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Research Paper

Rotary-bending fatigue characteristics of medical-grade Nitinol wire

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ABSTRACT

The rotary bending fatigue properties of medical-grade Nitinol wires were investigated under conditions of 0.5–10% strain amplitudes to a maximum of 10^7 cycles. The results from this study provide insight into the behavior of Nitinol under fully reversed ($\varepsilon_{min}/\varepsilon_{max}=-1$) fatigue conditions for three compositions, two surface conditions and three test temperatures. For pseudoelastic conditions there are four distinct regions of the strain-cycle curves that are related to phases (austenite, stress-induced martensite, and R-Phase) and their respective strain accommodation mechanisms. In contrast, there are only two regions for the strain-cycle curves for thermal martensite. It was further observed that the strain amplitude to achieve 10^7-cycles increases with both decreasing test temperature and increasing transformation temperature. Fatigue behavior was not, however, strongly influenced by wire surface condition. SEM of the fracture surfaces showed that the fatigue fracture area increased with decreasing strain amplitude. Finite element analysis was used to illustrate strain distributions across the wire as well as to calculate the tension-compression contributions to the rotary bending curves. The results from this investigation are discussed with respect to mechanisms of strain accommodation under cyclic tensile and compressive conditions.

1. Introduction

Nitinol (NiTi) exhibits well-known shape memory and pseudoelastic effects that are due to a first-order cubic (B2) to monoclinic (B19') martensitic transformation (Bhattacharya, 2003; Otsuka and Ren, 2005). This temperature- or stress-induced transformation is fully reversible, whereby strains as great as 10% may be fully recovered. Consequently, Nitinol has become the material of choice for many medical devices that undergo repetitive strain excursions, such as self-expanding, endovascular stents (Duerig and Pelton, 1999; Stöckel and Pelton, 2004) and endodontic dental files (Thompson, 2000; Bahia and Martins, 2005; Young and Van Vliet, 2005). Self-expanding Nitinol stents are initially strained 6–10% during crimping, and insertion into a catheter delivery system, with a partial strain release with deployment into the diseased vessel.

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The stent is then subjected to multi-axial cyclic deformations with combinations of mean strain (from stent/vessel oversizing) and strain amplitude (from cardiac cycles and musculoskeletal motions) (Pelton and Schroeder, 2008). In contrast, the outer fibers of the endodontic wires are subjected to alternating compressive and tensile strains with a (nominal) net zero-mean strain for many thousands or even millions of cycles.

A recent review article focused on the effects of processing and microstructure on Nitinol fatigue behavior under strain- and stress-life or damage tolerant conditions (Robertson and Pelton, 2012). It is now well known that thermomechanical processing has a profound effect on the mechanical behavior of Nitinol (Pelton and DiCello, 2000; Drexel and Selvadury, 2008; Pelton, 2011). Surprisingly, however, very few rotary-bending fatigue investigations have been published on medical-grade Nitinol with modern thermomechanical processing (Reinoehl et al., 2001; Sawaguchi and Kausträter, 2003; Sheriff and Pelton, 2005; Wick and Gong, 2005; Young and Van Vliet, 2005). Rotary-bending fatigue studies are a convenient method to determine the effects of processing parameters, compositional variations, and surface conditions on fatigue behavior of wire or microtubing under (nominal) zero-mean strain conditions. For these testing conditions, strain amplitude is approximated by the ratio of wire radius to radius of curvature, so a range of strain amplitudes may be achieved with slight adjustments to the bending curvature.

Miyazaki and co-workers studied the effects of strain amplitude (0.8–3.5%) and temperature (35–125 °C) on fatigue life on $Ni_{50.9}Ti_{49.1}$ wires to 10^6 cycles (Kim and Miyazaki, 1997; Miyazaki and Mizukoshi, 1999). Their wires were cold drawn 30% and then aged at 400 °C for 1 h, resulting in an Austenite Finish (A_f) temperature of 40 °C. A general trend of increasing fatigue life with decreasing test temperature in the low-cycle fatigue regions (high and intermediate strain–amplitude regions) was observed. From their limited data, the fatigue strain at 10^6 cycles appeared to be insensitive to temperature to a strain amplitude of 0.8%. The authors characterized the fatigue behavior with respect to the phases present at the respective test temperatures. Reinoehl and Bradley compared fatigue results at room temperature from 0.8% to 2.1% strain amplitude to a maximum of 10^7 cycles for wires that were manufactured from two ingot sources (Reinoehl et al., 2001). The two sets of wires were cold-drawn to a diameter of 0.267 mm and thermally treated at 500 °C to achieve A_f values of ~10 °C. They concluded that there were no substantial differences in the fatigue behavior despite the differences in ingot source and inclusion type, size and distribution. Sawaguchi and Kausträter (2003) measured the number of cycles to failure out to 10^6 cycles with strain amplitudes between 0.75% and 3% with three wire diameters (1.0, 1.2, and 1.4 mm). The $Ni_{50.9}Ti_{49.1}$wires were produced by hot extrusion, followed by 40% cold work, straightened at 510 °C for 1 min with a resultant 50 nm grains and transformation temperatures that ranged from 11 to 26 °C (DSC austenite peak temperature). However, since their experiments were conducted in room temperature air (non-isothermal conditions), they also observed differences with rotation speed as well as wire diameter.

The purpose of our research, therefore, is to report on an investigation of the rotating bending fatigue characteristics of shape memory and pseudoelastic medical-grade Nitinol wires as part of a larger investigation of Nitinol fatigue behavior. Specifically, medical-grade wires with four compositions, two surface conditions, and three test temperatures were investigated from 0.5% to 10% strain amplitudes to 10^7 cycles. These data are compared to conventional fatigue theories and are characterized with finite element analysis and fractography.

Table 1 – Wire materials and testing conditions.

Composition	A_f (°C)	Surface condition
$Ni_{50.8}Ti_{49.2}$	2	Bright
$Ni_{50.8}Ti_{49.2}$	4	Black
$Ni_{50.6}Ti_{49.4}$	7	Black
$Ni_{49.5}Ti_{50.5}$	64	Black

2. Material and methods

2.1. Material surface and transformation temperature

Table 1 summarizes the compositions, A_f, and surface conditions of the commercially available binary, 0.6 mm diameter Nitinol wires used in this investigation. Two of the wires have a composition of $Ni_{50.8}Ti_{49.2}$, whereas another wire had a slightly lower Ni concentration ($Ni_{50.6}Ti_{49.4}$) but was processed to achieve similar transformational and mechanical properties; all three wires conform to ASTM F2063 (ASTM, 2005). The fourth wire, $Ni_{49.5}Ti_{50.5}$, has a greater Ti content with a concomitant greater transformation temperature than the other wires. The wires were processed from vacuum arc re-melt (VAR) ingots and were manufactured in-house per industry standard methods (Pelton and DiCello, 2000). Prior to final thermal shape setting at ~500 °C, the wires were drawn with approximately 45% cold work (as measured by cross-sectional area reduction after the previous full anneal). The resultant transformation temperatures were approximately 5 °C (range 2–7 °C) for the nickel-rich compositions and 64 °C for the titanium-rich composition, measured by bend-free recovery methods per ASTM F2082 (ASTM, 2006a).

Surface conditions of the wires were characterized as *black* or *bright*; both conditions are used commercially as starting material for wire-based Nitinol medical devices. The *black* wires have their native thermal oxide with approximately 300 nm thickness. The *bright* wires were mechanically polished continuously after the wire drawing operation to remove the black oxide and draw lines as well as to smooth the surface. The mechanical polish is approximately equivalent to a 400 grit mechanical polish.

2.2. Uniaxial tensile characterization

Monotonic uniaxial tensile tests on a minimum of three wires from each condition were conducted with a mechanical