Phase Investigation of Nitinol SMA According to Melting Method

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Abstract

In this work investigation of phase(s) obtained in just melted alloy using x-ray diffraction XRD patterns was done. Many research articles suggested melting method to prepare Nitinol alloy in vacuum induction furnace, or vacuum arc furnace supported with stirring device, to prevent contaminants and oxidation. But because of its expense, four experimented method were utilized. First one is under cover in atmospheric pressure. Second method just like the first but at higher temperature. The third was done using evacuated capsules and the fourth was done using vacuum plasma spraying system with modulation. X-ray diffraction shows different phases according to procedure type.

Key Words: SMA, Shape Memory Alloy, Memory Alloy, X-Ray Diffraction.

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Introduction

Shape memory effect, a property of SMAs, is attributed to the thermo-elastic martensitic transformation and reverse martensitic transformation between the original phase and martensite phase. As martensitic transformations involve a characteristic of reversible transformation, which are deformation with no dislocation slip but twinning, the deformation behavior of alloys which is related to martensitic transformation is different from ordinary metals and alloys. SMA is smart material that exhibits shape memory. This occurs through a solid-state phase change bymolecular rearrangement. The two phases that occur in SMA are martensite and austenite. At highertemperatures, the material is in the austenite phase. As the temperature is lowered, the material changes to the martensite phase and grows until sufficiently low temperatures. This unusual characteristic of SMA actuators has a wide variety of applications in control systems, beside conventional types such as electric, hydraulic, and pneumatic actuators. [1] Capability for super elastic deformation (pseudoelasticity) and for reversion to previously defined shape were deformed and then heated past a set transformation temperature. These abilities stem from reversible displace transformation between symmetry-related solid states. In case of pseudoelasticity, transformation occurs between a high temperature, high-symmetry austenite phase and a low-temperature, low-symmetry marten site phase. Pseudelastic behavior arises when the material is deformed at a temperature sufficientlyabove the transformation temperature. [2]

The outstanding super elastic behavior of nickel titanium (NiTi) shape memory alloy (SMA) has found many important applications in areas, such as medical surgery, micro electromechanical system (MEMS) and even as a material for train wheels or as contact tires on train wheels [3].

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TiNi-based SMA has been widely applied for biomedical uses, since it has superior corrosion resistance, biocompatibility and excellent shape memory property by comparing with other alloys and metals. Hence, they are mainly used in medical field for many applications, for example, orthodontic wire, artificial heart valves, bone plate, artificial joints, etc. It seems that TiNi-based SMA is quite good for the biomedical application. [4]

Materials and Experimental Work

The first step in this work was the selection of starting materials, so a highly pure metals of purity (99.99%) were used to deals with accurate physical properties of Titanium and Nickel as given in table (1)

Table 1. The physical properties of Ti and Ni

Element	Atomic Number	Atomic Weight	Density (g/cm^3)	Melting Point (°C)
Titanium	22	47.9	4.5	1680
Nickel	28	58.71	8.9	1453

According to the phase diagram of NiTi shape memory alloy fig (1), the melting temperature of the equiatomic and near equiatomic of NiTi was 1310°C. [5].

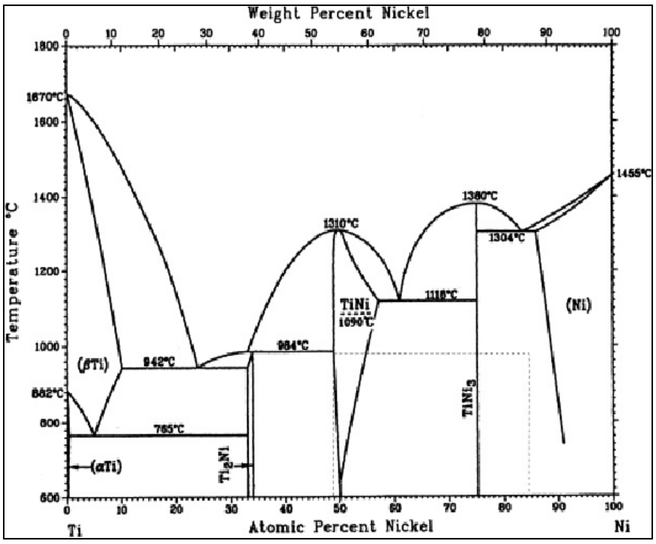

Fig. 1. Phase diagram of Nickel and Titanium metals.

The second step was to express the composition (concentration) of an alloy in terms of its constituent elements, using weight percent way, [6] for an alloy that contains two hypothetical atoms, the concentration of Ni in wt% C_{Ni}, is denoted as:

$$C_{Ni} = \frac{m_{Ni}}{m_{Ni} + m_{Ti}} \times 100\% \qquad (1)$$

Where: m_{Ni} and m_{Ti} represent the weight (or mass) of Ni and Ti respectively, the concentration of Ti would be computed in an analogous manner.

The third step was mixing the alloy component and forming as one centimeter diameter disk, mixing was done using two dimension mixer for 24 hours and forming was done using ordinary disk maker under 5000 Kg/m^2 press to avoid any air gabs.

Melting Experiments

Because of reactivity of the titanium in the alloy all melting methods must be in vacuum or in an inert atmosphere.

Procedure No.1

The samples desk putted here in alumina crucible and covered by two layers, 1st was graphite powder layer, and the 2nd was thermal cement as shown in fig. (2).

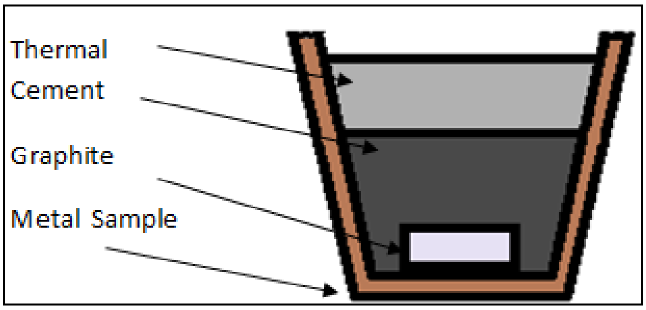

Fig. 2. Alumina Crucible with three layers covered Ni-Ti metal sample

Then the crucible inserted inside a box furnace at 1310°C for two hours ,finally the melted alloy inspected by x-ray diffraction (XRD) to determine the phase(s) type(s) fig(3), in this experiment the phases appear here are Ni_3Ti ,supposing apart of Ti interacting with carbon or oxygen of the furnace environment at the setting temperature during holding time.

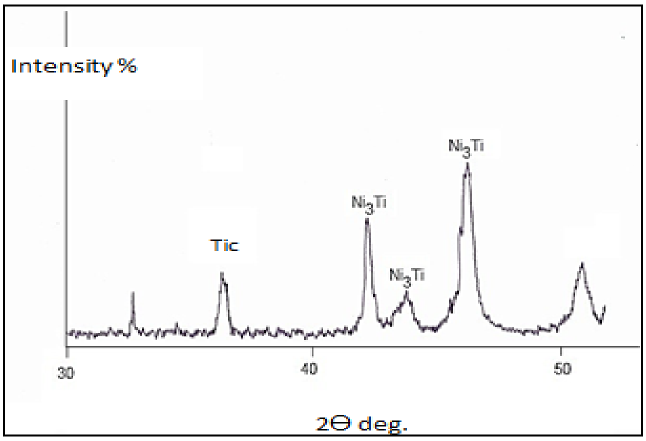

Fig. 3. X-ray diffraction pattern of Procedure one.

Procedure No.2:

As in the No.1 Procedure but the difference here is the temperature and time of melting process.

1400°C was set for one hour, from fig (4) one can see the appearing of new form of Ni_xTi_x phases like NiTi and Ti_2Ni in addition to Ni_3Ti ,this is may be based on low viscosity of melted alloy comparing with Procedure No.1.

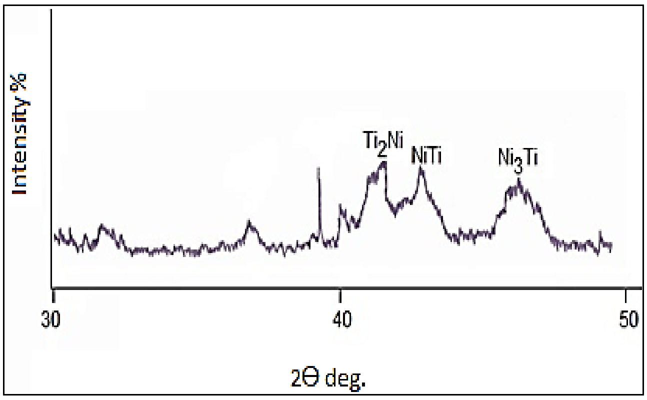

Fig. 4. X-ray Diffraction pattern ofProcedure two.

Procedure No.3:

In this experiment an evacuated quartz tube with 15 mm diameter was used as shown below fig (5):

I. Use piece of tube with length of 20 cm.
II. Weld one end of it.
III. Put the pre formed disk inside the tube.
IV. Evacuate the tube to 10^{-7} bar using two stages vacuum system (rotary and diffusion vacuum pumps).
V. Weld the other end of the tube.

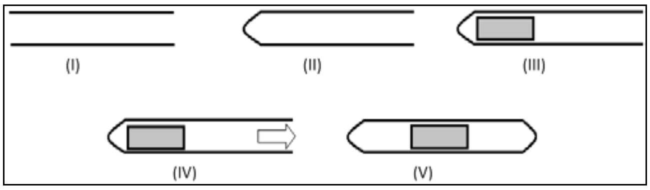

Fig. 5. Steps of preparation evacuated quartz tube container of Ni-Ti metals sample.

After that the quartz tube putted in electric tube furnace at 1310°Cfor one hour. Fig. (6) shows the XRD pattern of alloy and appearance of NiTi phase.

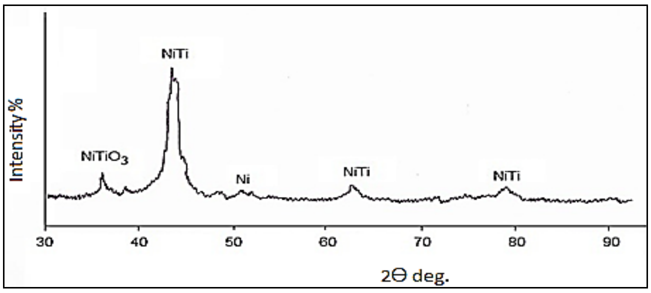

Fig. 6. X-ray diffraction pattern of Proceure three

Procedure No.4:

This is the longest experiment because of special design of melting crucible and high technology of plasma spraying system (robot programming, computerize mixing of plasma gases, three stages of vacuum system, operator experience required). The crucible (made by machining of graphite rod) has two sticking parts, 1st is melting part and the 2nd is molding part fig. (7). Then it is fixed before the plasma gun and rotated with slow velocity. The distance between the plasma jet and the cast is (15 cm), melting time (4 min) and the vacuum atmosphere is (70m bar). [7]X-ray diffraction pattern of this experiment product fig(8) shows few amount of NiTi crystalline phase only which can be increased by crystal growth heat treatment to unique the alloy properties.

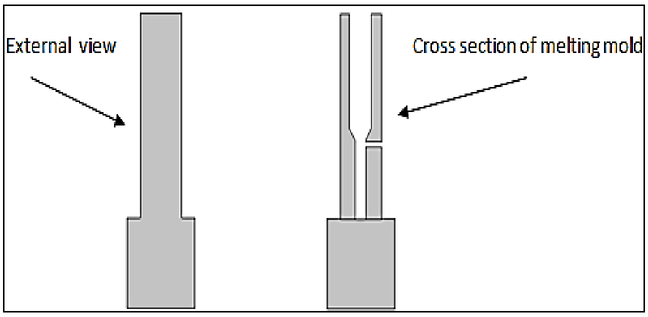

Fig. 7. Melting mold of Procedure four.

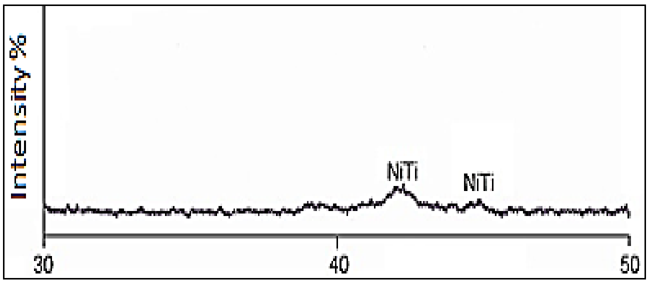

Fig. 8. X-ray diffraction pattern ofProcedure four.

Conclusions

From the XRD data of the present work. It can be concluded that:

1. The most successful way in this paper to prepare NiTi alloy is by plasma spraying system.
2. the vacuum induction furnace is still the best way to prepare SMA because of:

a) low cost comparing with VPS.
b) Stirring option of crucible.
c) Observation and handling options inside the furnace.

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