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INVESTIGATE OF MICROHARDNESS AND MICROSTRUCTURE OF Ti-Ni-Nb-X (Ta AND V) SHAPE MEMORY ALLOYS

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Abstract

Conference paper

In this study, based on designed Ti-50Ni, Ti-27Ni-23Nb, and Ti-27Ni-19Nb-4X (Ta and V) SMAs were prepared using arc melting. The microhardness and microstructure of the prepared alloys were examined. The optical microscope (OM) and Scanning Electron Microscopy (SEM) images can be noted that Nb, Ta, and V addition in substitution to nickel causes a change in the microstructure morphology of TiNi Shape Memory Alloy at room temperature. The microhardness results shown that when V and Ta elements were added to SMAs, the microhardness of the alloys was significantly increased. Microhardness measurements were determined by taking the alloys from random positions. The microhardness value of equal atomic Ti-Ni SMA was found to 243 $HV_{0.3}$. This value was determined to 354 $HV_{0.3}$ in the Ti-27Ni-23Nb ternary SMA. By the addition of Ta into the Ti-Ni-Nb alloy, the microhardness value was enhanced to approximately 380 $HV_{0.3}$, additionally, moreover, by adding Vanadium element to the ternary alloy, the microhardness was increased to about 500 $HV_{0.3}$.

Keywords: *TiNiNbV; TiNiNbTa; shape memory alloy; microhardness; optical microscope.*

Ti-Ni-Nb-X (Ta ve V) ŞEKİL HATIRLAMALI ALAŞIMLARIN MİKROYAPISI VE MİKROSERTLİKLERİNİN ARAŞTIRILMASI

Özet

Konferans bildirisi

Bu çalışmada hazırlanan Ti-50Ni, Ti-27Ni-23Nb, ve Ti-27Ni-19Nb-4X (Ta ve V) şekil hatırlamalı alaşımlar ark ergitme metodu kullanılarak oluşturuldu. Hazırlanan alaşımların mikrosertlik ve mikroyapıları incelendi. Optik mikroskop (OM) ve taramalı elektron mikroskop (SEM) görüntüleri, TiNi bazlı şekil hatırlamalı alaşımlara ilave olarak katkılanmış Ta, V ve Nb elementleri ile oda sıcaklığında alınan analizleri ile mikro yapı morfolojilerinde değişiklikler gözlendi. Mikrosertlik sonuçları, şekil hatırlamalı alaşımlara V ve Ta elementleri katkılandığında bu sonuçların değerini artırdığı tespit edilmiştir. Mikrosertlik ölçümleri alaşımlardan rastgele konumlardan alınarak belirlendi. Atomikçe eşit olan Ti-Ni ikili şekil hatırlamalı alaşımlarda bulunan mikrosertlik ölçüm sonucu 243 $HV_{0.3}$ olarak bulundu. Bu değer Ti-27Ni-23Nb üçlü şekil hatırlamalı alaşımlar için 354 $HV_{0.3}$ bulundu. Ti-Ni-Nb alaşımına Ta ilavesi ile mikrosertlik değeri yaklaşık 380 $HV_{0.3}$ ye yükselmiştir. Ayrıca, üçlü şekil hatırlamalı alaşıma V elementi katkılanması sonucunda mikrosertlik ölçüm değeri 500 $HV_{0.3}$ olmuştur.

Anahtar Kelimeler: *TiNiNbV; TiNiNbTa; Şekil hatırlamalı alaşım; mikrosertlik; optik mikroskop*

1 Introduction

"Shape memory" defines the ability to memorize its shape and returning to the original shape of the plastically deformed sample by applying thermomechanical or heat [1-3]. Shape memory alloys (SMAs) have been classified as a type of smart material with different families that are based on a binary alloy. In TiNi alloys, the importance of shape memory alloys has come to light with the shape memory effect [4, 5]. NiTi-based alloy systems have been studied most extensively and are used in the greatest number of commercial applications. The equiatomic NiTi alloy has superior properties, such as superelasticity [6], high corrosion resistance [7], high fatigue strength, and biocompatibility [8, 9]. These alloys have attracted considerable attention in recent years since they can exhibit wide transformation hysteresis and excellent ductility [10-14]. The shape memory effect is a result of the martensitic transformation that occurs in plastically deforming alloy [15]. On the other hand, TiNi alloy has a crystallographic structure that depends on two different temperatures [16]. Such as high-temperature austenite (cubic crystal structure-B2) and lower temperature martensite (monoclinic crystal structure B19/). Because of this reason,

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the microhardness of the other phases may be different in shape memory alloys. It is well known that the microhardness of the austenite phase has higher than the martensite phase in TiNi-based alloys [17]. Moreover, taking into account variations in different temperatures, stress-induced austenite phase to martensite phase transformation may occur during the microhardness test [17]. Many researchers studied how thermo-mechanical treatments can affect the microstructural and crystalline structures of alloys, to manipulate certain mechanical or thermal properties [18]. Moreover, thermomechanical treatment, the effect of aging, and the addition of new elements to form such as ternary or quaternary of the Ti-Ni-based alloy are of great interest [19-21]. Therefore, to improve NiTi alloy, the Ni concentration can be reduced by replacing it with a third and fourth element, such as Ta, Nb, V, Sn. Mareci et al. reported that the substituting of Ni with Nb in NiTiNb alloy increased the resistance of NiTi shape memory alloy to localized corrosion [22]. In another study, Balci et al. found that the dendritic microstructure of the shape memory alloys prepared in different proportions were different in terms of composition, e.g., although Ti element has a higher ratio, from the EDX analysis and SEM images showed that the dendritic structure observed of in the Ni27Ti50Nb21V2 (at.%) alloy almost consist of Nb [23]. Although the thermal and microstructural properties of Ni-Ti-based alloys have been studied in large numbers in literature, there are almost no studies on the microhardness properties of NiTiNb-4Ta and NiTiNb-V4 SMAs.

This study aims to compare the microhardness and OM and SEM images of TiNi, TiNiNb, and TiNiNb-X (X =V or Ta). The alloys were produced by an arc-melting method and in the same environmental conditions. Microhardness; OM and SEM images of these alloys were examined.

2 Experimental Procedure

Ti, Ni, Nb, Ta, and V powder, with high purity (99.9%), elements were used for preparing five different blended by a mechanical mixer. Three different ratios of these powders were chosen to prepare $Ni_{27}Ti_{50}$ (named F1), $Ni_{27}Ti_{50}Nb_{23}$(named F2), $Ni_{27}Ti_{50}Nb_{19}Ta_4$(named F3), and $Ni_{27}Ti_{50}Nb_{219}V_4$ (named F4). These compositions with powder materials were pressed under $5x10^3$ kg-force to become disk pellets. They were taken by arc-melting device and then argon was added to the chamber to avoid oxidation. Samples for microscopic observation were mechanically polished and chemically etched in a solution of ($HF+NOH_3+H_2O$: 1:2:5) for approximately 5 sec. The microstructures were observed by optic Microstructure Clemex Software-Nikon Eclipse MA200 and by Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy (SEM-EDX) device. The microhardness was measured for 10 seconds under 300 g-force load using Durascan Emcotest 20 model microhardness tester. The average of five measurements taken was evaluated for the microhardness test.

3 Results and Discussion

Firstly, the percentage (at %) contents and coding of the shape memory alloys prepared for this study are shown in Table 1. More than one method can be found in the literature for Microhardness test analysis of materials. There are three principal standard test methods for expressing the relationship between hardness. These methods; Brinell, Rockwell, and Vickers [17]. In this study, the microhardness test of the shape memory alloys was made using Vickerss hardness measurement method and it was obtained from five different regions by Duramin Microhardness tester (Struers, Denmark) with a load of 30 g for 10 s. Microhardness measurements were determined by taking the alloys from random positions. The average microhardness results of the alloys are given in Table 2.

Table 1. The atomic and weight composition of alloys prepared by arc-melting method.

	Concentration (at.%)					Concentration (wt.%)				
Samples	**Ti**	**Ni**	**Nb**	**Ta**	**V**	**Ti**	**Ni**	**Nb**	**Ta**	**V**
F1	50	50	-	-	-	55.1	44.9	0	0	0
F2	50	27	23	-	-	25.9	39.1	34.9	0	0
F3	50	27	19	4	-	37.1	24.4	27.3	11.2	0
F4	50	27	19	-	4	40.2	26.7	29.7	0	3.4

Table 2. The atomic and weight composition of alloys prepared by arc-melting method.

Sample	Microhardness ($HV_{0.3}$)	Standard Deviation
F1	243	3
F2	354	1
F3	394	3
F4	508	21

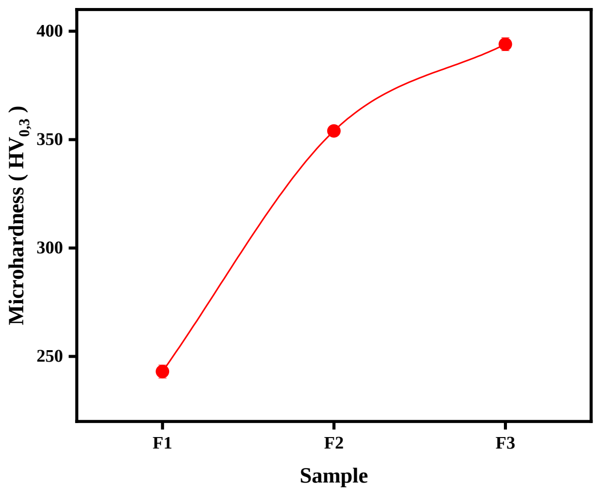

Figure 1. Microhardness measurement of NiTi based and addition different ration Nb (19%at.) and Ta (4%at.) elements.

When Table 2 examined, microhardness measurement results have increased as a result of different elements added to NiTi SMAs.

Figure 1 shows the microhardness measurement of NiTi-based and addition different ration Nb (% 19) and Ta (%4) elements. According to these results, adding by Nb element and the added Ta element at the rate of 4 percent increased the microhardness. The average microhardness measurement results were found to be 243 $HV_{0.3}$ in Ni50Ti50, 354 $HV_{0.3}$ in Ni27Ti50Nb23 ternary SMA. In another study, it was determined that the Sn element added to NiTi-based shape memory alloys in different proportions changed between 180-455 HV as a result of microhardness [24]. Also, in a study by C.Tatar et al., NiTiCu found to the microhardness measurement results of the shape memory alloy between approximately 200-500 $HV_{0.3}$ [25]. Figure 2 illustrates the microhardness results of Ni-50Ti, Ni-50Ti-23Nb, and Ni-50Ti-19Nb-4V shape memory alloys. The microhardness measurement result of the V element with 4 percent contribution was found to be 508 $HV_{0.3}$ on average. When element V was added to NiTiNb alloy, it was determined that its microhardness was the highest.

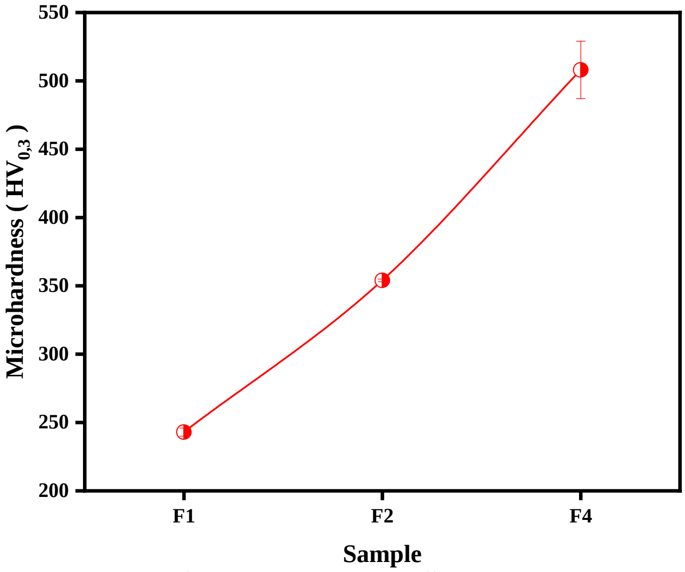

Figure 2. Microhardness measurement of NiTi based and addition different ration Nb (19%at.) and Ta (4%at.) elements.

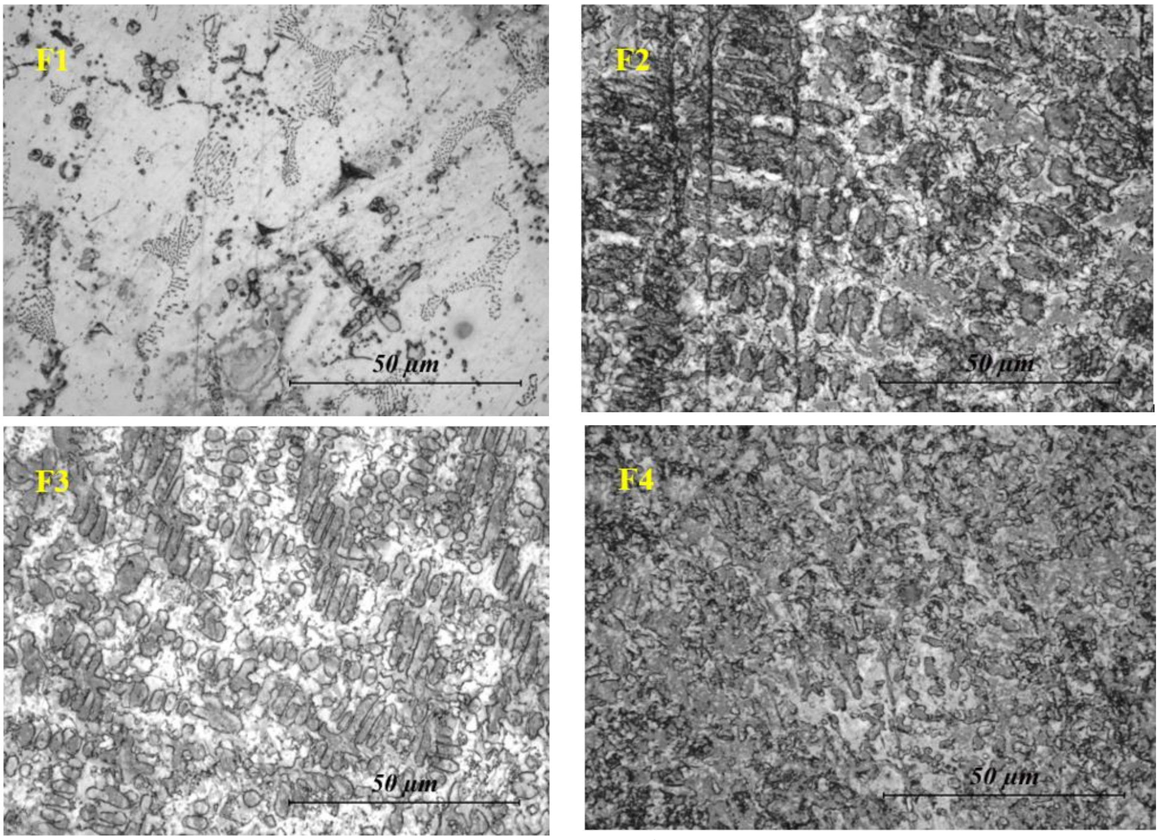

Figure 3. Optical microscope images of shape memory alloys.

Optical microstructure of NiTi shape reminder alloys is given in Figure 3 and its SEM view is given in Figure 4. The optical microstructure of the shape memory alloy with Ni and Ti elements shows that the NiTi main structure is an irregular microstructure (sample F1). The NiTiNb SMA (sample F2) structure produced by adding Nb to the structure performed a more regular dendritic solidification than the sample without Nb addition. It is clear that solidification occurs with the effect of the fourth element such as Ta and V on the structure with a much finer and smooth formation compared to the dendritic formation mechanism, as in the F3 and F4 samples. Furthermore, as can be seen in Figure 3 and Figure 4, primary dendritic arms are quite long in F2 and F3 samples. According to the EDS result, the primary dendrite arm is solidified with elements of at.63.19Nb%, at.32.63Ti, and the remainder Ni. The primary dendrite arm in the Ta added microstructure consisted of the elements at.56.89Nb, at.33.21% Ti at. It can be seen in the optical and SEM microstructure in Figure 3 and Figure 4 that it achieves a finer and homogeneous dendritic solidification with the effect of the Ta element. However, the structure of NiTiNbV SMA was quite different from other samples. Matrix structure 57.88%Ti, 31.21% Ni, 5.55%Nb and 5.36% V (at.) secondary phase 44.22%Ti, 37.99%Ni, 13.28%Nb (at.). It exhibited solidification from the 4,50V elements. It can be seen in Figure 2 that the secondary phases in the F4 sample, which exhibit a fairly homogeneous structure, also positively affect the mechanical properties of the shape memory material.

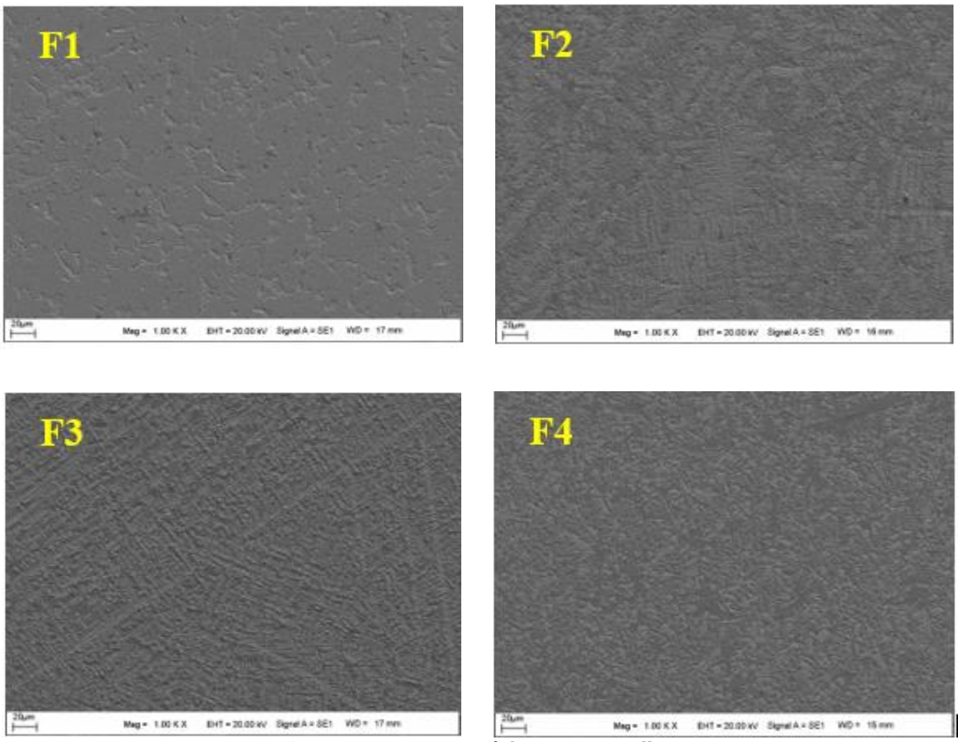

Figure 4. SEM images of shape memory alloy.

4 Conclusion

According to the obtained in NiTi-based shape memory alloys prepared, the main outcomes are as follows:

- Adding Nb, Ta, V elements to NiTi shape memory alloys increases the micro-hardness of alloy.
- When the microhardness measurement results of NiTiNb-4Ta and NiTiNb-4V shape memory alloys are compared, the NiTiNb-4V SMA has a high micro-hardness.
- The surface morphology analysis shows that the NiTiNb, NiTiNbTa, and NiTiNbV SMAs consist of irregular dendritic forms. The dendritic solidification of the alloys changes the mechanical properties of the alloy and increases its microhardness.
- The dendrite arms of NiTiNb based alloys consist of Nb-rich structure phase.

Declaration

This study titled "Investigate of Microhardness and Microstructure of Ti-Ni-Nb-X (Ta and V) Shape Memory Alloys" was presented at PCFM 2021.

Ethics committee approval is not required for this study.

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