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Research Article

Development and Characterization of $Ni_{55}Ti_{45}$ Shape Memory Alloy for Biomedical Applications

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Abstract: In this study, a shape memory alloy of $Ni_{55}Ti_{45}$ was developed by Vacuum Arc melting technique under a vacuum of 10^{-5}mbar. It was followed by homogenization in Nabertherm Tube furnace at a temperature of 1000°C for 54 hrs and subsequent furnace cooling. A series of tests had been performed in the laboratory in order to characterize the behavior of this alloy. Scanning electron microscopy results confirmed the presence of primary NiTi and secondary Ni_3Ti phases respectively. X ray diffraction confirmed the BCC (body centered cubic) and HCP (hexagonal close packed) structures of NiTi and Ni_3Ti respectively. In differential scanning calorimetry, no transformation was observed in the temperature range of 0°C-1200°C. This is indicative of the fact that the transformation temperature for shape memory effect lies below this temperature range.

Keywords: Biomaterials, shape memory, nickel-titanium, X-ray diffraction; scanning electron Mmicroscope

1. INTRODUCTION

Shape memory alloys are excellent materials having incredibly unique properties. They have unique ability to regain their previous shape by the application of some thermo mechanical procedures [1]. They satisfy the requirements of excellent corrosion resistance, good biocompatibility; super-elasticity and stable shape memory phase [2, 3, 5]. Among all the shape memory alloys NiTi based alloys play a significant role in various applications [4, 5].They possess quite unique properties compared to other shape memory alloys making them suitable for their use in various dynamic applications [6].Their major areas of applications are in biomedical industry where they are used as orthopedic, cardiovascular and dental implants and in aeronautical and automobile industry [1]. NiTi shape memory alloy with Ni 55% and Ti 45% named as Nitinol is a very attractive material having low elastic modulus and very high toughness [3]. Gil et al. [8] studied this alloy as orthodontic arch wires to investigate the mechanical properties of it. Actually the super-elastic behavior of Nitinol wires can be explained in the way that on unloading they return to their original dimensions before deformation. This is the only alloy composition that can utilize the super elastic properties. The sensitivity to nickel content in this alloy composition is much important to use this alloy as an orthodontic material [3]. Miyazaki et al. [5] studied that the alloy possesses much high strength and low elastic modulus as compared to stainless steel. He concluded in his study that the instrument made of this alloy can be successfully used in the preparation of root canals. This alloy is majorly used for joining of fractured bones, for the replacement of hard tissues, as dental arch, as stents in body arteries and many more [4]. An important point in this regard is that for biomedical applications, implants made of NiTi must have martensitic transformation temperature below the normal human body temperature [2].

In the current research, $Ni_{55}Ti_{45}$ shape memory alloy has been developed by vacuum arc meltig technique and then characterized by XRD, SEM and DSC. The objective of this work is to determine its specific properties so that applications of the said alloy can be recommended.

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2. MATERIALS AND METHODS

An alloy of $Ni_{55}Ti_{45}$ was developed in the current study. All constituents were weighed to an accuracy of 0.001g and thoroughly washed with acetone to avoid any contamination. Melting was carried out in vacuum arc melter evacuated to attain a vacuum of 10^{-5} mbar and flushed with high purity Argon many times to ensure contamination/oxidation free melts. . Each alloy was kept molten for 4 minutes, allowed to solidify, overturned and remelted 3 times to ensure homogeneity. Alloy buttons weighing 10g each were prepared. Weight losses during melting were found to be about 1.0% which lies within the limits of the equipment. The alloy samples were subjected to homogenization in Nabertherm Tube furnace at a temperature of 1000°C for 54 hrs. Samples were furnace cooled. Mounting was done in a Mounting machine Leco PR-25 and it took 8 minutes for complete mounting. After grinding on emery papers up to 1200μ, samples were polished and etched. Etching was done by using pure Nitric acid, HNO_3. LecoTM LX31 Inverted Metallurgical Optical Microscope and S-3000N Scanning Electron microscope were used to investigate the microstructures of the samples. The phase transformation temperature was measured using a Differential Scanning Calorimeter (DSC) (LINSEIS STA PT-1600). Specimens were analyzed by DSC at above room temperature to 1200°C with scanning rate of 100° C/15min till 700° C and then 100°C/5min till 1200°C. The heat

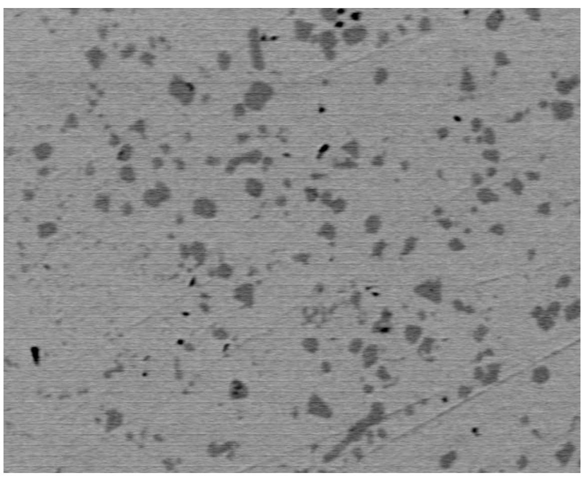

Fig. 1 (a). Microstructure of as cast NiTi alloy at 500X (Scanning Electron microscope).

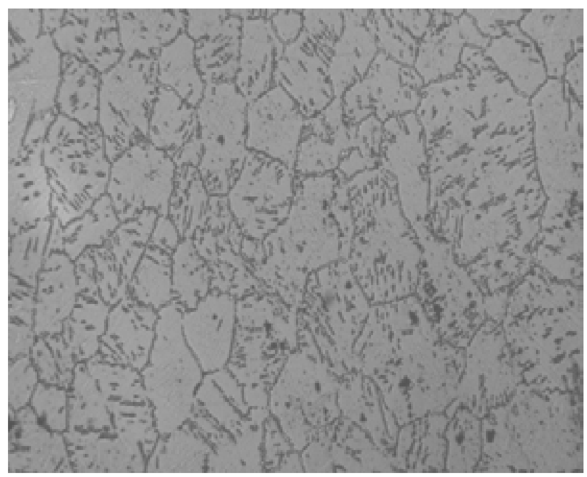

Fig. 1 (b). Microstructure of Homogenized NiTi alloy at 200X (Optical microscope).

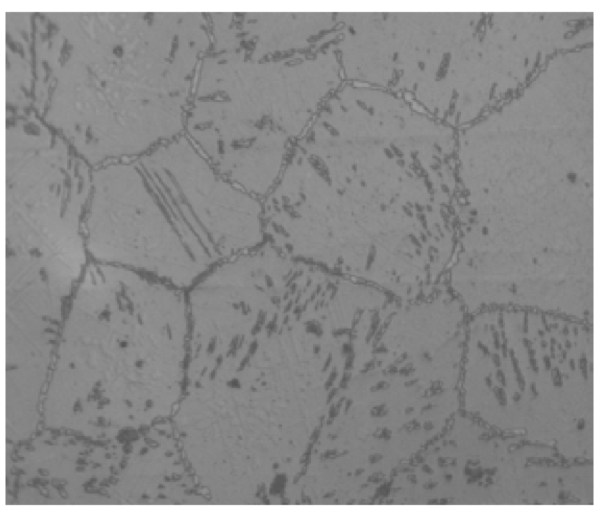

Fig. 1(c). Microstructure of Homogenized NiTi alloy at 500X (Optical microscope).

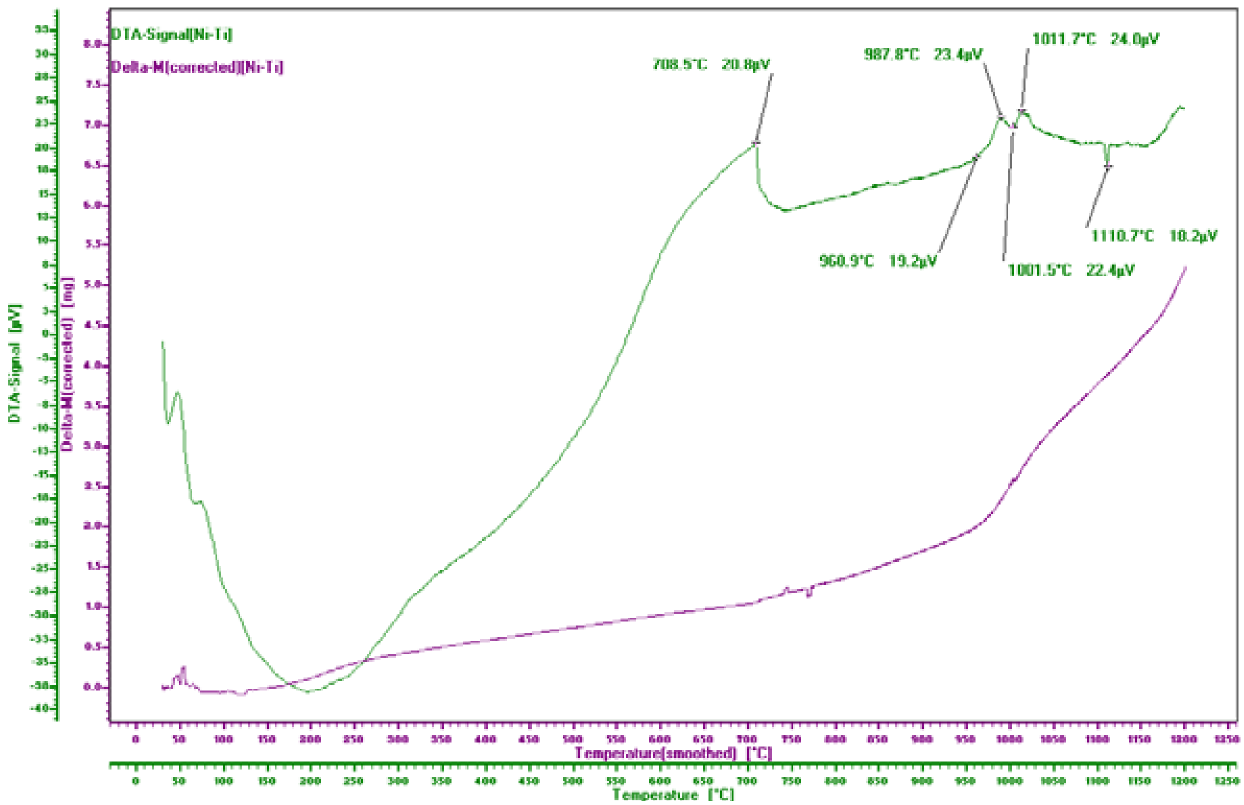

Fig. 2. DSC Thermogram of Homogenized NiTi alloy.

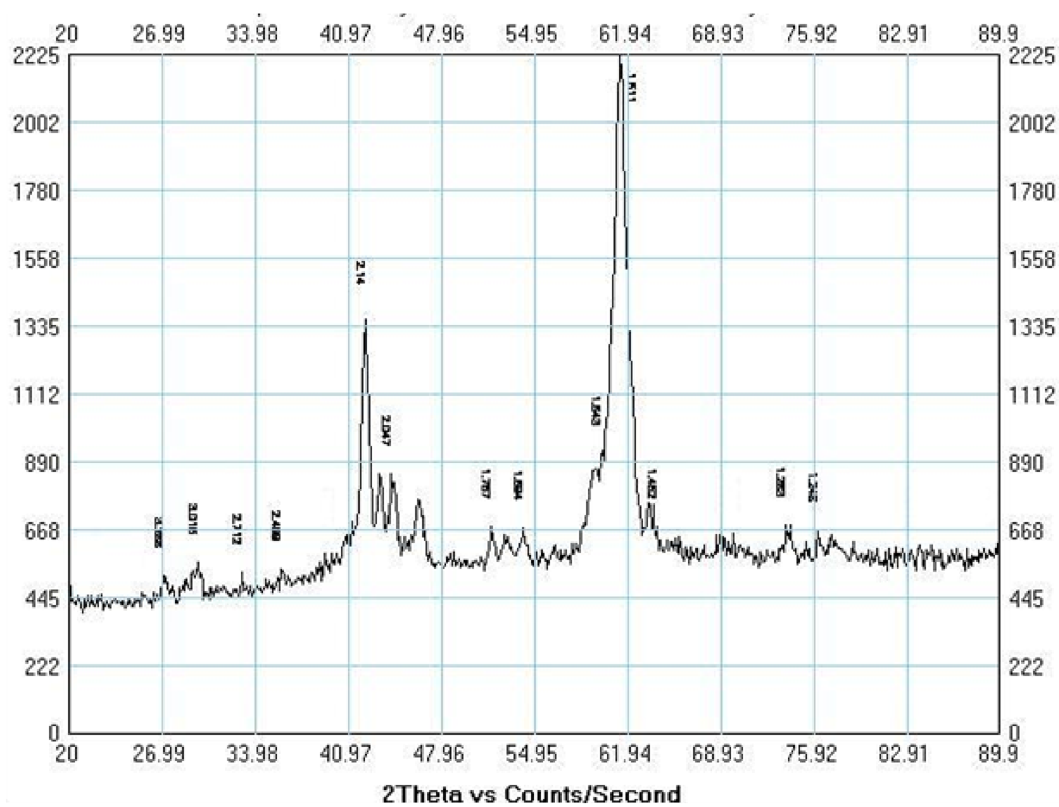

Fig. 3. XRD pattern of Homogenized NiTi alloy.

Table 1. Structure and Lattice parameters of the compounds detected in Ni-Ti alloy.

Compound	Structure	Lattice parameter (a*c)		Lattice parameter (a**c)		References
NiTi	Cubic(BCC)	3.02		3.01		Semenova. E.L.Kudryavtsev.Yu.V.J.Alloys,Compds.203.165-1(1994)
Ni_3Ti	Hexagonal	5.12	8.188	5.093	8.276	Duwez and Taylor,J.Metals188,Trans,11736(195)

(*) Calculated in this investigation
(**) Reported in the literature

curve was obtained in the temperature range of 750-1200°C and the phase transformations occurring in this temperature range was analyzed. X-Ray Diffraction sample was carried out by using Bruker D-8 equipped with monochromatic Cu k-α radiation having wavelength of 1.54060Å and Ni filter was employed. Diffraction patterns were interpreted using JCPDS cards.

3. RESULTS AND DISCUSSION

The microscopic examination of the sample of NiTi alloy under investigation revealed that in the as cast and Homogenized Ni-Ti alloy sample, the microstructure shows two phases i.e., NiTi and Ni_3Ti as depicted in Fig. 1 (a, b). In homogenized Ni-Ti alloy sample, the Ni_3Ti precipitates have grown along the grain boundaries in NiTi matrix. The phase with a light contrast is the primary phase i.e., NiTi whereas, the phase with a dark contrast is identified as Ni_3Ti as shown in Fig. 1. Optical microscopy results are shown in figure 1 (c) which is in good comparison with Fig. 1 (a, b). NiTi is the major phase in the alloy that's why it is seen as the base in the microstructure that is of lighter contrast while Ni_3Ti is the secondary phase present at the grain boundaries and clearly visible in the microstructure as a darker contrast. The DSC curve showed transformation at 708.5^{0}C due to variation in heating rate. The exothermic reaction at 987.8°C corresponds to the presence of NiTi compound. The endothermic reaction at 1110.7°C corresponds to the presence of Ni_3Ti Compound. The transformation at 1011.7 °C lying in the middle of both transformations is due to presence of secondary precipitates as depicted in Fig.2. The two phases NiTi and Ni_3Ti were identified from the X-Ray diffraction data. Numerous crystalline peaks were observed at various angles. The major peaks observed at 2θ = 42.17, 42.9, 52.3 and 61.94 degrees represent NiTi that is the primary phase while the peaks observed at 2θ values other than these represent secondary phase i.e. Ni $_3$Ti. The results are showed in table 1. This is in confirmation with the phase diagram of nickel and titanium and results of Microscopy depicted by Fig.3. Lattice parameters of the compounds were calculated from available JCPDS data cards (Semenova, E.L. Kudryavtsev, Yu. V. J. Alloys Compds. 203. 165-1 (1994) and Duwez and Taylor, J. Metals 188, Trans, 1173-6 (1950)

4. CONCLUSIONS

The studied shape memory alloy developed by vacuum arc melting technique exhibit the two phase's presence of primary NiTi and secondary Ni_3Ti phases respectively. Both phases are confirmed by scanning electron microscopy and optical microscopy results. X-ray diffraction confirmed the BCC and HCP structures of NiTi and Ni_3Ti respectively. Phase transformation temperature determined by differential scanning calorimetry was found to lie below 0^{o}C-1200^{o}C. As the transformation temperature is below normal human body temperature so this alloy is a successful material for dentistry. On the basis of all the results found in this work, It is recommended that this alloy can be successfully used in the biomedical applications specifically in dentistry.

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