ENGINEERING CHARACTERISTICS OF DRAWN FILLED NITINOL TUBE

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ABSTRACT

The elastic behavior of various thermally set Nitinol-DFT®-Platinum[1] composite wires was investigated in both axial and flexural modes. Two varieties of composite materials were analyzed, each unique in the amount of cross-sectional area made up of Platinum. The measured properties of these composites were found to be partially dependent on the mode of testing employed, the mechanical properties of the core, and the methods of processing. Theoretical relationships were used to predict the loading and unloading plateau strengths, the stress hysteresis, and the ultimate tensile strength of the composite materials. These derived values were then compared with data obtained from actual testing. Additional work was done to study the effect of the platinum core on the permanent set of the composite in both axial and bending load situations. The low core composite was found to exhibit strength and permanent set characteristics very similar to solid Nitinol wire.

KEYWORDS

Nitinol composite, NiTi-DFT-Pt, NiTi-DFT-Platinum, NiTi-DFT®-Pt, DFT wire, composite wire, Nitinol wire, superelastic composite, pseudoelastic composite, radiopaque composite, radiopaque wire, radiopaque guidewire, Nitinol guidewire, Nitinol stent, DFT wire stent, stent, radiopaque stent, radiopaque guidewire, guidewire.

INTRODUCTION

The binary nickel-titanium alloy system, otherwise known as Nitinol, plays an important role in today's medical device technology [1]. Nitinol has found use in a wide range of applications: the material is used in guidewires, stents, temperature-sensitive actuators, cardiac massage devices, etc. The fundamental benefit of NiTi over most other metallic alloys is its ability to return elastically after significant strain deformation. The strain recovery characteristics of Nitinol depend upon many variables, including processing methods, Nitinol material composition, test temperature, mode of deformation, and material configuration [2].

Primarily due to its low density, Nitinol wire is often difficult to locate under fluoroscopic examination [4]. In recent years, advances in materials processing technology have allowed the use of Nitinol in various composite forms [3]. Highly dense cores such as Platinum, Tantalum, and Gold are capable of providing enhanced visibility under x-ray fluoroscopy. The three materials tested here were a solid Nitinol wire, Nitinol 10% platinum composite, and a Nitinol 30% platinum composite wire (See Figure 1).

Figure 1 [Original Magnification: 200X]: The three materials tested here were (from left to right): Solid Nitinol wire, NiTi-DFT-10%Pt, and NiTi-DFT-30%-Pt. A clean Nitinol to platinum interface, as shown, is an important characteristic of Nitinol composite material.

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A designer must consider a number of important variables when attempting to achieve enhanced visibility under X-ray fluoroscopy using a composite Nitinol wire. The core material selection, composite geometry, and processing conditions can all significantly impact end properties of composite wire. The primary question of interest remains: to what extent are the superelastic properties of Nitinol degraded by the presence of a radiopaque core?

The most widely publicized characteristic of nickel-rich NiTi is the material's ability to exhibit the psuedoelastic effect when deformed at a temperature slightly above the austenite finish temperature. Deformation at this temperature is normally accommodated by the formation of stress induced martensite (SIM) and upon removal of the applied stress the martensitic phase spontaneously transforms back to its original austenitic state thereby returning the wire shape to its original form [5,6].

To design composite Nitinol wires with enhanced radiopaque performance, it is necessary to consider the mechanical effects caused by the core on the superelastic effects of the Nitinol. In this investigation the performance of Nitinol–platinum composite wires were evaluated by looking at several key mechanical parameters commonly used in the industry. The effects studied were the loading plateau stress, unloading plateau stress, permanent set, stress strain hysteresis, and bending stiffness. This paper does not attempt to quantify fatigue performance.

EXPERIMENTAL PROCEDURE

Two different Nitinol-Platinum composite wires were processed using equivalent reduction and thermal practices. As a base for comparison, a solid Nitinol specimen was processed identically to the composite wires. All of the Nitinol used in this study was binary alloy containing nominally 50.8 at% Nickel. All of the specimens were heat straightened at a temperature of 495°C in Argon at a controlled dwell time of less than 5 minutes. These materials were tested at a room temperature of 23°C for various mechanical properties and compared to theoretically derived data. All of the axial testing was performed at a strain rate of 2% per minute using a 12.7 cm [5 inch] gage length.

Table 1: Test sample description including ingot properties, overall outside diameter, and nominal coldwork prior to final straightening heat treatment process.

Material	Ingot A_s Temperature [°C]	Overall Diameter mm [in]	% Coldwork prior to Heat Treat
Solid Nitinol	-30.00	.281 [.01108]	40% nom.
NiTi-DFT®-10%Pt	-24.61	.282 [.01111]	40% nom.
NiTi-DFT®-30%Pt	-28.84	.282 [.01111]	40% nom.

The platinum material was 99.95% commercially pure produced in accordance with ASTM B561. Much of the analysis that follows assumes a platinum core ultimate tensile strength of 172 MPa [25 ksi]. This value was found by processing solid platinum fine wire according to the thermal and reduction schedule used to produce the Nitinol. This material was tested for axial properties after completing a final thermal heat treatment identical to the composites to which it was compared (see Figure 2).

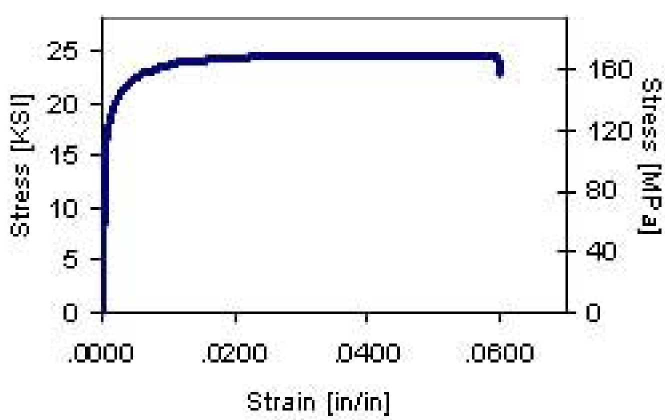

Figure 2: CP Grade platinum wire was processed according to the Nitinol composite regimen, the stress quickly approached 172 MPa after just 1% strain. The platinum core is believed to resist fracture in axial loading beyond 6% strain due to the compressive forces exerted by the surrounding Nitinol.

RESULTS AND DISCUSSION

LOADING PLATEAU STRENGTH

The loading plateau strength of Nitinol occurs in pseudoelastic material during load application through a range of approximately 1% to 8% strain. It was assumed the deformation stress in the core through this plateau range was approximately equivalent to the platinum ultimate strength of 172 MPa [25 ksi]. Under this assumption, Equation 1 can be used to predict the loading plateau stress of a Nitinol composite with a low elasticity core.

$$\sigma_l' = \sigma_l(1-X) + \sigma_c X \quad \text{(loading plateau stress)} \tag{1}$$

Where, $X \equiv CoreRatio = \dfrac{ID^2}{OD^2}$, $\sigma_l' =$ Composite loading plateau Strength, $\sigma_l =$ sheath loading plateau strength, $\sigma_c =$ ultimate tensile strength of core.

UNLOADING PLATEAU STRENGTH

The unloading plateau strength is directly related to the strain restoring force required to return the composite material to its original form. During strain recovery, the lower plateau strength must be sufficiently high to plastically deform the less elastic core material. Similar to the loading stress relationship (Equation 1), the unloading stress can be estimated by Equation 2. This relationship was used to predict the unloading stresses in this study by assuming equivalent ultimate core strengths in both tensile and compressive modes.

$$\sigma_u' = \sigma_u(1-X) - \sigma_c X \quad \text{(unloading plateau stress)} \tag{2}$$

Where, $\sigma_u' =$ composite unloading plateau strength, $\sigma_u =$ sheath upper plateau strength, $\sigma_c =$ ultimate compressive strength of core.

In Equations 1 and 2, the sheath plateau stress refers to the known plateau stress for a solid reference Nitinol wire having similar composition and processing conditions. Both predictive expressions for loading and unloading composite stress depend upon a core material that deforms at a stress near the core materials ultimate strength without fracture during a typical Nitinol axial strain cycle of 6% (see Figure 3). The assumption regarding the deformation stress of the core material would not be valid if the core were to fracture or separate from the Nitinol sheath during loading. A composite sample that had undergone a 6% strain cycle was dissected and found not to have fractured. The predicted and measured plateau stresses are in close agreement and presented graphically in Figure 4.

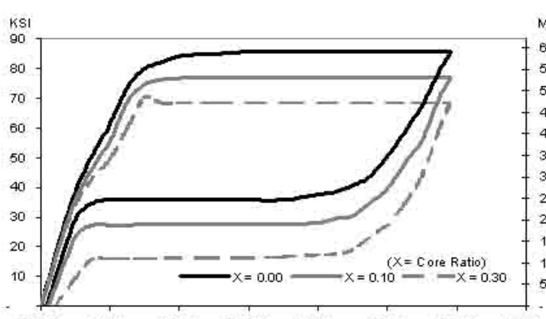

Figure 3: Each specimen was pulled at a strain rate of 2% per minute to a total of 6% strain and then allowed to fully return. Shown are cycle curves for solid Nitinol wire, NiTi-DFT-10%Pt wire, and NiTi-DFT-30%Pt wire.

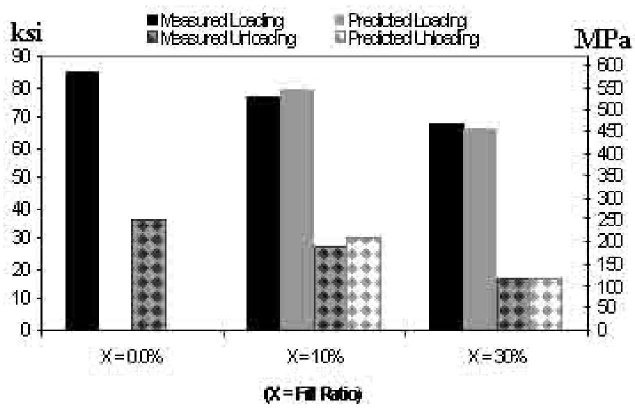

Figure 4: Equations 1 and 2 were shown to closely predict the actual plateau strengths of each of the composite wires.

ULTIMATE TENSILE STRENGTH

The ultimate tensile strength of the composite materials was predicted by assuming the platinum core to reside at its ultimate tensile strength through failure. Equation 3 represents the relationship for the composite ultimate strength.

$$\sigma'_{ts} = \sigma_{ts,s}(1 - X) + \sigma_c X \quad (3)$$

The agreement between the predicted and measured values for the ultimate tensile strength was found to be within a 4% margin of difference (see Table 2).

Table 2: Actual versus predicted ultimate tensile strength of composite Nitinol wires. Table includes total strain to failure for each.

Parameter	Nitinol	NiTi-DFT®-10%Pt	NiTi-DFT®-30%Pt
Ultimate Tensile Strength, MPa [ksi]	1504 [218]	1414 [205]	1134 [165]
Predicted Ultimate Strength, MPa [ksi]	1504 [218]	1373 [199]	1090 [158]
%Difference	0.0%	2.9%	3.9%
Measured Strain to Failure	15.50%	15.10%	14.70%

STRESS HYSTERESIS

Stress hysteresis has been defined in this document as the difference between the upper and lower plateau strengths. In most medical Nitinol applications, knowledge of the stress hysteresis is critical in the design process. In a DFT composite Nitinol material, the stress hysteresis will inevitably be affected by the presence of the core species. In this study the composite stress hysteresis was predicted using Equation 4 where $\sigma'_h =$ composite stress hysteresis and $\sigma_h =$ Nitinol sheath stress hysteresis. This result was obtained by subtracting Equation 2 from Equation 1.

$$\sigma'_h = \sigma'_l - \sigma'_u = \sigma_h(1 - X) + 2X\sigma_c \quad (4)$$

$$\sigma_h = \sigma_l - \sigma_u \quad (5)$$

The validity of Equation 3 was checked against actual test results for both specimens. The maximum deviation from the predicted results was found to be just over 3%. These results have been presented in Table 3.

Table 3 Predicted stress hysteresis

Parameter	Nitinol	NiTi-DFT®-10%Pt	NiTi-DFT®-30%Pt
Stress Hysterisis MPa [ksi]	335 [48.5]	337 [48.8]	349 [50.6]
Predicted Hysterisis MPa [ksi]	335 [48.5]	335 [48.6]	338 [49.0]
%Difference	0.00%	0.40%	3.30%

PERMANENT SET IN AXIAL DEFORMATION

When pseudoelastic Nitinol wire is stressed to a level within its upper plateau region and subsequently allowed to return, a slight amount of non-recoverable strain is left in the mildly disrupted NiTi matrix. The magnitude of this non-recoverable strain (i.e. permanent set) is dependent on the type of Nitinol, the processing conditions, the test temperature, and other variables. In a NiTi-DFT-Platinum wire, the force generated by the lower plateau must overcome and deform the core material to its original reference position. However, at a certain level of strain the sheath unloading stress becomes insufficient to further return the core and the force components in the composite reach equilibrium. A force balance (Figure 5) can be applied to the equilibrium state to predict the additive effects of the additional non-recoverable strain caused by the core presence and the inherent non-recoverable strain (ε_0) of the Nitinol sheath.

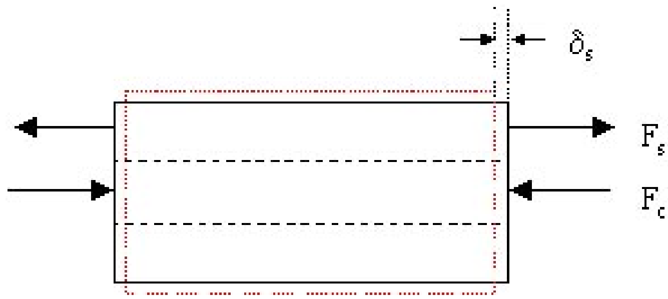

Figure 5: An equilibrium force balance is achieved between the core and sheath materials.

The final relationship for permanent set (Equation 6) depends upon the core ratio (X), ultimate strength of the core (σ_c), austenitic modulus of the Nitinol sheath (E_s), and the permanent set of the reference solid Nitinol material (ε_0). The austenitic modulus was used, assuming the matrix was virtually free of stress-induced-martensite at relatively low levels of strain (typically less than 0. 2 %).

$$\varepsilon' \cong \frac{X\sigma_c}{E_s(1-X)} + \varepsilon_0 \tag{6}$$

After a 6% strain cycle, the measured amount of permanent strain found in the composite wires closely matched the predicted values (see Table 4). The austenitic modulus of the Nitinol used in this calculation was derived directly from the loading curve for the reference Nitinol specimen. This value was calculated as 68.2 GPa [$9.9\text{x}10^6$ psi].

Table 4: Predictions of permanent set in composite Nitinol wire

Parameter	Nitinol	NiTi-DFT®-10%Pt	NiTi-DFT®-30%Pt
Permanent Set [%]	0.08	0.1	0.21
Predicted Permanent Set [%]	0.08	0.1	0.18
Difference	0.00%	-4.90%	13.10%

The 30% core composite product exhibited a permanent set that was 13% greater than that predicted using the above relationship. One explanation for this discrepancy could be related to the strain hardening of the platinum core. It is possible that the platinum could have experienced a very slight increase in ultimate tensile strength as it was initially elongated. As the core material was compressed back to its original geometry, a force greater than that predicted by its assumed 172 MPa [25 ksi] strength may have been present.

PERMANENT SET IN FLEXURAL DEFORMATION

The ability of Nitinol to fully return to its originally set shape after flexural deformation is a key performance characteristic of the alloy. After load removal, a low elasticity platinum core will impede the return of the Nitinol sheath. The less elastic core will resist the return moment generated by the Nitinol. A moment balance (Figure 6) can be applied at equilibrium by assuming that the resisting moments must be equal and opposite.

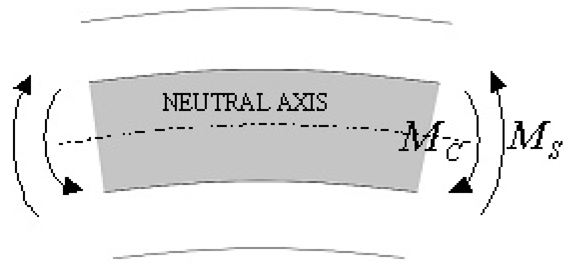

Figure 6: The moment balance between the core and sheath materials.

In the case of a low strength, low elasticity core such as platinum, the stress at the surface of the platinum will quickly approach the ultimate tensile stress during heavy bending. For this reason, it was assumed that during the return phase, the platinum core was at a maximum stress level approximately equivalent to its ultimate tensile strength. The case described should represent the maximum permanent bending deflection that will be present in the overall composite wire structure. Equation 7 can be used to approximate the radius of curvature that will permanently reside in the composite wire after removal of a uniform bending load. This result will only be accurate if the Nitinol is not deformed beyond its elastic limit of approximately 8% strain and the previous assumptions are made with respect the core deformation and stress conditions.

$$\frac{r_b}{d_s} = \frac{(1-X^2)E_s - X^{3/2}\sigma_c}{2X^{3/2}\sigma_c} \qquad (7)$$

The derivation of Equation 7 was performed by solving for the radius of curvature, r_b in the following strain relationship: $\varepsilon_s = \frac{M_s c_s}{E_s I_{NAs}} = \frac{d_s}{2r_b + d_s}$. M_s is the moment generated by the Nitinol sheath, M_c the moment generated by the core; d_s represents the overall composite diameter. The upper curve shown in Figure 7 represents the normalized residual radius of curvature for Nitinol-platinum composite wire. The next lower curves indicate the trend for increasing core strengths of 50,000 and 300,000 psi. As the core strength increases, the amount of strain recovered by the Nitinol will be significantly diminished.

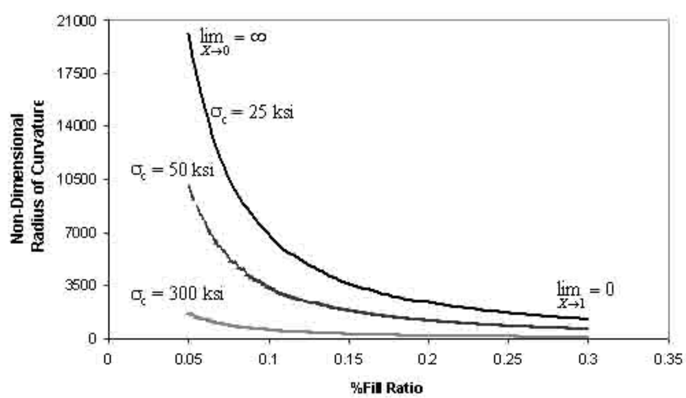

Figure 7: The normalized residual radius of curvature, $\frac{r_b}{d_s}$, plotted as a function of the fill ratio

FLEXURAL LOAD PERFORMANCE

In order to quantify flexural performance, bending moment data was collected for each of the wire specimens. This data was collected on a Tinius Olsen bending stiffness tester at a span of 1.27 cm [1/2 in.] and a load range of zero to 1.13 N-cm [0.10 in-lb]. Figure 8 presents the data that was gathered with respect to the described parameters. During the loading phase, at 20 degree deflection there was virtually no difference between the solid Nitinol and either DFT configuration. Throughout the flex cycle, the NiTi-DFT-10%Pt tracked very close to its solid counterpart in terms of the generated moment. At a deflection of 70 degrees, the moment generated by the NiTi-DFT-30%Pt was around 10% less than that of the solid Nitinol wire. Permanent deflection was measured in neither the solid Nitinol nor the 10% composite wire. The 30% composite wire exhibited a permanent deflection of approximately 2 degrees.

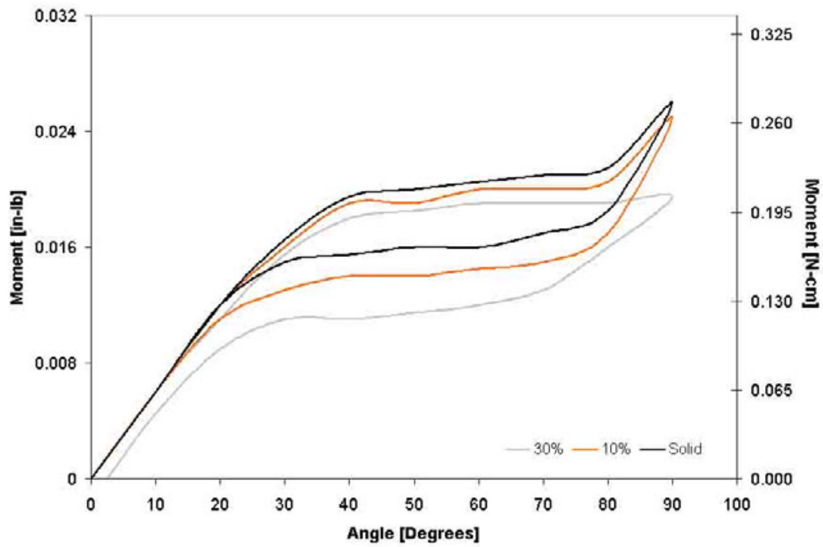

Figure 8: Flex cycle curves for solid Nitinol, NiTi-DFT-10%Pt, and NiTi-DFT-30%Pt wire.

CONCLUSIONS

The predictive equations set forth for the plateau stresses, ultimate tensile strength, stress hysteresis, and permanent set yielded excellent quantitative results when compared with the measured values. A medical device engineer should be able to reasonably predict the mechanical performance for a variety of NiTi-DFT-Pt configurations. Furthermore, assuming appropriate material properties were available, other core materials could be modeled using the same equations. The close agreement between the predicted and measured results strongly supports the previously stated assumptions, namely:

- The platinum core deforms at a stress level near its ultimate strength during the 6% strain cycle and through failure.
- The integrity of the platinum core is maintained through the 6% strain cycle.
- During strain recovery, the Nitinol matrix is essentially free of SIM at less than 0.20% strain, thus allowing use of the austenitic modulus for permanent set prediction.

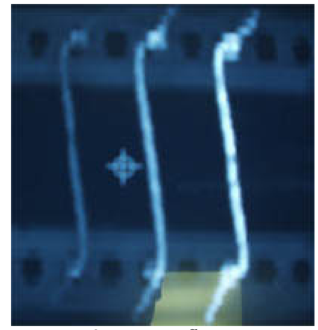

Figure 9: X-ray fluoroscopy of materials; from left to right: solid Nitinol, NiTi-DFT-10%Pt, and NiTi-DFT-30%Pt wire.

The impact of platinum on the radiographic performance of Nitinol composite wire is substantial even at the lowest core ratio tested (see Figure 9). In addition to enhanced radiopacity, the NiTi-DFT-10%Pt wire possessed mechanical properties very similar to solid Nitinol. The NiTi-DFT-30%Pt wire exhibited strongly increased radiopacity with somewhat diminished mechanical performance. All three of the wires performed well during flexural strain testing.

There are some applications in which high core ratios could be used without adverse effect. As an example, the moment generated by the Nitinol sheath in a NiTi-DFT-30%Pt composite could be used to resist blood vessel closure in applications such as stents or filters. In a product such as a guidewire where proximal to distal angular control is required, or straightness is critical, the NiTi-DFT-10%Pt would be a much better choice.

In recent years, the overall trend in medical devices has been decrease their profile, and thus use finer and finer wire products that tend to disappear under most imaging conditions. Nitinol DFT composite products allow designers to realize both excellent mechanical and radiopaque properties.

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