Fatigue Crack Propagation in Drawn Filled NiTiNol Wires for Medical Applications

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Abstract Shape memory alloys have vast use in medical applications. Currently it is mainly about the construction of stents or other types of implants. Nowadays, new types of wires with shape memory effect are tested for medical use. These wires are prepared as drawn filled tubes. Drawn filled wires have unique mechanical and physical properties. Specific properties of drawn filled wires arise through a combination of two materials with different physical properties. This article is devoted to fractographical analysis of fatigue fracture process in drawn filled wires made of nitinol with platinium core.

Introduction

Shape memory alloys (SM) and especially NiTiNol based materials (NitiNol, an alloy composed of 55% nickel and 45% titanium), have enjoyed a long-standing interest with scientists and inventors. Shape memory alloys have vast use in all areas of engineering, especially in the mechatronics or medical engineering [1-5]. These alloys are often used in medical applications [5-11]. The role of material science, particularly metallurgy, in biomedical implants has grown considerably in recent years. Perhaps the most common medical applications of shape memory materials are stents, whether coronary stents or stents used in the esophagus or gastrointestinal tract (other applications made from NiTi are guidewires and embolic filters). The main reason for the use of materials with shape memory effect in the design of stents is the ability to "self-expand". Self-expandable metallic stents are typically inserted at the time of endoscopy, usually with assistance with fluoroscopy or x-ray images taken to guide placement. [12]. Perhaps the most important properties of stents is their fatigue life, fatigue life of stent must be more than ten years. Stent failure can cause internal bleeding resulting in death of the patient.

One way to achieve improved mechanical properties of wires is a combination of several materials with different properties into one functional unit. Currently, such composites (drawn filled wires) are tested for the production of stents. Drawn filled wire or Drawn Filled Tube (abbreviation DFT is also used as trademark) is a metal-to-metal composite developed to combine the desired physical and mechanical attributes of two or more materials into a single wire or ribbon system. As a result of extreme compressive forces imparted during the processing of the dissimilar materials, the mechanical bond formed between surfaces has been found to be metallurgically sound. This feature has given rise to a number of novel applications of drawn filled wire. The composite typically uses the outer sheath to impart strength while the core material is designed to provide resiliency and for medical aplication very important properties such radiopacity and enhancement of magnetic resonance imaging visibility.

DFT wires

Composites are long used in engineering and have found use in nearly every area of industry. Drawn Filled Tube is composite material that involves the filling and drawing of a tube. The most important feature of NiTi alloys is the mettals ability to exhibit the psuedoelasticeffect when deformed at a temperature slightly above the austenite finish temperature A_F. Deformation at austenite finish temperature A_F is normally accommodated by the formation of stress induced martensite and if the applied stress disappeared, the martensitic phase spontaneously transforms back to its original austenitic state, thereby returning the wire shape to its original form [13-15]. Properties of Nitinol–platinum composite wires are described by several different parameters. The composite wires are described by the ratio of core diameter D_C to the diameter of wire D_W is $R_D = D_C / D_W$. Two critical characteristics specific to combination of Nitinol or other materials with shape memory effect are the loading plateau and the unloading plateau. Both critical characteristics are associated with austenitic phase of alloy. The loading plateau stress is the stress level at which material at a specific temperature above active austenitic finish A_F (The active austenitic finish temperature is a material property that is measured after heat treatment. This is the temperature at which the material has completely transformed to Austenite, which means that at and above this temperature the material will have completed its shape memory transformation) will force Austenite phase into Martensite. This produces an almost constant stress level over a relatively large range of strain, up to about 8%. The unloading plateau stress is the stress level at which the Martensite will return to the Austenitic phase. The loading plateau can be expresed by equiton:

$$\sigma_{LP} = \sigma_{SLP}\left(1 - R_D^2\right) + \sigma_{UTC} R_D^2 , \tag{1}$$

where, σ_{LP} is composite loading plateau strength, σ_{SLP} is sheath loading plateau strength, σ_{UTC} is ultimate tensile strength of core. The unloading plateau is expresed as:

$$\sigma_{UP} = \sigma_{SUP}\left(1 - R_D^2\right) - \sigma_{UCC} R_D^2 , \tag{2}$$

where, σ_{UP} composite unloading plateau strength, σ_{SUP} is sheath upper plateau strength, σ_{UCC} is ultimate compressive strength of core.

The ultimate tensile strength of the composite material can be written as:

$$\sigma_{UP} = \sigma_{SUP}\left(1 - R_D^2\right) - \sigma_{UCC} R_D^2 , \tag{3}$$

where, σ_U is composite tensile ultimate strength and σ_{SU} is sheath tensile ultimate strength. The NiTi-Pt composite wire is characterised by permanent set in axial deformation, a slight amount of non-recoverable strain is left in the mildly disrupted NiTi matrix.

$$\varepsilon \approx \frac{R_D^2 \sigma_{UTC}}{E_S\left(1 - R_D^2\right)} + \varepsilon_0 , \tag{4}$$

Where, E_S is austenitic modulus of the NiTi sheath, and ε_0 is the permanent set of the reference solid Nitinol material. The ability of NiTi alloy to fully return to its originally shape after flexural deformation is a key performance of SM materials. After relief, a low elasticity platinum core will impede the return of the Nitinol sheath. However, this calculation must be considered rather than tentative, this fact is given by the small size of the wire.

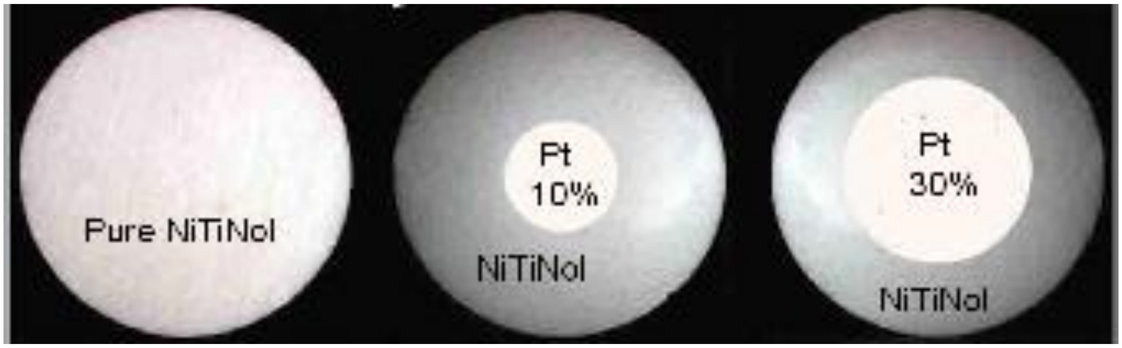

Fig.1. Cross section of : pure NiTiNol wire

Experimental procedure

Three different wires were used for experimental tests in this study: solid NiTiNol wire, drawn filled wire NiTi-DFT-10%Pt (the platinium core represents 10% of wires cross-section) and drawn filled wire NiTi-DFT-30%Pt (30% of wires cross-section), see Fig.1. The diameter of wires was 0,356 mm. The Nitinol specimens used in this study was made of alloy composed 50.8% of Nickel and Titanium 48.5%. Other additives and impurities detected in NiTi alloy are Oxygen 0.03%, Carbon 0.02%, Hydrogen 0.005%. The platinum core was 99.95% commercially and its ultimate tensile strength was 168 MPa. Physical properties of analyzed Nitinol alloy (every physical property must be determined separately for austenitic and martensitic phase): density 6.45 g/cm^3 (austenitic phase), 6.45 g/cm^3 (martensitic phase); modulus of elasticity: 75GPa (austenitic phase), 40GPa (martensitic phase); coefficient of thermal expansion: 11x10^{-6} °C^{-1} (austenitic phase), 6.6x10^{-6} °C^{-1} (martensitic phase). The platinium core was processing according to the thermal and reduction schedule used to produce the Nitinol. Specimens used in this study were heat straightened at a temperature of 497°C in atmosphere composed of technicaly pure Argon (99.98%) at pressure about 0.1MPa and the process was halted for dwell time of 4.5 minutes. The specimens were subjected to pull-off loading and another experiments done in this study are rotary-beam tests. All specimens were loaded to final fracture. All fatigue fracture surfaces were examined with scanning electron microscopy (SEM). Mechanical properties of used wires are expressed in Table 1.

Table 1. Measured mechanical properties of studied wires

Wire	NiTiNol	NiTi-DFT-10%Pt	NiTi-DFT-30%Pt
Ultimate Tensile Strength, [MPa]	335	338	356

The cyclic frequency of 60 Hz was used for rotary-beam testing in a system equipped with wire fracture detection. The rotary-beam tests were carried out on the Rotary Beam U-Bend Wire Spin Fatigue Tester. The wire, with a known length, is mounted into the drive chuck system while the other end is inserted into the free bushing. To prevent vibration, two support guides are positioned on the radius of the specimen, but outside of the apex, such that the guides do not affect the region of maximum strain. Pull-pull test were caried on tensile fatigue machine. Working frequency of tensile fatigue machine was 50 Hz. All tests were performed at room temperature.

Usual separation of fatigue tests on low and high-cycle region (with a boundary around 10^5) is not suitable for the shape memory alloys [16]. The rotary-beam fatigue tests were realised at three different stress levels. At high strain levels ten samples are typically chosen, due to the expected shortness of the fatigue life; in contrast, six samples are tested at low strain levels. Low strain experiments represents the time to failure higher then 10^7 cycles ($N_f > 10^7$). High strain levels samples have the time to failure about 10^3 cycles ($N_f \sim 10^3$). Ten samples were loaded to the fracture in region from 10^5 to 10^7 cycles. For the pull-pull test were used only six samples for every strain level.

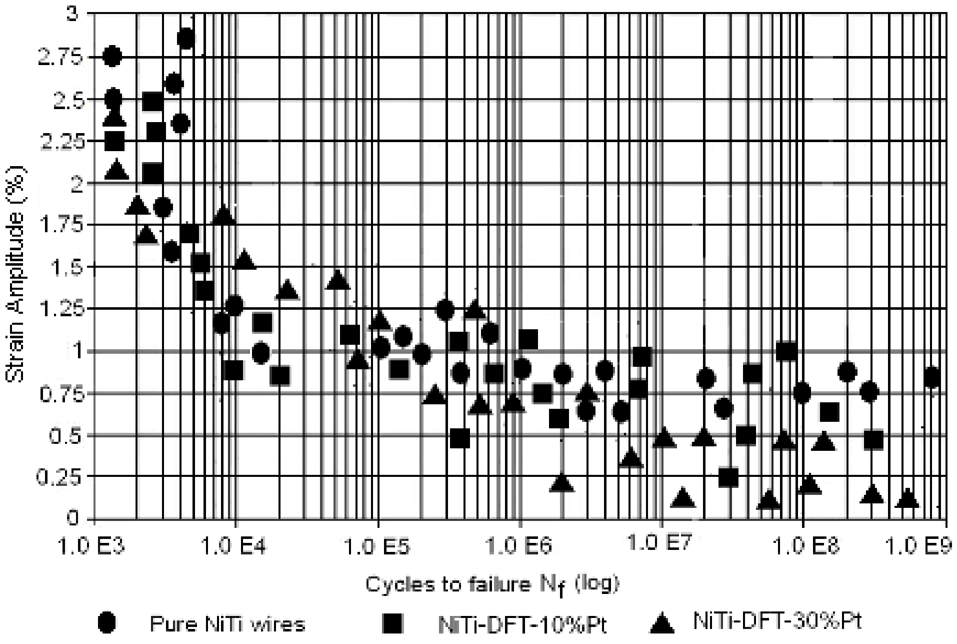

Fig.2. Strain-life diagram

Results

Nitinol fatigue test data are presented in the form of an $\varepsilon \sim N_f$ strain-life diagram as shown in Fig. 2. This figure shows that pure nitinol have higher strain ε in the ($N_f \sim 10^3$) and ($N_f > 10^7$) region, in the region ($10^5 < N_f < 10^7$) strain portion is higher for composite wires. The fracture surfaces may be analyzed using SEM. Fig. 3 (a) represent fatigue surface of pure NiTi wires and Fig. 3(b) shows inclusion. The nonmetallic inclusion is located at the wire surface of the initiation site.The fatigue fracture area comprised about 50 % of the wire cross section. The inclusion had an angular shape and was of approximately 4.4 μm across. High cycle fatigue failure in wires widouth core is initiated by either surface defects or internal non-metallic inclusions closed to the surface. In the ($N_f > 10^7$) region grain boundary as the suspect stress raiser and initiation place was found, see Fig.3(c). These facts are consistent with the observation. In the case of wires with core made of different metal is proces more complicated and fatigue crack often initiated on defects closed near the interface between core material and outer material, see Fig. 3 (d). Metallographic analysis has provided evidence of a bond that is theoreticaly able eliminate interface slippage. This bond is a result of the significant amount of co-processing that is involved in the production of wires. This includes the extreme compressive forces associated with typical wire drawing, and the thermal processes needed to impart ductility to the highly cold-worked materials prior to further deformation. In the case of (N_f ~10^3) region partial tensile rupture of high strength core is typical.

On the other hand, in and ($N_f > 10^7$) region, partial tensile rupture of outer is occur frequently. Only short cracks were observed in nitinol and in platinium core of NiTi-DFT-10%Pt wires. On other hand, both the short and long crack growth regime was found in platinium core of NiTi-DFT-30%Pt wires. The stress intensity factors ΔK were calculated for pull-pull loaded specimens. Fatigue crack growth data for ductile materials are usually presented in terms of the crack growth rate, da/dN, and the stress intensity factor range, ΔK [17,18]:

$$\frac{da}{dN} = C\Delta K^m , \tag{5}$$

The Fig.4 shows the speed (da/dN) versus ΔK in the case of NiTi-DFT-30%Pt, pure platinium wire with the same diameter as the core. The speed (da/dN) is in the case of pure platinium wire higher then in the case of DFT wire.

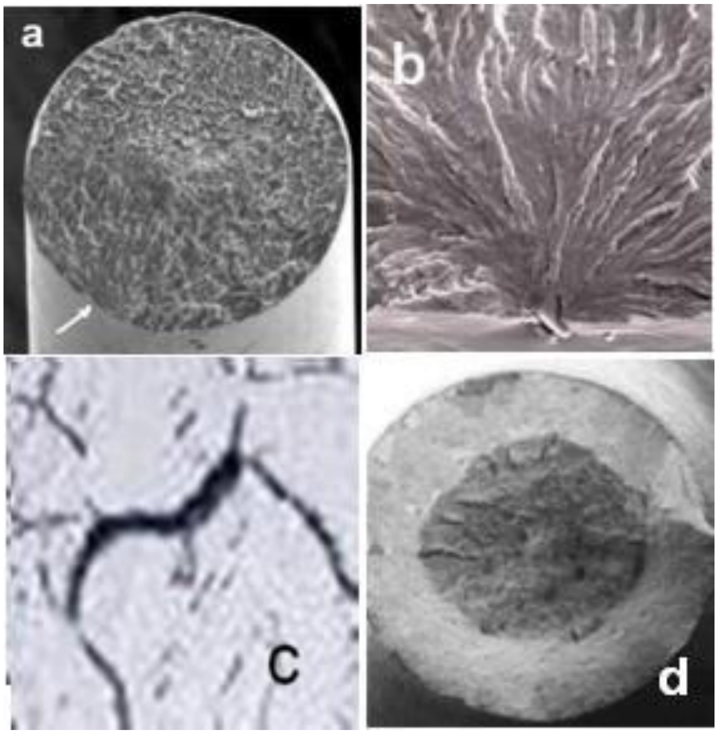

Fig.3. (a) Fatigue surface of pure NiTi wires, (b) site of crack initiation – inclusion

(c) region grain boundary as the suspect stress raiser and crack initiation place

(d) crack initiated on defects closed near the interface between core material and outer material

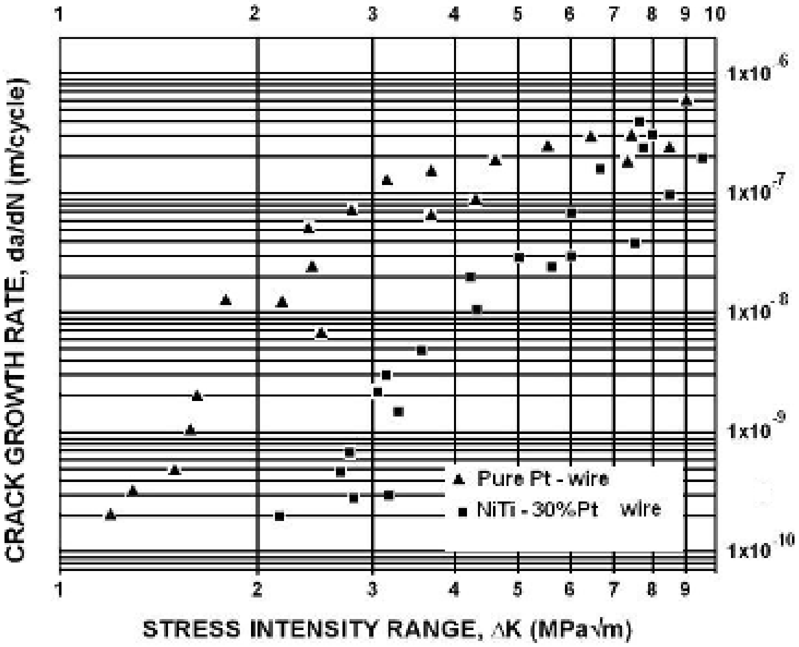

Fig.4. Stress intensity factors ΔK versus (da/dN) diagram

Summary

A fractography study revealed that different fatigue crack initiation mechanisms are active. Based on these different fatigue crack initiation mechanisms, the fatigue data can be divided into three groups. All three groups are significantly different in fatigue process, meaning that the different fatigue crack initiation mechanisms lead to different fatigue lives. Although the ultimate strength of the wire NiTi-DFT-30%Pt appears to be higher then NiTi-DFT-10%Pt and NiTINol wires, its fatigue performance is lower.

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