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Research Article

Surface and Subsurface Defects Investigation of Ni-Ti Samples Processed by Different Fabrication Methods

Wisam M. Abu Jadayil
Department of Mechanical and Industrial Engineering, American University of Ras Al Khaimah, P.O. Box 10021, UAE

Abstract: The use of Ni-Ti alloy in medical applications gave a great importance for the right composition of this alloy and the method of fabricating this alloy parts. 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. A comparison between samples made in by each technique was made. Surface and subsurface defects found in these fabricated samples have been investigated using non-destructive techniques. These non-destructive techniques used were die penetrant testing, electromagnetic method and finally samples were inspected using ultrasonic waves. Based on the results of surface and subsurface non-destructive tests used, samples prepared using sand casting were found to have the lowest surface and subsurface defects and so it is highly recommended to fabricate Ni-Ti alloy implants.

Keywords: Implants, nondestructive techniques, Ni-Ti composition, sintering

INTRODUCTION

Near equiatomic alloy had attracted Scientifics to use as functional materials long time ago like Ishida and Martynov (2002) and Machado and Savi (2003). The first Nitinol alloy was prepared by Buehler *et al*. (1963) in the Naval Ordinance Laboratory in 1963 and Buehler and Wang (1967). The first fatigue study of NiTi SMAs was performed by Melton and Mercier (1978); pseudoelastic fatigue tests were run on wire specimens with different temperatures. This study was soon followed by that of McNichols *et al*. (1981), 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 as reported by Song (2010). Takeshita *et al*. (1997) implanted a cylindrical NiTi parts in rats for 168 days. An electropolished NiTinol samples were implanted in a periosteum osteoblasts, for the first 26th week it showed no toxicity effects but a small deceleration in proliferation process by Shabalovskaya (2001). Nickel percentage of NiTinol composition where the issue that studied by Kapanen *et al*. (2002) a fabricated samples with different percentages of nickel and titanium had been investigated for in vitro studies by Bogdanski *et al*. (2002). The highest biological compatibility assured to be within a maximum 50% nickel element of the alloy weight, higher percentages of Ni have revealed nickel releases and the released nickel rapidly reached cytotoxic concentrations within one day.

Also many similar researches, like Wu *et al*. (2007), Liu *et al*. (2007), Rhalmi *et al*. (2007), Shishkovsky *et al*. (2007), Rocher *et al*. (2004), Kujala *et al*. (2002) and Sun *et al*. (2002) handled later toxicity and corrosion resistance. It was found that the "memorial" effect point of transitions from structure to another fits perfectly body temperature. Equiatomic Nitinol, with its pseudoelastic effect, was found to have several ideal properties for such objective. Fortunately, McKelvey and Ritchie (2001) found that (Ni50Ti50) casted alloy samples showed a full Austenitic structure at body temperature, which means a perfect mechanical condition providing super elasticity. Bogdanski *et al*. (2002) assured that the same alloy composition was the best biologically fit as an implant within the human body. 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*. (2004) studied mechanical behavior of Ni-Ti parts fabricated using sintering method. Sadrnezhaad *et al*. (2006) studied Ni-Ti alloy samples fabricated by powder metallurgical method.

Because of the importance of the Ni-Ti composition in many engineering applications, many researches like Abu Jadayil and Flugrad (2007), Abu Jadayil (2008), Abu Jadayil and Jaber (2009), Abu Jadayil and Khraisat (2010) and Abu Jadayil and

Mohsen (2011) have been conducted to investigate the fatigue life of components made of Ni-Ti alloy. On the other hand, Abu Jadayil and Alnaber (2013) investigated the fatigue life of Ni-Ti samples prepared by different techniques.

Strengthening and enhancing the properties of the composite material by alloy it with another element like iron or copper was done by Khraisat and Abu Jadayil (2010) and Khraisat *et al*. (2009). Abu Jadayil (2010) investigated the most recent techniques of the solid object modeling. Although sand casting after converting the composite metals into molten is the most common technique, many factors should be taken into considerations when using this method; like the pouring rate as discussed in Abu Jadayil (2011a) and the solidification time as discussed by Abu Jadayil (2011b). To continue research conducted in this direction, this research is comparing between three techniques for producing Ni-Ti samples and testing samples produced by these three techniques using three nondestructive tests.

MATERIALS AND METHODS

Preparation of the Ni-Ti composite material:
Raw metals used: Nickel and Titanium metals were used each with the size and purity of powders as shown in Table 1.

Table 1: Specifications of metals used

Metal	Purity	Melting temp (C)	Shape
Ni	99.5%	1453	Plates and bullets
Ti	99.2%	1670	Powder (150 micron)

Ni-Ti samples prepared by sand casting and powder- sand casting techniques: Samples of Ni-Ti were prepared using two techniques, sand casting and powder-sand casting. These samples were made to be used in tensile and fatigue testing. But before these samples are used for destructive testing of fatigue and tensile testing, they were first tested nondestructively by surface and subsurface nondestructive tests. Two composition of Ni-Ti alloy samples are fabricated for fatigue and tensile testing. Table 2 illustrates the two compositions and No. of samples fabricated by sand casting method where Table 3 illustrates compositions and No. of samples fabricated by powder-casting method.

Table 2: Samples fabricated by sand casting method

Destructive testing purpose	Ni %	Ti %	No. of samples
Fatigue test	50.0	50.0	12
Fatigue test	52.8	47.2	12
Tensile test	50.0	50.0	12
Tensile test	52.8	47.2	12

Figure 1 illustrates standard shape and dimensions for the prepared fatigue test samples, where Fig. 2 illustrate standard shape and dimensions for tensile test samples:

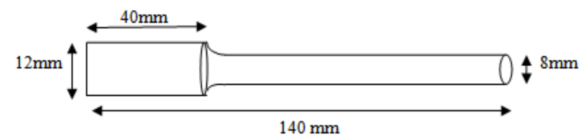

Fig. 1: Fatigue test sample dimensions

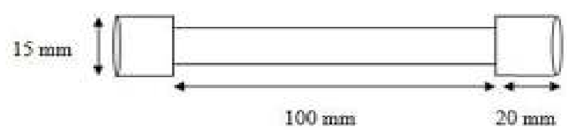

Fig. 2: Tensile test sample dimensions

Ni-Ti samples prepared by powder metallurgy method: Six sample of each Ni-Ti 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 3 illustrates powder metallurgical sample shape and dimensions.

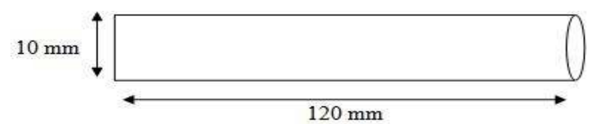

Fig. 3: Powder metallurgical sample dimensions

Techniques used for preparation of Ni-Ti samples:
Sand casting method: Ti powder and Nickel plates were weighed in the appropriate proportions as shown in Table 2. These metals were put in a spout. Spout then placed in electromagnetic furnace (1700°C) for 75 min to make sure it is completely melted and a molten is formed. The two metals are irrigated regularly to assure homogenous mixture. Mixture is poured in already prepared cylindrical shaped sand moulds. Molten mixture of the two metals left to be cooled by room air. Cylindrical samples are taken out of sand to grinding and polishing processes in order to produce the required sample shapes for fatigue and tensile testing, as shown in Fig. 1 and 2.

Powder-sand casting method: The same procedure previously is applied. The only difference here is Ni pullets are melted at first sight for 75 min at a temperature of 1500°C, then Ti powder is poured into molten Ni and irrigated regularly for 15 min in order to get homogeneous mixture. This procedure was suggested such that samples are fabricated without requiring elevated furnace temperature. Around 1500°C was enough for fabrication of these Ni-Ti samples, whereas 1700°C was needed in case both metals are required to be melted and casted.

Powder metallurgical samples: A stainless steel die was specially designed as shown in Fig. 4 for the consolidation of the metal powders into a mould. The powder was proportionally weighed and well mixed according to proportions in Table 3.

Table 3: Samples fabricated by powder-sand casting method

Testing purpose	Ni %	Ti %	No. of samples
Fatigue test	50.0	50.0	6
Fatigue test	52.8	47.2	6
Tensile test	50.0	50.0	6
Tensile test	52.8	47.2	6

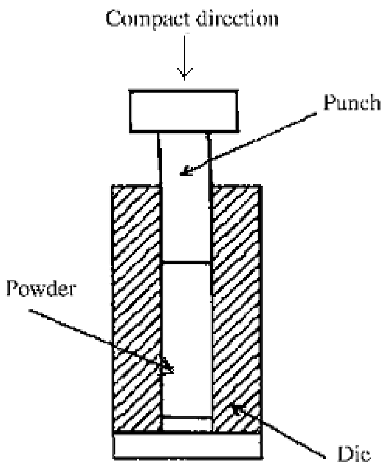

Fig. 4: Powder metallurgical sample preparation

Fig. 5: Applying die penetrant to fatigue samples

Fig. 6: Fatigue samples of different compositions covered with a developer spray

Fig. 7: Fatigue sample magnetized by means of electrical prods

Fig. 8: Fatigue sample inspection using ultrasonic method

Powder mixtures poured into steel mould and pressed at (5500 kg) force with a manual pellet press and held for about 30 min as shown in Fig. 4. The pressed specimen is then carefully ejected out of the die and placed in oven (1235°C) for 75 min.

Non destructive testing used for defects investigation: Surface and subsurface defects, like cracks and inclusions, can be tested and investigated using nondestructive techniques. Since these samples are going to be tested destructively later to investigate their fatigue lives and their tensile strengths, we need here to test them without changing their microstructure or creating any more defects in these samples. As a preliminary indication about samples conditions, their surface has been tested non-destructively using penetrant die. Moreover, since NiTinol 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 loading or tensile testing if it's really propagated or existed in any critical region. On the other hand, internal defects and inclusions were tested using ultrasonic testing.

Penetrant test: Samples are cleaned by a special cleaner solution, sprayed with a red coloring die as shown in Fig. 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 15 min" to assure die prevalence. Samples are cleaned by cleaner and a piece of fabric. A developer liquid sprayed all over the samples as in Fig. 6.

Magnetic particles 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 Fig. 7.

Ultrasonic test: Internal structure was examined in case for the existence of any internal defects or inclusions. It could be as a result to some errors in casting method, air inclusions, or inhomogeneity in the mixture. Ultrasonic method applied to samples before other nondestructive testing. Grease used as a coolant material between probe and sample surface. Figure 8 illustrates inspection procedure.

RESULTS AND DISCUSSION

Penetrant test: Testing all samples prepared by the penetrant test, with the two methods of sand casting and

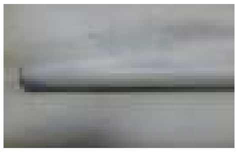

Fig. 9: 50% of wt. Ni-Ti fatigue sample covered with developer shows no surface defects

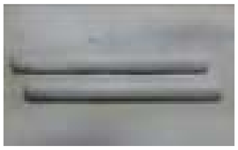

Fig. 10: Powder tensile samples covered with developer material

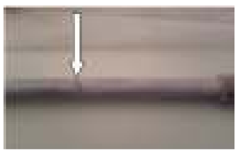

Fig. 11: Crack showed on 52.8% of wt. Ni-Ti tensile sample surface after applying developer

Fig. 12: 50% of wt. Ni-Ti fatigue sample prepared by sand casting and subjected to magnetic inspection

Fig. 13: 52.8% of wt. Ni-Ti tension sample prepared by sand casting and subjected to magnetic inspection

Fig. 14: Edge reflections for samples prepared by powder metallurgy method and inspected by ultrasonic waves showed on the digital screen

the powder metallurgy showed no clear surface defects were found. Examples of that is shown in Fig. 9 and 10. In Fig. 9, there were no surface cracks clearly shown for 50% of wt. Ni-Ti fatigue samples. Same happened for all other samples made by sand casting, all samples were free of any surface defects. Figure 10 shows that no surface defects found for powder metallurgical samples as well. May be the reason for that is the 5.5 tons used in making these metallurgical samples helped in giving good surface finish for the sample, without clear defects. Moreover, heating the metallurgical samples after pressing might reduce surface defects. On the other hand, when the powder-sand casting samples were tested, some defects were seen on the surface of 52.8% Ni-47.2% Ti samples. Clear surface defects were seen when the percentage of Ti powder was increased in the 50% samples. That might be related to incomplete melting of Ti powder, which caused some surface roughness and that increases as the percentage of Ti increases. Example of that is shown in Fig. 11, where crack was noticed on the surface somewhere near the end at one of the 52.8% Ni-47.2% Ti tensile samples. Surface defects in tensile samples were clearer than fatigue sample and powder metallurgy samples, which might be related to a little more complicated geometrical shape.

Magnetic test: Exactly the same results shown by penetrant test have claimed by magnetic test. It's obviously shown in Fig. 12 and 13 that no surface cracks or clusters appeared for tensile and fatigue samples prepared by sand casting and powder metallurgy methods. Just samples prepared by powder-sand casting methods showed some surface cracks and defects.

Ultrasonic test: Ultrasonic test had failed to inspect powder metallurgical samples. Only edge reflections had showed on the digital screen. Figure 14 illustrates initial and backward reflections. The reason for that might be the inhomogeneous structure while preparing mixture and so the existing of many vacancies and defects in the internal structure of the samples.

Samples prepared by Powder-sand casting were efficiently inspected. While inspecting these samples, an intermediate reflection showed on the screen between front and back reflections. This reflection indicates the existence of cracks inside. An example for inspecting these samples is Fig. 15, where indications of existence of internal cracks inside 52.8% of wt. Ni-Ti sample are shown on the screen.

Sand casting samples were inspected successfully using ultrasonic and indications of existence of minor vacancies and defects were shown on the screen.

Summary of the results of non-destructive testing: The results of using the three non-destructive testing of Penetrant test, Magnetic test and the ultrasonic test can be summarized in Table 4.

Table 4: Comparison between non-destructive techniques used for testing different samples

	Sand casting	Powder-casting	Powder metallurgy
Penetrant test	No external defects	Some external defects	No external defects
Magnetic test	No external defects	Some external defects	No external defects
Ultrasonic test	Few internal defect found	Some internal defects found	Could not be tested: Indication of many internal defects

Table 5: Comparison between different fabrication methods of different Ni-Ti samples

	Advantages	Disadvantages
Sand casting method	• Less surface defects • Few subsurface defects	• Needs machining • High temperature for melting Ti and Ni • Few subsurface defects
Powder-Sand casting method	• Lower temperatures are needed just to melt Ni pullets. • Less subsurface defects compared to powder metallurgy method	• Non-homogenous mixture might result • Some surface defects • Brittle samples to be machined • Subsurface defects and voids are found.
Powder metallurgy method	• Less surface defects • Low temperatures are needed	• Internal structure has many defects and vacancies and so might be very weak • Fine particles of Ni and Ti are needed

Fig. 15: An intermediate sign reflection shows on digital screen while inspecting 52.8% of wt. Ni-Ti sample made by powder-sand casting method

Summary of the results of sample fabrication methods: As explained earlier the non-destructive testing were used in this research to compare between three different methods for fabrication the Ni-Ti alloy, the sand casting method, the powder-sand casting method and the powder metallurgy method. Based on the results obtained by non-destructive testing of samples prepared the three methods and in different shapes, the comparison between these three methods of fabrication can be summarized in Table 5.

CONCLUSION

In this research samples were fabricated using sand casting, casting-powder and powder metallurgical method. Non-destructive techniques were used to check samples for surface and subsurface defects. The main conclusions could be outlined as follows:

- Although it requires lower melting temperatures than sand casting method, powder-sand casting method is not recommended for fabrication of Nitinol alloys. Samples resulted had highly level of surface and subsurface defects, non-homogeneity and hence brittle structure.
- Powder metallurgy method needs lower temperature levels to fabricate Nitinol alloys and produces free defects surfaces, but it is not recommended for fabrication of Nitinol alloys because of weak internal structure with very high level of defects and voids, which results in brittle structure.
- Sand casting method requires high melting temperatures and produces samples with few surface and subsurface defects, even though it is highly recommended to fabricate Ni-Ti ally implants.
- Penetrant test and magnetic test could be applied efficiently to check for any surface defects. Penetrant is easier but it is qualitative rather quantitative test.
- Since medical Ni-Ti alloy implants are case sensitive, it is 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.
- Some standard sample shapes and dimensions are difficult to be inspected using ultrasonic method. Moreover, when the internal structure has high level of defects and voids, ultrasonic test is unable to predict the amount of defects. So, it's recommended to use other techniques instead of ultrasonic waves such as XRD to check samples for any subsurface defects.

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