**Microstructure and Properties of Shape Memory NiTi Alloy**

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**Abstract.** Many years ago, the shape memory alloys properties of Nickel Titanium (NiTi) were first discovered in the early 1960s, as shape memory alloys had many applied applications, in which oxidation problems were not of concern for the most part. However, over the past decades, NiTi alloys have been increasingly considered in external and internal biomedical devices, for example cardiac stent wires, orthodontics, vascular and bone fractures, fixing plates and screws, self-expanding urinary tracts. The aim of the research is to study the effect of the elements molybdenum and zirconium on the shape memory alloys. When adding small amounts of zirconium leads to a smoothing of the granular size, when adding Molybdenum improves the hardening process. The alloy was prepared from primary powders of nickel and titanium, using metallic powder technology, under pressure of 800 MPa. Then the sintering method was carried out in a 4-10 tor vacuum at 950 °C. The results upon XRD analysis revealed that NiTi were completely changed into NiTi (both cubic and mono phases) and Ni3Ti phase. The samples are ground in a dry atmosphere and the samples are polished after sintering. Optical microscopy, x-ray diffraction techniques. hardness test was conducted using Vickers hardness machine. It was observed that as the Zr content is increased, the hardness values, in VPN, increased. For instance, equi at 30% of Ni Ti had a hardness of 127.05 which increased significantly as Zr content was increased to 20 at %. This is mainly observed due to precipitation hardening which occurs due to the presence of multiple phase in alloy D. Increase in hardness also suggests that the workability. It was observed that as the Mo content is increased, the hardness values, in VPN, increased. For instance, Ni Ti had a hardness of 127.05 which increased was increased to 40at%. This is mainly observed due to precipitation hardening which occurs due to the presence of multiple phase in alloy G. Due to molybdenum, Optical microscopy reveals surface characteristics such as open pores and grain borders, as well as the distinction between the phases NiTi and Ni3Ti.

1. **Introduction**

The growing interest in NiTi (SMAs) appears to have entered many engineering and medical fields as they possess a shape memory alloys effect and superior flexibility. In general and noticeable, as the phase change temperature and mechanical possessions are the two critical factors affecting the application of NiTi in engineering and medicine.\[1\] Shape memory alloys are known as basic and unique properties, which are the ability to remember and recover from large strains without permanent deformation. Shape memory alloys can exist in two different temperature-dependent crystal structures (phases) called the low temperature phase (Martensite) and the high temperature (austenite) phase. Unlike conventional metals that recover less than 1% stress before plastic deformation, these shape memory alloys undergo a non-diffuse and thermally flexible ceramic phase transformation that enables the material instead of using the conventional known slip mechanism, as the twinning process allows for a complete recovery of strains up to 8% \[2,3\]. Where there are two separate mechanical effects that characterize the response of this shape memory alloy; The effect of stress memory and pseudo-elasticity. Without heating, martensite reverts to the austenite process when unloaded in
pseudoelasticity or superelasticity. On the other hand for these alloys, the effect of stress memory requires first heating in order to convert the martensite phase to the austenite phase in order for the deformation to take place permanently. The effect of the stress memory can also be thermally induced, and in this known case, where the structure is formed from the thermally moved martensite [4]. NiTi shape memory alloys are the most important group of shape memory alloys. The chemical composition of NiTi shape memory alloys are ranges from 53wt% to 57wt% nickel balance titanium[4,5]. Many marketable applications of shape memory alloys appeared. As these alloys exhibit strong shape memory effect and well known false elastic behavior, which are completely new properties compared to other conventional metal alloys. These new properties make this material suitable for a wide variety of applications. It also exhibits good corrosion resistance and is biocompatible, making it suitable for uses in various fields of engineering and biomedical applications such as those orthodontic, cardiovascular, neurosurgical, and orthopedic applications. [6,7]. In recent years, the production of NiTi shape memory alloys (SMAs) as fine porous materials have received great attention due to their mechanical properties and unusual porous structures similar to those found in some natural biomaterials for hard tissue culture. [8]. It was found that the porous nature of the existing bone tissue could migrate inward, which increases the good bonding force. Moreover, it enables engineering the properties of the implant to match the properties of the bone [9].

Where the main concern of using the medical shape memory alloy application is to dissolve free toxic nickel ions which may cause carcinogenic effects. This can prevent harmful metal from being modified to the appropriate surface of the alloy [10].

The shape memory alloys resistance to corrosion is mainly affected by the chemistry of the surface state, it is a very active element and when the shape memory alloy surface with a weak oxidizing environment comes into contact with the shape memory alloy, where it becomes a very thin original negative layer dominated by titanium dioxide on the surface of the shape memory alloy. As it can provide the adding of zirconium at conversion temperatures up to 200 °C [11], for potential applications it is used in rocket engines, gas turbines, nuclear reactors, and automobile engines. In most of the previous studies, it was found when zirconium was added to the solid solution to change Ti, thus raising the transformation temperatures, corrosion resistance and increased hardness. Current studies on knowledge of the mechanical, corrosion and functional behavior of Ni-Ti-Zr alloys are still very, very limited. Addition of zirconium also gives rise to unknown secondary phases that directly affect the properties of shape memory alloys. And when adding the element molybdenum, its effect is to increase the sedimentation stiffness that occurs due to the presence of multiple stages of phases. The aim of the research is to study the effect of the elements mobidium and zirconium on the shape memory alloys. When adding small amounts of zirconium leads to a smoothing of the granular size, when adding Mobidium improves the hardening process.

1.1. Experimental procedures

1.1.1 Materials

In this research, the materials used to make and prepare NiTi shape memory alloys, as shown in Table 1, were used with the alloys.

| Alloys | Ni% | Ti% | Zr% | Mo% |
|--------|-----|-----|-----|-----|
| A      | 55  | 45  | 0   | 0   |
| B      | 54  | 45  | 1   | 0   |
| C      | 53  | 45  | 2   | 0   |
| D      | 52  | 45  | 3   | 0   |
| E      | 54  | 45  | 0   | 1   |
| F      | 53  | 45  | 0   | 2   |
| G      | 52  | 45  | 0   | 3   |
1.1.2. Samples Preparation:
The materials powder with amalgams (A,B,C,D,E,F and G) was weighted by utilized delicate equilibrium Aligned sesnetive equilibrium type (L220 S D) with (0.0001 % exactness Z) german made. The powders were blended in electric moving blender, Alumina balls with various distance across have been utilized to blend and refine metal powder for (6 hours). Mixed on dry.
1.1.3. Compacting
This interaction includes compaction of the powders for each examples by utilizing a kick the bucket made of high combination steel with pass on measurement of 14 mm. by utilizing water driven presser. The powder was squeezed under tension of 800 MPa. Why was the pressure 800 used, where different pressures were used so that we got the ideal pressure to get the good mechanical properties .At that point tests with measurement 14 mm and 4 mm stature were delivered appeared . Hydraulic presser, 4387-4NE0000,Carver,USA shown in Fig 2
1.1.4. Sintering Process
According to the research plan, sintering of the pressurized green samples was done by using a very high temperature vacuum tube furnace. Where an oven was used with Arkon. The pressure of the vacuum furnace was 10-4 tons. Upon completion of the sintering scheme, the sintered samples are left in the oven to cool to room temperature. As shown in Figure 1 and Figure 3.

![Fig 1 Sintering program of compacted samples](image)

1.1.5. Preparation of Samples for Testing
As grinding papers were used on all surfaces of the amalgam samples, including the edges, were moistened with the use of earth (120,220, 400,600,800,1000,1500 and 2000) coarseness silicon carbide papers. At that point 4.1past of 6 μm to get a brilliant mirror finish for the last advance. At that point these examples degreased with CH3)2CO. Subsequent to drying, these examples were put away in Zip - lock sacks. The components of tests were estimated and recorded. Carving of tests accomplished in arrangement with fixings as the accompanying .[12]

I-HF 10 ml ,ii-HNO3 20ml ,iii-H2O 70ml

The readied tests are inundated scratching answer for 15 seconds ,at that point washed with refined water and dried ,at last examples was prepared for microstructure perceptions
1.2. Tests

1.2.1 Hardness Test

The shape memory effect (SME) was found out after the hardness test at room temperature. The microhardness account for all samples used in the search before and after thermo-mechanical treatments. An average of three readings was taken for each test. Micro Vickers hardness testing machine form (a Digital Micro hardness tested HV1000) was used in this study. this apparatus is found in Materials Department Laboratories/ the Material Engineering College/ University of Babylon. The microhardness was used to evaluate the product, with soaking time 10 sec. Microhardness values were obtained by using the equation [13]:

\[ H_v = 1.854 \frac{P}{d^2} \]  \hspace{1cm} \text{ (2)}

\( H_v \) refer to Hardness Vickers (kg/mm²).

\( P \) Refer to an applied load (Kilogram).

1.2.2. Microstructure Examination

The microstructure for the samples was observed by using optical microscopy (OM) type (Olympus microscope).

1.2.3. X-Ray Diffractions(XRD)

X-Ray diffraction method used mainly to Characterize the crystallographic structure of material. In the current study, production of copper powder. The powder has characterized by the diffraction of x-ray (XRD), type (Shimad Zo, XRD6000, diffractometers Japand) X-rays generated using Copper (E4-K) radiation out 30kV, 40mA and wavelength (\( \lambda = 1.5406 \) Å) radiations for generated pattern of diffraction From a powder Sample at room temperature in a 2\( \Theta \) ranges of 30 TO 80s to check if the structures are crystalline or amorphous

Fig. 2 The electric hydraulic press used to compacted samples
1.3. Results and Discussion

1.3.1. Vickers Hardness

The microhardness measurements have been made for the samples produced by powder metallurgy by taking the average of 5 readings at each point. The results obtained are represented graphically in figure 4.

Hardness test was lead using Vickers hardness mechanism. It was observed that as the Mo content is increased, the hardness values, increased. Which increased significantly as Mo content was increased to 40at%. This is mainly observed due to precipitation hardening which occurs due to the presence of multiple phase in alloy G.[14]
1.3.2. Microstructure Observation

Figs 5 indicated the optical microscopy images of bare samples. Fig 5 shows the light optical microscopy after etching. The phases NiTi and Ni3Ti can be differentiated in this picture, as well as some surface features such as open pores and grain boundaries.[15]
1.3.3. X-Ray Diffraction

XRD can be used to characterize shape memory alloys as shown in the figure 6 resulted in a three-phase structure of Through examination , They discovered that for all alloys, the significant phases that appeared were And NiTi phases cubic, Ni3Ti hexagonal phase and NiTi monoclinic phase.

Figure 6 X-Ray Diffraction
1.4 Conclusions

- Where the results obtained from the research appeared, where we reached the following conclusions:
  - It was found that sintering the alloy at a temperature of 950 °C for a period of 6 hours is effective to fully satisfy the sintering and converting Ni Ti into alloy structure.
  - The sample (cooling in furnace) compressed at 800 MPa and sintering at 950°C for 6 hours caused in a three phase structure (hexagonal Ni3Ti phase, NiTi monoclinic phase and NiTi cubic phase) at room temperature.
  - The microstructure was obtained explain accuracy the phases that found in X-ray analysis.

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