NON DESTRUCTIVE TESTING FOR Ni-Ti SAMPLES PREPARED BY DIFFERENT TECHNIQES

Manar N. AlNaber
Department of Civil Engineering
The Hashemite University
Zarqa, Jordan 13135

Wisam M. Abu Jadayil*
Department of Mechanical and Industrial Engineering
American University of Ras AlKhaima
Ras AlKhaime,UAE

Abstract — Long series of searches were applied to emphasize Ni-Ti alloy composition harmony within human body. Ni-Ti alloy chemical and mechanical testing results paved for a revolution in the field of medical implants. Despite the fact that Ni-Ti alloy has simple composition, fabrication of implants remains a great deal of concern. Medical implants should be tested to assure that there are no defects or imperfections included, and so no harmful effects could take place. In this study different Ni-Ti alloy composition samples have been fabricated using three different techniques. These techniques are sand casting, powder – sand casting method and sintering method. Non destructive techniques were used to test samples structure and their surface and subsurface defects. Die penetrant test were applied to all samples then inspected with electromagnetic method and finally samples were inspected using ultrasonic waves. It was found that samples prepared using sand casting showed the best results and so sand casting is highly recommended to fabricate Ni-Ti ally implants.

Keywords: Ni-Ti composition,implants,sintering,Nondestructive techniques

I. INTRODUCTION

Near equiatomic alloy had attracted Scientifics to use as functional materials long time ago [1] [2] .The first Nitinol alloy was prepared by Buhler in the Naval Ordinance Laboratory in 1962 [3-4]. The first fatigue study of NiTi SMAs was performed by Melton and Mercier [5] in 1978; pseudoelastic fatigue tests were run on wire specimens with different temperatures. This work was soon followed by that of McNichols and Brookes [6], who studied the fatigue life of NiTi springs. It wasn't until the early 90s, when the medical industry began to push for less invasive medical procedures and alternative implants [7]. Takeshita et al. [8] implanted a cylindrical NiTi parts in rats for 168 days in the year 1997.

An electro polished Ni-Tinol samples were implanted in a periosteum osteoblasts, for the first Nickel percentage of NiTinol composition where the issue that studied by kapanen et. al. [10] in the 2002 a fabricated samples with different percentages of nickel and titanium had been investigated for in vitro studies by Bogdanski and co-workers. The highest biological compatibility assured to be within a maximum 50 % nickel element of the alloy weight [11], higher percentages of Ni have revealed nickel releases and the released nickel rapidly reached cytotoxic concentrations within one day [12]. Also many similar researches later handled toxicity and corrosion resistance [13-18].

Equiatomic Nitinol, with its pseudoelastic effect, was found to have several ideal properties for such aim [8-9]. Fortunately, Mcklevey & O.Ritchie [19] found that (Ni50Ti50) casted alloy samples showed a full Austenitic structure at body temperature, which means a perfect mechanical condition providing super elasticity. Begdanoski et. al. [11] assured that the same alloy composition was the best biologically fit as an implant within the human body.

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Clues provided pushed later for experiencing fatigue life for alloy samples of different compositions, where Nickel wt. of the alloy composition was around 50%. Krone et. al. [20] studied mechanical behavior of Ni-Ti parts fabricated using sintering method. Sadrnezhaad S. K., Arami H. et. al. [21] also studied Ni-Ti alloy samples fabricated by powder metallurgical method.

II. MATERIALS

A. *Raw Metals*

Nickel, Titanium, Copper and Tin metals were used each with the size and purity of powders as shown below:

TABLE I. USED METALS SPECIFICATIONS

Metal	Purity	Melting Temp (c)	Shape
Ni	99.8%	1453	Plates & bullets
Ti	99.6%	1670	Powder (150 micron)
Tn	98.2%	232	Irregular pieces

B. Sand Casted and Powder-Sand Casted Samples

Two composition of Ni-Ti alloy samples are fabricated for fatigue and tensile testing. Table II illustrates the two compositions and No. of samples fabricated by casting method where table III illustrates compositions and No. of samples fabricated by powder – casting method:

TABLE II. SAMPLES FABRICATED BY SAND CASTING METHOD

Testing Purpose	Ni % - Ti %	No. of Samples
Fatigue test	50 - 50	12
Fatigue test	52.8 - 47.2	12
Tensile test	50 - 50	3
Tensile test	52.8 - 47.2	3

TABLE III. SAMPLES FABRICATED BY POWDER- SAND CASTING METHOD

Testing Purpose	Ni % - Ti %	No. of Samples
Tensile test	50 - 50	3
Tensile test	52.8 - 47.2	3

Figure 2.1 illustrates standard shape and dimensions for fatigue test samples, where figure 2.2 illustrate standard shape and dimensions for tensile test samples:

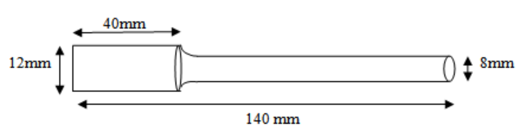

Fig.2.1 Fatigue test sample dimensions

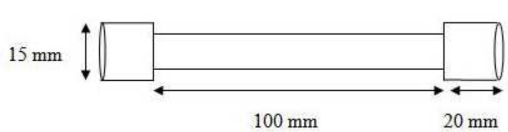

Fig.2.2 Tension test sample dimensions

C. Powder Metallurgical Samples

Sample of each composition is fabricated by sintering method. Since there is no Nickel powder in local market, Nickel bullets were smashed into powder to use in the fabrication of metallurgical samples. Figure 2.3 illustrates powder metallurgical sample shape and dimensions.

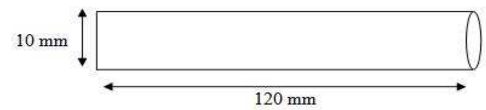

Fig.2.3 Powder metallurgical sample dimensions

III. METHODOLOGY

A. Sand Casted Samples

Ti powder and Nickel plates were weighed in the appropriate proportions as shown in Table 2.2. These metals were put in a spout. Spout then placed in electromagnetic furnace (1700 c) for nearly an hour till it melts. Metals are irrigated regularly to assure homogenous mixture. Mixture is poured in already prepared cylindrical shaped sand moulds. Molten metal left to be cooled by room air. Cylindrical samples are taken from moulds to lathing in order to shape as shown in figures 2.1 and 2.2

B. Powder-Sand Casted Samples

The same procedure previously is applied. The only difference here is Ni pullets are molted at first sight. Ti powder is poured in to liquefy Ni and irrigated regularly in order to get homogeneous mixture. This procedure was suggested such that samples are fabricated without requiring elevated furnace temperature.

C. Powder Metallurgical Samples

A stainless steel die was specially designed as in figure 2.4 for the consolidation of the metal powders into a mould. The powder was proportionally weighed and well mixed as in table 2.3. Powder mixtures poured in to steel mould and pressed at (5 tons) force with a manual pellet press and held for about 30 min as shown in figure 2.4. The pressed specimen is then carefully ejected out of the die and placed in oven (1235 c) for an hour

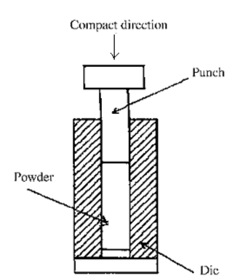

Fig.2.4 Powder metallurgical sample preparation

IV. Nondestructive Testing

As a preliminary indication about samples statues, their surface has been tested nondestructively using penetrant die. Since Ni-Tinol samples are ferromagnetic alloy, magnetic particles method was used for surface inspection too. Surface configuration is important; some surface cracks or any showed defects may lead into easier failure or maybe underestimated life valued strength under fatigue or tension testing if it's really propagated or existed in any critical region.

A. Penetrant Test

Samples are cleaned by a special cleaner solution, sprayed with a red coloring die as shown in figure 2.5. Small die particles give the advantage for osmotic property to improve small hidden cracks visibility. Die left enough time or what is called a delay time "nearly 10 minutes" to assure die prevalence. Samples are cleaned by cleaner and a piece of fabric.

Figure 2.5 Applying die penetrant to fatigue samples

Figure 2.6 Fatigue samples of different compositions covered with a developer spray

B. Magnetic Test

Magnetic particles test has the same concept of die penetrant test except that magnetic field is the actor not osmosis. Samples are cleaned thoroughly and dried. Specimens are charged with a current of electrons to form an electromagnetic field area by means of prods. When attraction felt between sample and prods sample is magnetized, it's removed and covered with fine magnetic particles as shown in figure 2.7.

An excellent style manual for science writers is the work done by Chengli in 2010 [7].

Figure 2.7 Fatigue sample magnetized by means of prods

C. Ultrasonic Test

Internal structure was examined in case for the existence of any internal defects. It could be as a result to some errors in casting method, air inclusions .., etc. Ultrasonic method applied to samples before testing. Grease used as a couplant material between probe and sample surface. Figure 2.8 illustrates inspection procedure.

Figure 2.8 Fatigue sample inspection using ultrasonic method

V. Results and Discussion

A. Penetrant Test

As shown in figure 2.9, there were no surface cracks cleared for any of fatigue samples. Crack was noticed on the surface somewhere near the end at one of the 52.8 % of wt Ni-Ti tensile samples, where other samples were free of any surface defects. Figure 2.10 shown obviously the crack found. Figure 2.11 shows that no surface defects found for powder metallurgical samples.

Figure 2.9 50% of wt. Ni-Ti fatigue sample covered with developer showed no surface defects

Figure 2.10 Crack showed on 52.8% of wt. Ni-Ti tension sample surface after applying developer

Figure 2.11 Powder tension samples covered with developer material

B. *Magnetic Test*

Exactly the same results shown by penetrant test have claimed by magnetic test. It's obviously shown in figure 2.12 and figure 2.13 that no clusters appeared. No surface cracks appeared for the casted samples but only for one of the 52.8 % of wt. Ni-Ti tension samples. Magnetic test couldn't be applied to fatigue sample containing copper since it's a non- ferrous alloy.

Figure 2.12 50% of wt. Ni-Ti fatigue sample applied to magnetic inspection

Figure 2.13 52.8% of wt. Ni-Ti tension sample applied to magnetic inspection

C. *Ultrasonic Test*

Ultrasonic test had failed to inspect fatigue samples. Only edge reflections had showed on the digital screen. Figure 2.14 illustrates initial and backward reflections. Tension samples were efficiently inspected. While inspecting 52.8% of wt. Ni-Ti sample, an intermediate reflection showed on the screen between front and back reflections. This reflection indicates the existence of cracks inside. This method also did not work for powder metallurgical samples. The reason why might be cause of inhomogeneous structure while preparing mixture.

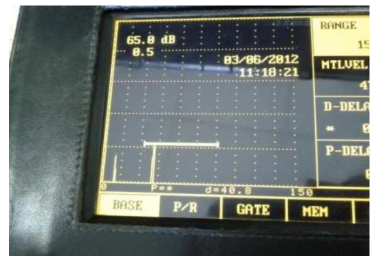

Figure 2.14 Edge reflections for fatigue sample inspected by ultrasonic waves showed on the digital screen

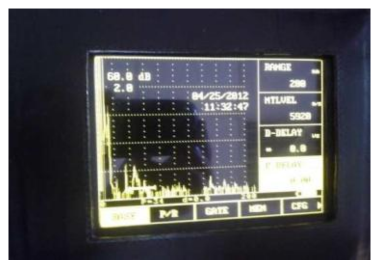

Figure 2.15 An intermediate sign reflection shows on digital screen while inspecting 52.8% of wt. Ni-Ti sample

VI. CONCLUSIONS

In this research samples were fabricated using sand casting, casting-powder and powder metallurgical method. Sand casting-powder samples fabricated by means of melting Nickel and solving Ti powder. Method failed due to inappropriate structure homogeneity and brittleness. Samples were broken during lathing process. ND techniques were used to check samples for surface and subsurface defects. The main conclusions could be outlined as follows:

- Casting-powder method is not recommended for fabrication of Nitinol alloys. Samples resulted had highly level of non homogeneity and brittle structure hence, testing difficulties.

- Casting method is highly recommended to fabricate Ni-Ti ally implants.
- It's recommended to use other techniques instead of ultrasonic waves such as XRD to check samples for any subsurface defects. Some standard sample shapes and dimensions are difficult to be inspected using ultrasonic method.
- Penetrant test and magnetic test could be applied efficiently to check for any surface defects.
- Since medical Ni-Ti alloy implants are case sensitive, Its highly recommended that surface defects are inspected using florescence dye penetrant instead of colored die. This type of dies assists in revealing smaller cracks and surface defects.

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