Research Article GU J Sci 33(2): 541-546 (2020) DOI:10.35378/gujs.569401

Gazi University

Journal of Science

http://dergipark.gov.tr/gujs

Effect of Heat Treatment on the Structural Properties and Martensitic Transformation of Ni-26.5at. %Ta High Temperature Shape Memory Alloy

Koksal YILDIZ*

Firat University, Department of Physics, 23119, Elazig, Turkey

Highlights

- Heat treatment effects were investigated in the Ni-Ta alloy.
- Structural properties of the are alloy by changed heat treatment.
- Heat treatment affected transformation behaviour of the alloy.

Article Info

Received: 23/05/2019
Accepted: 29/11/2020

Keywords

Ni-Ta
Martensitic
Transformation
High temperature shape
Memory alloy

Abstract

The effect of heat treatment at 450 ºC, 550 ºC and 650 ºC temperatures for 1 hour on the structural properties and the transformation behaviour of Ni-26.5at. %Ta high temperature shape memory alloy have been examined by XRD, SEM-EDX analysis and DSC measurements. The SEM-EDX investigations showed that microstructures of all heat-treated alloy samples consisted of Ta-rich $NiTa_2$ precipitate phase and matrix. The XRD results indicated that both numbers and intensities of martensite phase reflections of the alloy highly increased as a result of heat treatment at 650 ºC. This was indication that the structural properties of the alloy were significantly affected by heat treatment at 650 ºC. The thermal measurements revealed that the high temperature shape memory behaviour of the alloy did not changed with heat treatment. However, it was seen that heat treatment performed at 450 ºC caused shifting martensitic transformation temperatures of the alloy and hence transformation hysteresis value of the alloy increased.

1. INTRODUCTION

Transformation temperatures of conventional shape memory alloys (SMAs), such as Ni-Ti and Cu-based alloys, generally do not exceed 200 ºC and shape memory alloys with transformation temperatures above 200 ºC are required for many applications in advanced technological fields, such as automotive, robotics and space industries [1-3]. Many different SMAs are currently being studied for these purposes [4-6] and the intermetallic Ni_3Ta compound, which exhibits shape memory behaviour with a martensitic transformation temperature above 300 ºC, has become a candidate material in these areas [7]. Thus, in recent years, many researchers have tried to determine the mechanism of shape memory behaviour exhibited by the Ni_3Ta compound and to examine how different factors, such as third element addition, have an influence on its shape memory behaviour and structural properties. Rudajevova and Pospíšil [8] studied shape memory behaviour of single and polycrystalline Ni_3Ta alloys by performing dilatometric measurements in non-deformed and pre-deformed states. Biffi et al. [9] investigated the effect of B content on the microstructure and martensitic transformation of this alloy by adding B element at different ratios to Ni_3Ta alloy. Kosurokova et al. [10] studied the effect of Co and Nb additions on the structural properties and phase transformation characteristics of the intermetallic Ni_3Ta compound.

The aim of this work was to examine the effect of heat treatment on the morphological and the structural properties and martensitic transformation behaviour of the Ni-26.5at. % Ta high temperature SMA first time.

*e-mail:kyildiz@firat.edu.tr

2. MATERIAL METHOD

The Ni-26.5at. % Ta SMA was produced by the arc-melting method [11]. Before heat treatment, ingot was cut in pieces under 3 groups. The cutting samples for thermal, morphological and structural analysis were heat treated at 450 ºC (NiTa-450), 550 ºC (NiTa-550) and 650 ºC (NiTa-650) for 1 hour under air atmosphere. Surface morphologies and chemical analysis of phases in NiTa-450, NiTa-550 and NiTa-650 samples were investigated by scanning electron microscope (SEM, ZEISS EVO MA10) images and energy-dispersive x-ray spectrometer (EDS) spectra. The reverse and forward transformation temperatures of samples were determined by differential scanning calorimeter (DSC) measurements taken at a heating/cooling rate of 10 °C/min in a nitrogen gas atmosphere via SETERAM TG-DSC-800 system. The structural properties of the samples were realised by x-ray diffractometer (XRD, Bruker Discover D8) using CuKα radiation at room temperature.

3. THE RESEARCH FINDINGS AND DISCUSSION

SEM images of the NiTa-450, NiTa-550 and NiTa-650 samples are displayed in Figure 1. All samples exhibit similar morphological features: small-sized and rarely dispersed precipitate phases in the matrix. The chemical compositions of the precipitates in Figure 1-(a-c) were determined by taking EDX spectra. According to EDX spectra, the precipitates observed in the NiTa-450, NiTa-550 and NiTa-650 samples are of 68.92at. % Ta, 62.2at. % Ta and 63.79at. % Ta contents, respectively. The EDX results has indicated that these small-sized grey precipitates in the NiTa-450, NiTa-550 and NiTa-650 samples is the intermetallic Ta-rich $NiTa_2$ phase as observed in as-homogenized sample [11]. The SEM-EDX analysis also reveal that the all samples do not contain another precipitate phase. Compared with as-homogenized sample, it is clearly seen that the heat treatment at low temperatures for 1 hour do not have a significant effect on the morphological properties of Ni-26.5at. % Ta high temperature SMA. Differently, the Ta contents in the Ta-rich $NiTa_2$ precipitate phase observed in the heat treated samples are lower than that in as-homogenized sample (75.75at. % Ta) [11].

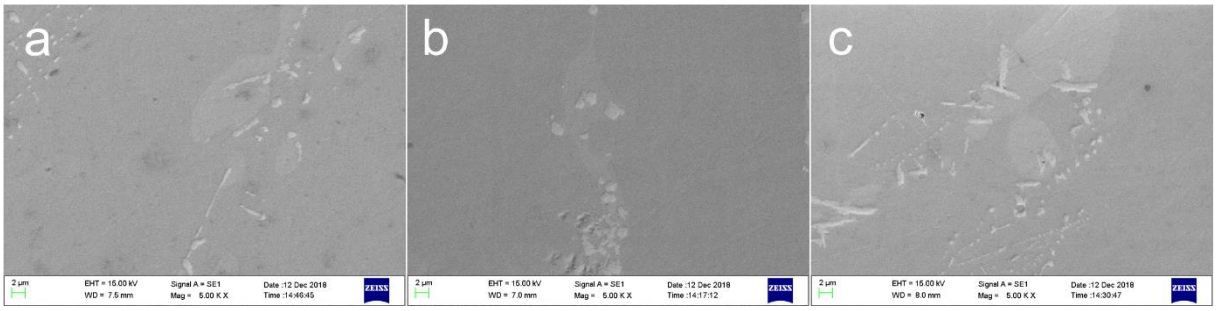

Figure 1. *SEM images of (a) NiTa-450, (b) NiTa-550 and (c) NiTa-650 samples*

Figure 2 indicates XRD patterns of the as-homogenized [11], NiTa-450, NiTa-550 and NiTa-650 samples. The peaks belonging to the monoclinic martensite (PDF: 01-073-7070) and tetragonal austenite (PDF: 00-018-0893) Ni_3Ta phases are indexed on the patterns in Figure 2-(a-d). From Figure 2, it is clearly seen that the heat treatment at low temperatures for 1 hour cause radical changes in the structural properties of the Ni-26.5at. % Ta high temperature SMA. The main diffraction peak $(004)_M/(112)_T$ located at $2\theta \approx 42.9^o$ in as-homogenized sample (Figure 2-a) disappeared completely as a result of the heat treatment at 450 ºC (Figure 2-b) and 550 ºC (Figure 2-c). And also, intensities of $(012)_M/(101)_T$ at $2\theta \approx 27.62^o$ and $(-305)_M/(006)_T$ at $2\theta \approx 76.98^o$ peaks highly increased in the XRD pattern of NiTa-450 sample, as shown in figure 2-b. In the XRD pattern of NiTa-550 sample shown in Figure 2-c, the intensities of $(-104)_M$ and $(-122)_M/(103)_T$ peaks at $2\theta \approx 43.36^o$ and $2\theta \approx 44.22^o$, respectively, are higher than the others. However, the most significant changes in the structural properties of Ni-26.5at. % Ta high temperature SMA occurred in NiTa-650 samples (Figure 2-d). Especially, as can be clearly seen in Figure 2-d, the peak intensities of the monoclinic martensite phase dramatically increased. The main diffraction peak for the NiTa-650 sample is $(201)_M$ with reflection at $2\theta \approx 43.98^o$. It is understood from the XRD pattern in Figure 2-d that the structural properties and martensitic crystal orientation of the Ni-26.5at. % Ta high temperature SMA changed significantly as a result of heat

treatment at 650 ºC compared with other samples. Consequently, due to the peak numbers belonging to the monoclinic martensite phase in Figure 2-d, it can also be claimed that the volume fraction of the martensite phase in NiTa-650 sample has increased. Additionally, there is no diffraction peaks belonging to the intermetallic $NiTa_2$ precipitate phase in the XRD patterns of all samples.

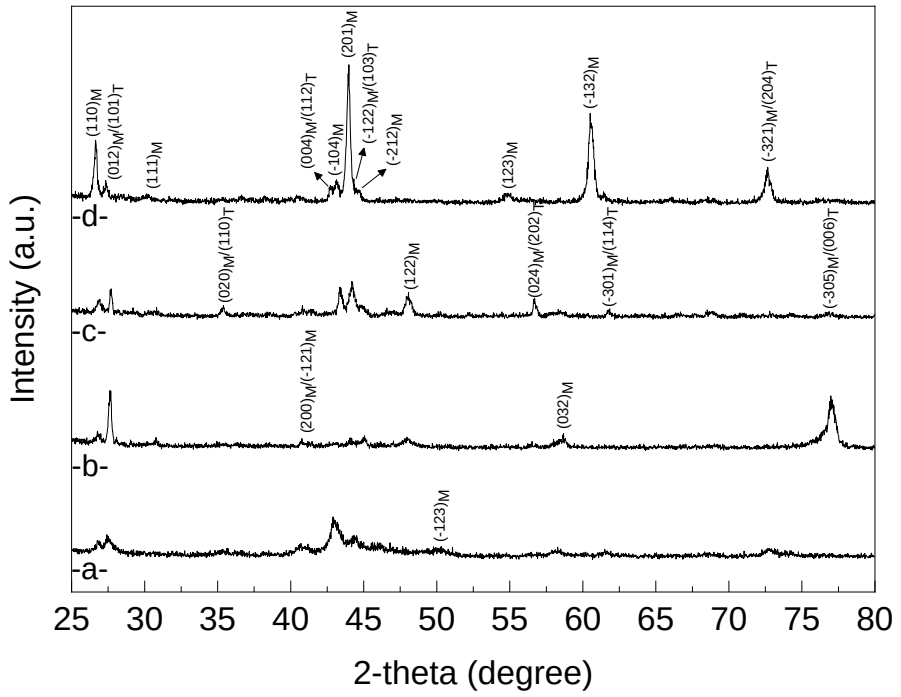

Figure 2. *XRD patterns of (a) as-homogenized [11], (b) NiTa-450, (c) NiTa-550 and (d) NiTa-650 samples*

The effect of heat treatment performed at different temperatures for 1 hour on the transformation temperatures of the Ni-26.5at. % Ta high temperature SMA were examined by taking DSC scans on heating and cooling curves. DSC scans of NiTa-450, NiTa-550 and NiTa-650 samples between 150-400 ºC are plotted in Figure 3-(a). Reverse (A_s and A_f) and forward (M_s and M_f) transformation temperatures of the samples and transformation hysteresis (A_f-M_f) values obtained from the DSC scans in Figure 3-(a) are presented in Table 1. It is known from previous work [11] that the Ni-26.5at. % Ta alloy exhibits high temperature shape memory behaviour with fully completed martensitic transformation above of 220 ºC. However, as can be clearly seen in Table 1 and Figure 3-(b), the heat treatment performed at 450 ºC caused shifting martensitic transformation temperatures of the Ni-26.5at. % Ta high temperature SMA and as a result of this, martensitic transformation temperature of the alloy decreased below of 200 ºC. From this, it has been understood that, among different heat treatment temperatures, the heat treatment temperature of 450 ºC is of the most dramatic effect on the transformation characteristic of the Ni-26.5at. % Ta high temperature high temperature SMA. Additionally, the NiTa-450 sample possesses higher transformation hysteresis value of 120.2 ºC than those of the NiTa-550 and NiTa-650 samples. On the other hand, the transformation hysteresis values of the all heat treated samples are higher than that of as-homogenized sample [11], as given in Table 1. Herewith, it is obvious that the heat treatment performed at temperatures of 450 ºC, 550 ºC and 650 ºC lead to increase in the transformation hysteresis of the Ni-26.5at. % Ta high temperature SMA and this reveals that the Ni-26.5at. % Ta alloy can have different application areas with performing different heat treatments [12].

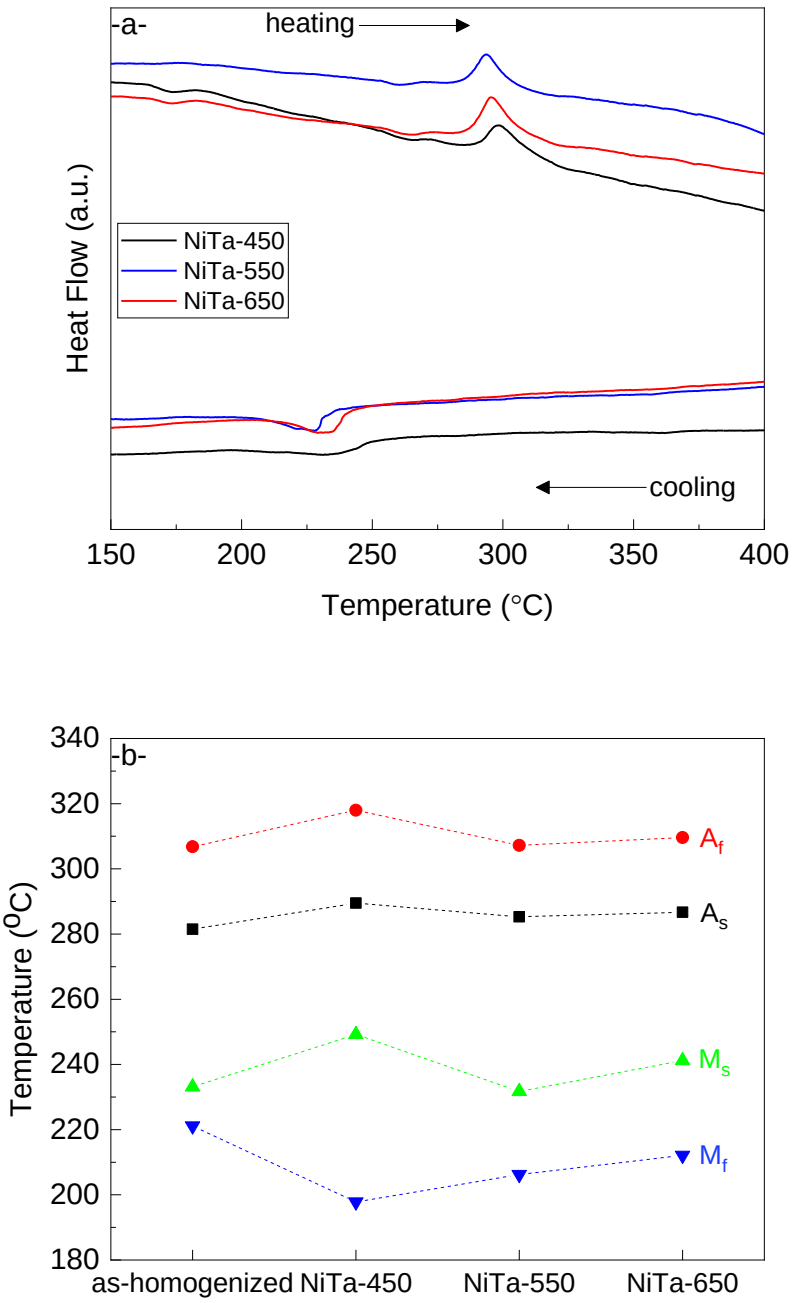

Figure 3. *(a) DSC scans of NiTa-450, NiTa-550 and NiTa-650 samples. (b) Variation of transformation temperatures of as-homogenized [11], NiTa-450, NiTa-550 and NiTa-650 samples*

In the last years, among many high temperature SMAs, the Ni-Ta alloy has increasing interest and its structural and morphological properties, martensitic transformation behaviour and shape memory effect has been studied depending on its composition [11], by adding third alloying elements [9,10] and under pre-deformed states [7,8]. However, the Ni-Ta alloy system has some disadvantages: cracks in microstructure [7] and tetragonal austenite phase at room temperature with high volume fraction [13]. It has been seen that the crack problems in the Ni-Ta alloy could be tackled with changing homogenization conditions [11]. When examined XRD pattern in Figure 2-(d) belonging to the NiTa-650 sample, it is now though that high volume fraction of austenite phase in the Ni-Ta alloy at room temperature may be decreased with

performing heat treatments under different conditions, because both the numbers and the intensities of the tetragonal austenite phase reflections in Figure 2-(d) are low compared with those in the XRD patterns of other samples in Figure 2-(a-c). Briefly, it is seen that the heat treatment has a significant effect on the structural properties of the Ni-Ta high temperature SMA, but it is needed further studies in detail.

Table 1. *Reverse and forward transformation temperatures and transformation hysteresis values of as-homogenized [11], NiTa-450, NiTa-550 and NiTa-650 samples*

	A_s (°C)	A_f (°C)	M_s (°C)	M_f (°C)	A_f-M_f (°C)
As-homogenized	281.5	306.8	233.1	221.1	85.7
NiTa-450	289.5	318	249.2	197.8	120.2
NiTa-550	285.3	307.2	231.7	206.2	101
NiTa-650	286.7	309.6	241.2	212.1	97.5

4. RESULTS

The results obtained from this study can be summarized as below:

- Morphological investigations show that the microstructural features of the Ni-26.5at. % Ta high temperature SMA are not influenced by heat treatments performed at 450 °C, 550 °C and 650 °C temperatures for 1 hour. All samples have same microstructural characteristics: the matrix and rarely dispersed intermetallic Ta-rich $NiTa_2$ precipitate in the matrix.
- Structural analysis reveal that the crystallographic properties of the Ni-26.5at. % Ta high temperature SMA are very sensitive to heat treatment, especially performed at 650 °C. In this heat treatment temperature, it is observed that whereas both the numbers and the intensities of tetragonal austenite phase reflections decrease, those of monoclinic martensite phase reflections increase highly.
- Thermal examinations indicate that the martensitic transformation characteristic of the Ni-26.5at. % Ta high temperature SMA can be changed depending on heat treatment temperature. With performing heat treatment especially at 450 °C, it is seen that both its martensitic transformation temperatures can be lower and its transformation hysteresis can be wider compared with the others.

ACKNOWLEDGEMENTS

This work was supported by Scientific Research Projects Coordination Unit of Firat University under Project number: FF.16.36. The author also thanks to Professor Soner ÖZGEN (Firat University) for his technical support under Project number: FF.15.17 and Dr. Fatih SEMERCİ (Kirklareli University) for DSC measurements.

CONFLICTS OF INTEREST

No conflict of interest was declared by the author.

REFERENCES

[1] Firstov, G.S., Van Humbeeck, J. and Koval, Y.N., "High-temperature shape memory alloys: Some recent developments", Mater. Sci. Eng. A, 378: 2-10, (2004).

[2] Gama, J.L.L., Dantas, C.C., Quadros, N.F., Ferreira, R.A.S. and Yadava, Y.P., "Microstructure-mechanical property relationship to copper alloys with shape memory during thermomechanical treatments", Metall. Mater. Trans. A, 37A: 77-87, (2006).

[3] Celik, H., Aldirmaz, E. and Aksoy, I., "Effects of deformation on microstructure of Cu-Zn-Ni alloy", GU J. Sci., 25: 337-342, (2012).

[4] Karakoc, O., Hayrettin, C., Evirgen, A., Santamarta, R., Canadinc, D., Wheeler, R.W., Wang, S.J., Lagoudas, D.C., Karaman, I., "Role of microstructure on the actuationfatigue performance of Ni-rich NiTiHf high temperature shape memory alloys", Acta Mater. 175: 107-120, (2019).

[5] Milhorato, F.R., Mazzer, E.M., "Effects of aging on a spray-formed Cu-Al-Ni-Mn-Nb high temperature shape memory alloy", Mater. Sci. Eng. A, 753: 232-237, (2019).

[6] Paulsen, A., Frenzel, J., Lamgenkämper, D., Rynko, R., Kadletz, P., Grossmann, L., Schmahl, W.W., Somsen, C., Eggeler G., "A kinetic study on the evolution of martensitic transformation behavior and microstructures in Ti-Ta high-temperature shape-memory alloys during aging", Shap. Mem. Superelasticity, 5: 16-31, (2019).

[7] Firstov, G.S., Koval, Yu.N., Van Humbeeck, J. and Ochin, P., "Martensitic transformation and shape memory effect in Ni_3Ta: A novel high-temperature shape memory alloy", Mater. Sci. Eng. A, 481-482: 590-593, (2008).

[8] Rudajevová, A. and Pospíšil, J., "Shape memory behavior of a Ni_3Ta alloy pre-deformed in compression", Mater. Sci. Eng. A, 527: 2900-2905, (2010).

[9] Biffi, C.A., Agresti, F., Casati, R. and Tuissi, A., "Ni_3Ta high temperature shape memory alloys: effect of B addition on the martensitic transformation and microstructure", Mater. Today: Proceedings, 2S: S813-S816, (2015).

[10] Kosorukova, T.A., Firstov, G.S., Koval, Yu.N., Van Humbeeck, J. and Zhuravlev, B., "Phase transformations and shape memory behavior in Ni_3Ta-based intermetallics", Mater. Today: Proceedings, 2S: S793-S796, (2015).

[11] Yildiz, K., "Thermally induced martensitic transformation and structural properties in Ni-Ta high temperature shape memory alloys", Eur. Phys. J. Plus., 134: 11, (2019).

[12] Chen, H.R., Shape Memory Alloys: Manufacture, Properties and Applications, Nova Science Publishers, New York, (2010).

[13] Kosorukova, T., Firstov, G., Noël, H. and Ivanchenko, V., "Crystal structure changes in the Ni_3Ta intermetallic compound", Chem. Met. Alloys., 6: 196-199, (2013).