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Fabrication of Spiral Stent with Superelastic/ Shape Memory Nitinol Alloy for Femoral Vessel

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A B S T R A C T

Stent is a metal mesh tube for opening the obstructed vessels of the body. Ni-Ti alloy is a suitable metal for fabrication of stent due to its potential for applying the appropriate stress and strain to the vessel walls. In this study, super-elastic Nitinol wire was used to build stent samples usable to open femoral vessel. Ageing was performed at 500°C for different periods of time to determine the most appropriate transformation temperatures and shape memory/superelasticity behavior of the sample. Mechanical and structural properties of the alloy were determined by differential scanning calorimetry (DSC), electron probe micro analysis (EPMA) and metallographic studies. Ability to stand vessel wall pressure was studied by crush test. Images of scanning electron microscope (SEM) showed that the surface integrity was not affected by strain. The artificial silicon vessel in Simulated Body Fluid (SBF) at 37°C was used for implanting of the crimped stent. The recovery of strain and exertion of stress to the vessel wall was investigated after removal of the stent from the catheter.

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1. INTRODUCTION

Stent is a metal mesh tube that is used to open an obstructed vessel in the human body [1]. A balloon-expandable coil-type device usually made from 316 L stainless-steel wire coil attached to a single longitudinal support is inserted into the obstructed vessel to reinstate the flow of blood in the vein. Stainless-steel stents, however, have some drawbacks like low fatigue performance and difficulty in stress/stain control which may cause damage to the skin and blood vessel at the site of insertion of the catheter. Other serious problems like infection, restenosis and complications in kidneys function due to the dye usage during the procedure can also be of high risk. NiTi superelasticity and low fatigue crack threshold gives rise to superb performance at high amplitude and high mean strains [2-4]. Design and fabrication of NiTi tube mesh has, therefore, been considered as a desirable alternative to the 316L usage [5-8]. Nitinol can exhibit elastic deformation behavior close to the structural materials of the living body. Natural organs like hair, tendon or bone can deform sometimes with up to 10% strain in a non-linear way. When the alloy is inserted into the vessel, the recovered strain can open the blocked vain for reinstatement of the flow in the vessel [9].

Self-expanding Nitinol stents are manufactured with diameters larger than obstructed target vessel. Their austenite finish temperature (A_f) is usually set to 30^{o}C [8]. These stents can be crimped in the delivery system at room temperature. For treatment, it releases from the delivery system and expands into a scaffold for the vessel wall [9]. Shape of the raw material usable for stent-manufacture can be wire, sheet or tube. Fabrication methods are different depending on this shape.

In this paper, results of the experimental works done to fabricate stent samples suitable for implantation into the femoral vessel are reported. Design parameters have previously been studied by mathematical simulation investigations [10, 11]. Both shape memory and superelasticity properties of the material are considered in design of the sample. Mechanical and physical

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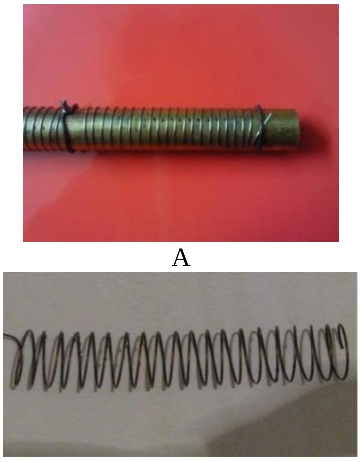
properties of the samples are improved by heat treatment of the wires [12]. These properties are determined by crushing tests to survey the appropriate behavior of the produced stents.

2. EXPERIMENTAL PROCEDURE

A piece of orthodontic Ni-Ti wire with 0.3 mm diameter was used to fabricate a stent with mixed shape memory/superelasticity behavior. Dimension of the stent was determined by size of the target vessel. Henceforth, a steel mandrel with 1 cm diameter was used to form a helical shape in the stent (Figure 1A).

Our findings showed that the appropriate ageing temperature for stent is 500^{o}C which is consistent with results of the previous researches [13-16]. For obtaining desirable time, mold was placed inside a heated furnace for 1, 1.5 and 3 h and after a certain time, DSC analysis (Maia200F_3 machine) showed the effect of the ageing time on formation of new phases. The best heating time for fixation of the shape was 15-20 min. Metallographic analysis was conducted to consider the microstructure of the alloy before and after the heat treatment. The wires were hot mounted by compound resin and then etched by HF-HNO_3-CH_3COOH solution (with respective ratio of 1:5:5) based on the literature [17-19].

Analysis by Electron probe micro-analyzer (EPMA) having Cameca SX100 showed changes of the composition during the heat treatment[2]. Manufactured stent should tolerate pressures exerted by the vessel. Thus mechanical properties of the stent were measured by crushing tests done by Instron 5566. Since the maximum force of the vessel is 1.14 N, maximum and minimum forces applied in the crushing test was 1.6 and 0.8 N, respectively. This was consistent with the values given in the literature [20, 21].



A

B

Figure 1. Stent screwed around steel mandrel (A) and stent after heat treatment and shaping (B)

[2] *www-2.unipv.it/compmech/cofin_02/stent_crush.htm*

After crushing, surface morphology and effect of reapplying forces was taken into account using SEM microscopy (LEO, 1450, VPP). Finally, in the dish with SBF solution and artificial silicon vessel, stent was implanted inside an artificial vessel and behavior of the stent was studied.

3. RESULTS AND DISCUSSION

Application times and temperature were all suitable because in all 3 times stents were formed to mold shape (Figure 1B). However, increasing the residence time of stent in oven caused phase transformation temperatures to rise which are shown in DSC diagrams of Figures 2 and 3. Figure 2 depicts the DSC diagram of the sample before the heat treatment in which austenite finish temperature (A_f) is 38.7^{o}C. Figure 3 depicts the DSC diagram of the sample after heat treatment that A_f is 51.5^{o}C. Due to the fact that A_f of stent should be below 37^{o}C, this enhancement of phase transformation temperatures is not desirable.

Figures 4A and 4B illustrate microstructures of the alloy before and after heat treatment, taken by optical microscopy at 500 magnification. According to these figures, before heat treatment, austenite parent phase is present in the sample; while after heat treatment, martensite phase develops in the austenitic parent phase. Table 1 presents alloy compositions before and after heat treatment. It is seen that the percentages of titanium slightly reduces during heat treatment. This is due to higher activity of titanium than nickel which results in Ti higher vapor pressure and oxidation tendency. The weight percentages of nickel and titanium in the samples show that besides Ni and Ti, there is a few percentages of other elements (like Co) which are usually added to the alloy to obtain the required properties for orthodontic application.

In the crushing tests, we found that the stent could resist against maximum force without collapsing. When stress was removed, the stent returned to its initial shape and size which indicates it superelastic behavior (Figures 5 and 6). According to the SEM images, there were no cracks, fracture or structural changes onto the stent surfaces (Figures 7A and 7B).

Figure 8A shows the effect of heating by 32°C in SBF solution on the stent patency after stent implantation in the artificial vessel. As is observable, the stent just expanded to 5.2 mm. This expansion was less than the amount the stent had been crimped. The value was related to the superelastic property of the produced sample. However, at 37°C, the stent expanded to 7.9 mm indicating some shape memory effect in addition to the previously observed superelastic behavior. This result indicated that the produced stent could be considered a suitable scaffold for vessel wall (Figure 8B).

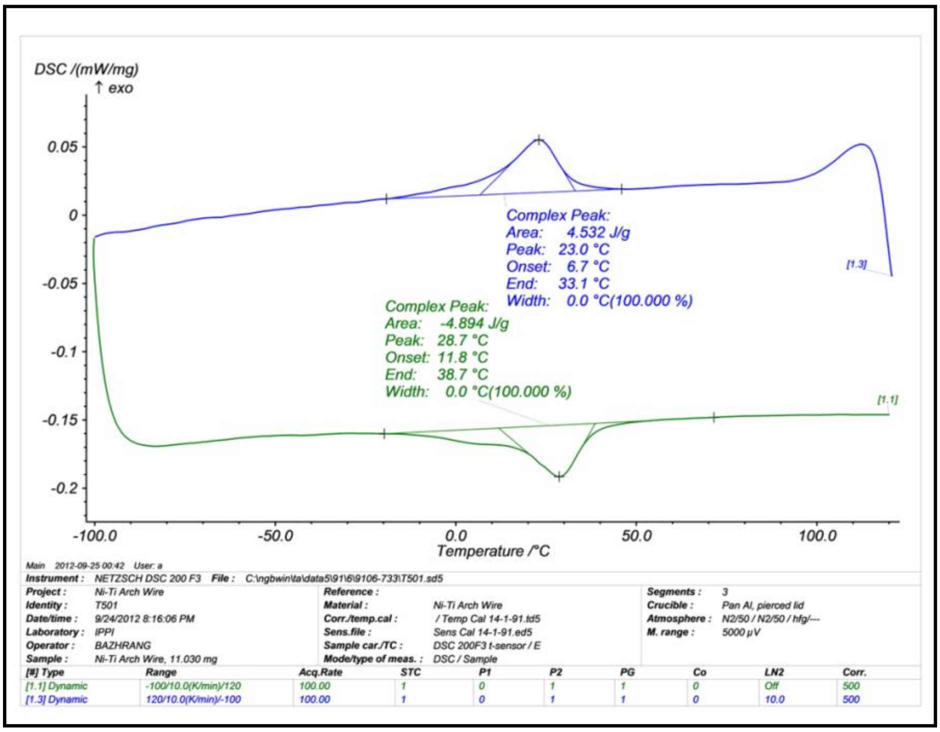


Figure 2. DSC diagram related to sample before heat treatment

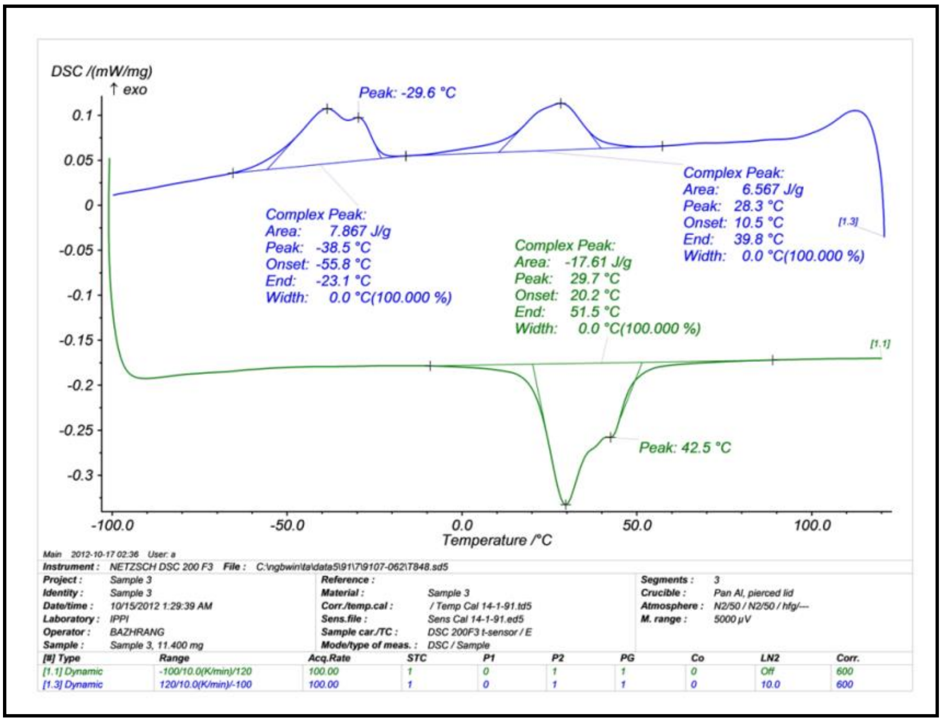
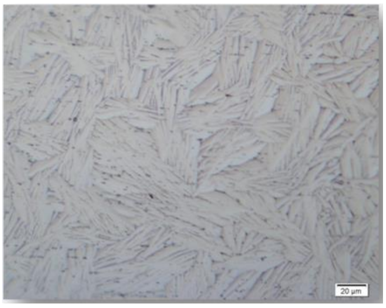
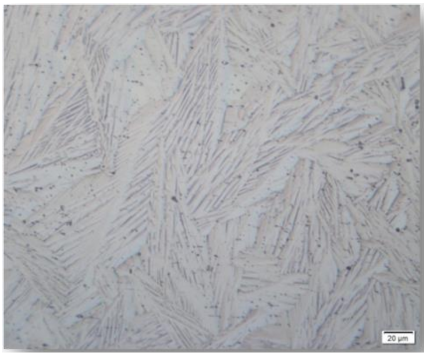


Figure 3. DSC diagram related to sample after 3 hours heat treatment

At 42°C the produced stent not only was a scaffold, but it exerted a force against the wall which caused 8.8 mm expansion in the vessel (Figure 8C).



A



B

Figure 4. Optical microscope image of the sample at 500x: (A) before heat treatment and (B) after heat treatment

TABLE 1. EPMA weight percentages of nickel and titanium in the samples before and after heat treatment

Data Set/Point	Before			After		
	Ni	Ti	Total	Ni	Ti	Total
1 / 1.	52.73	42.64	95.37	53.57	41.75	95.32
1 / 2.	52.92	43.21	96.13	53.60	41.92	95.51
1 / 3.	52.56	42.49	95.05	53.35	41.85	95.2
1 / 4.	52.68	43.15	95.83	53.24	41.95	95.19
1 / 5.	52.02	43.1	95.11	53.73	41.91	95.64
1 / 6.	52.41	43.01	95.41	53.38	42.01	95.38
1 / 7.	52.11	43.15	95.26	53.70	41.91	95.6
1 / 8.	52.37	43.31	95.68	53.59	41.9	95.49
1 / 9.	52.14	43.62	95.77	53.55	41.88	95.43
1 / 10.	52.85	42.88	95.74	53.85	41.82	95.67

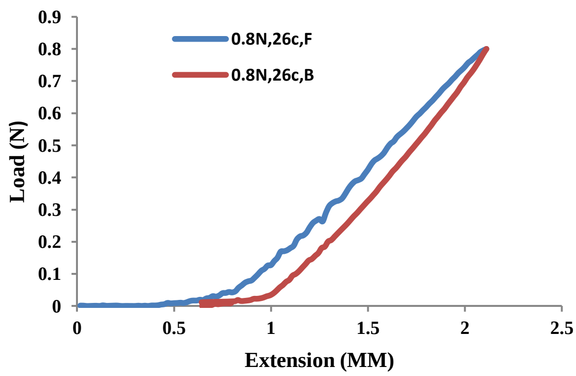


Figure 5. Crushing test with 0.8 N force at the room temperature (26°C). F shows forward and B shows backward loading of the sample

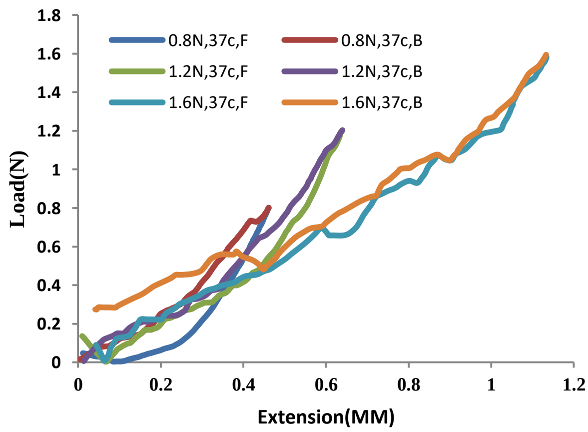


Figure 6. Crushing test with1.6, 1.2 and 0.8 N force at 37°C

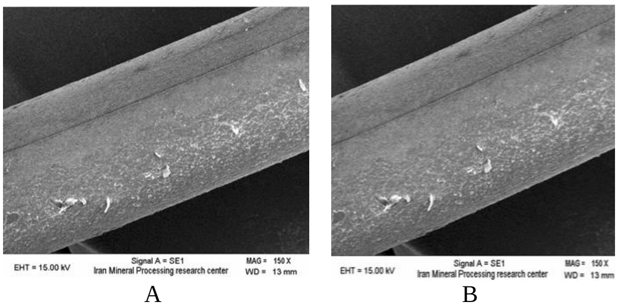


Figure 7. SEM image of the stent: (A) before and (B) after crushing test

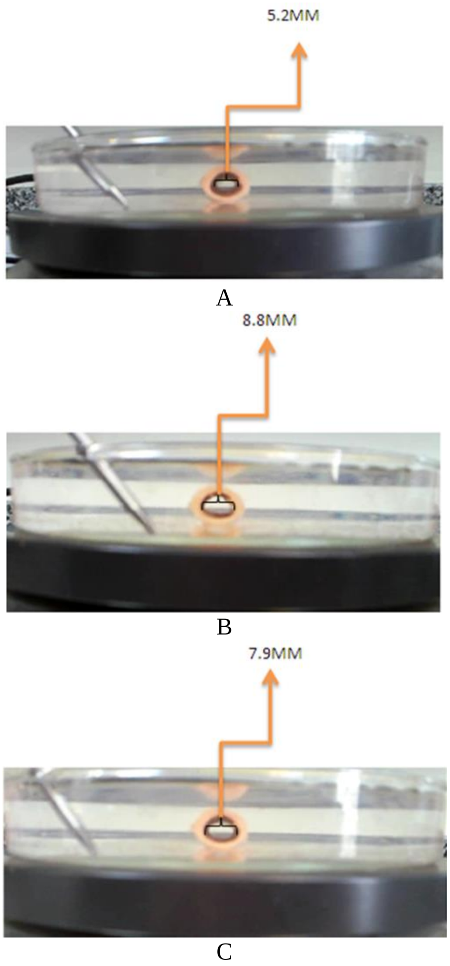


Figure 8. Effect of SBF temperature on patency of the stent after implantation inside an artificial vessel at: (A) 32, (B) 37 and (C) 42°C

4. CONCLUSIONS

Fabrication of spring-model stent for implantation inside femoral vessel by orthodontic Ni-Ti wire proved practically achievable. According to the results, for making a stent with this wire type, 15-20 minute heat treatment is enough for fixation of the shape. Longer heat treatment caused higher phase transformation temperatures which were not desirable for appropriate operation of the stent. The produced stent could be crimped and then fixed in a catheter ready for delivering to position of the application. They were implanted inside an artificial vessel which then opened up to the apt size required. It could exert enough force against the vessel wall and prevented vessel wall from unwanted collapse.

5. ACKNOWLEDGEMENTS

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چکیده

استنت یک لوله توری فلزی برای باز کردن عروق مبتلا به انسداد است. آلیاژ Ni-Ti یک فلز مناسب برای ساخت استنت است؛ زیرا توانایی اعمال تنش و کرنش مناسب برای باز کردن رگ مسدود را دارد. این مقاله به کاربرد سیم ابرالاستیک نایتینول برای ساخت نمونه استنت قابل استفاده در رگ فمورال می پردازد. برای دستیابی به دماهای استحاله و رفتار سوپرالاستیسیته/حافظه داری مناسب، پیرسازی در ۵۰۰ درجه سانتی گراد برای دوره های زمانی مختلف انجام شد. خواص مکانیکی و ساختاری آلیاژ از طریق تحلیل روبشی گرمایی (DSC)، میکروآنالیز پروب الکترونی (EPMA) و مطالعات متالوگرافیکی تعیین شد. توانایی تحمل فشار اعمالی دیواره رگ با استفاده از آزمون له شدگی مورد مطالعه قرار گرفت. براساس تصاویر میکروسکوپ الکترونی روبشی (SEM) ، یکپارچگی سطح در اثر اعمال کرنش از بین نرفت. از کانال مصنوعی سیلیسیومی در سیال شبیه سازی شده بدن (SBF) در ۳۷ درجه سانتی گراد برای کاشت استنت تحت نیروی له شوندگی استفاده شد. بازیابی تنش و کرنش اعمال شده به دیوار کانال بعد از خروج استنت از کاتتر مورد بررسی قرار گرفت.

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