shape memory alloy (SMA). The microstructure and phase transformation behavior of the alloys were investigated by optical microscopy (OM), transmission electron microscopy (TEM), X-ray diffraction (XRD) and differential scanning calorimetry (DSC) analysis. The results show that the matrix of the Ni<sub>50</sub>Ti<sub>50</sub> alloy consists of both B19' (martensite) phase and B2 (austenite) phase. Moreover, the substructures of twins could be observed in the B19' phase. However, the ternary alloys of NiTiFe exhibit B2 phase in the microstructures. Such microstructures were also characterized by large presence of Ti<sub>2</sub>Ni precipitates dispersed homogeneously in the matrix of the two kinds of alloys. The addition of Fe to the NiTi SMA results in the decrease in phase transformation temperatures in the ternary alloys. Based on mechanism analysis, it can be concluded that this phenomenon is primarily attributed to atom relaxation of the distorted lattice induced by Ni-antisite defects and Fe substitutions during phase transformation, which enables stabilization of B2 phase during phase transformation.

**Key words:** shape memory alloy; NiTi alloy; NiTiFe alloy; microstructure; austenite; martensite

## 1 Introduction

Near-equiatomic NiTi shape memory alloys (SMAs) have attracted much attention due to their unique shape memory effect, excellent superelasticity, good mechanical properties and perfect corrosion resistance. As a result, they have become promising candidates for biomedical, control engineering and aerospace applications in recent years [1–5]. It is well known that adding a third element to replace Ni and/or Ti has a substantial effect on phase transformation behavior and microstructures of NiTi alloys. Previous studies have shown that addition of elements, such as Cu, Fe, Nb, Hf and Cr, to binary NiTi alloys satisfies some specific needs [6–10]. Among these NiTi-based alloys, NiTiFe alloys have been widely used for pipe joints owing to their excellent shape memory effect. This is attributed to the fact that the finish temperature of martensite transformation  $M_f$  of pipe joints made from NiTiFe SMA

is below 0 °C and the finish temperature of austenite transformation  $A_f$  of it is lower than the room temperature. The operation principle of the pipe joint made from NiTiFe SMA is as follows. Firstly, a pipe joint whose inner diameter is smaller than the outer diameter of the two pipes is manufactured by machining. Secondly, the machined pipe joint is cooled below  $M_f$  and subsequently its inner hole is enlarged by mandrel reaming process to the size that its inner diameter is larger than the outer diameter of the pipes. Thirdly, the enlarged pipe joint is assembled with the pipes and then is heated above  $A_f$ . As a consequence, the pipe joint recovers its shape and binds the pipes tightly at room temperature [11]. It has been concluded that the phase transformation temperatures of the SMAs could be lowered by Fe addition because Fe has a strong preference for entering into Ni-site and substitutes Ni [12,13]. If Fe atoms merely substitute the site of Ni atoms and exhibit the similar chemical property as Ni, their phase transformation temperatures should

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approximate to those of the binary NiTi SMAs. However, it has been found that phase transformation temperatures of all the NiTiFe SMAs are much lower than those of the binary NiTi SMAs according to the current references [7,13–15]. In these works, FAN et al [7] investigated the phase transformation of  $\text{Ni}_{48}\text{Ti}_{50}\text{Fe}_2$  SMA and their results showed that the phase transformation temperatures of the alloy are much smaller than those of the  $\text{Ni}_{50}\text{Ti}_{50}$  SMA. OTSUKA and REN [13] listed the phase transformation temperatures of some NiTi-based SMAs in their article, where the phase transformation temperatures of NiTiFe SMAs are much lower than those of  $\text{Ni}_{50}\text{Ti}_{50}$  SMA. XUE et al [14] investigated the phase transformation of  $\text{Ni}_{47}\text{Ti}_{50}\text{Fe}_3$  SMA and their results showed that the finish temperature of austenite transformation  $A_f$  of the alloy is below 0 °C, which is lower than that of  $\text{Ni}_{50}\text{Ti}_{50}$  SMA. CHOI et al [15] investigated the stability of the B2-type structure in a series of Ti-(50-x)Ni-xFe ( $2 \leq x \leq 20$ ) alloys and found that Fe element has the ability to stabilize B2 austenite structure. However, all the literatures are related to the NiTiFe alloys with Fe substituting Ni. So far, no literatures related to the NiTiFe alloys with Fe substituting both Ti and Ni have been found.

Therefore, in the present study, the influence of Fe addition on phase transformation behavior of NiTi SMA was investigated by means of comparison among three NiTi-based alloys, whose compositions were  $\text{Ni}_{50}\text{Ti}_{50}$ ,  $\text{Ni}_{49}\text{Ti}_{49}\text{Fe}_2$ ,  $\text{Ni}_{45}\text{Ti}_{51.8}\text{Fe}_{3.2}$  (mole fraction, %), respectively, so that the mechanism of influence of Fe addition on phase transformation behavior of NiTi SMA could be revealed.

## 2 Experimental

Three different NiTi-based alloy ingots, whose nominal compositions were  $\text{Ni}_{50}\text{Ti}_{50}$ ,  $\text{Ni}_{49}\text{Ti}_{49}\text{Fe}_2$  and  $\text{Ni}_{45}\text{Ti}_{51.8}\text{Fe}_{3.2}$  (mole fraction, %), respectively, were prepared from 99.4 % Ti sheet, 99.98% Ni plate and 99.7% Fe slice (mass fraction) by vacuum arc melting. Each ingot was melted repeatedly for three times so as to achieve homogeneity. The as-cast ingots were placed into three quartz tubes, respectively. Afterward, they were flush with high purity argon gas for three times and then were evacuated to a pressure of about 0.1 Pa. Subsequently, the quartz tubes were sealed and then were placed into a furnace. The quartz tubes containing the ingots were heated for 12 h at 1000 °C and were quenched into ice water immediately after they were removed from the furnace. The ingots were taken out by breaking up the tubes once they were put into the ice water, so that they could be cooled as soon as possible.

In order to perform metallographic observation,

X-ray diffraction (XRD) analysis, differential scanning calorimetry (DSC) analysis and transmission electron microscopy (TEM) observation, a series of specimens were removed from the three ingots subjected to solution treatment by electro-discharge machining (EDM), respectively. The specimens for metallographic observation were etched in a solution with the composition of  $V(\text{HF}):V(\text{HNO}_3):V(\text{H}_2\text{O})=1:2:10$ . Subsequently, the metallographic morphologies were characterized by OLYMPUS311 optical microscope. Phase compositions of the alloys were obtained by XRD using an X-ray diffractometer (X-pert PRO). The phase transformations were analyzed by DSC employing a differential scanning calorimeter (Pyris Diamond DSC). The specimens for TEM observation were thinned by twin-jet polishing in an electrolyte consisting of 90%  $\text{C}_2\text{H}_5\text{OH}$  and 10%  $\text{HClO}_4$  by volume fraction, and subsequently were observed using a FEI TECNAI G2 F30 microscope.

## 3 Results

### 3.1 Microstructure analysis

Figure 1 illustrates metallographic morphologies of the three NiTi-based alloys. It can be seen that  $\text{Ni}_{50}\text{Ti}_{50}$  alloy is characterized by equiaxed grains and the sizes of the grains are uniform. Compared with the microstructures of  $\text{Ni}_{50}\text{Ti}_{50}$  alloy in Fig. 1(a), the grain sizes of  $\text{Ni}_{49}\text{Ti}_{49}\text{Fe}_2$  alloy are much larger and the grain boundaries of it are more curved. In addition, plenty of fine precipitates are dispersed homogeneously in the matrix of the  $\text{Ni}_{49}\text{Ti}_{49}\text{Fe}_2$  alloy. As for the  $\text{Ni}_{45}\text{Ti}_{51.8}\text{Fe}_{3.2}$  alloy, the sizes of the grains are smaller than those of the aforementioned two alloys and the precipitates with much larger size are dispersed in the matrix.

Figures 2–4 illustrate the typical TEM photographs and the corresponding selected area diffraction (SAD) patterns of the  $\text{Ni}_{50}\text{Ti}_{50}$  alloy, the  $\text{Ni}_{49}\text{Ti}_{49}\text{Fe}_2$  alloy and the  $\text{Ni}_{45}\text{Ti}_{51.8}\text{Fe}_{3.2}$  alloy, respectively. In these figures, the crystal directions labeled in the lower right corner of the insets represent the crystal zone axis, which means that all the crystal planes displayed in the diffraction pattern are parallel to the crystal zone axis. The subscripts [M] and [T] stand for the crystal planes of matrix and twin, respectively, and the subscript [M,T] represents the twinning plane, where all the crystal planes are parallel to the crystal zone axes labeled in the lower right corner of the insets. It can be seen from Fig. 2 that the matrix of the  $\text{Ni}_{50}\text{Ti}_{50}$  alloy consists of both B19' (martensite) phase and B2 (austenite) phase. Furthermore, the substructures of twins could be observed in the B19' phase, as illustrated in Figs. 2 (c) and (d). The TEM morphologies of the  $\text{Ni}_{49}\text{Ti}_{49}\text{Fe}_2$  alloy illustrated in Fig. 3 are quite different from those of the  $\text{Ni}_{50}\text{Ti}_{50}$  alloy. Based

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