Characterizing Non-metallic Inclusions in Superelastic Nitinol Fine Wires and Effects on Mechanical Properties

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Continued advancements in medicine, biomedical device design, and a drive to minimally invasive surgeries have combined to create a stronger demand for increasingly smaller medical devices and surgical tools. In many of these applications, the components or subcomponents of the devices are fine wires. Stents, filters, staples, clips, catheters, needles, and orthodontic braces represent a sampling of the devices used in cardiology, orthopaedics, surgical instruments, and dentistry, with wire diameters ranging from the tens to hundreds of micrometers [1-3]. Miniaturization of these devices and/or subcomponents requires increased performance from less material, and therefore necessitates investigation of the connection between material processing and performance. Moreover, it is imperative to understand how impurities that result from processing affect the lifetime performance of that material. Research on the material purity of fine Nitinol wires and its effect on fatigue performance are not frequently found in literature [4].

This study compared superelastic Nitinol fine (< 140 µm) wires that were identified by the manufacturer as standard purity (SP) and high purity (HP) material. The nominally equiatomic nickel and titanium wires were provided with bright (SP) and black oxide (HP) surface finishes. Wires segment were mounted and polished in the longitudinal direction for traditional metallographic analyses. Non-metallic inclusions (NMIs) were generally titanium-rich nickel oxides with varied morphology, occurred with and without pores, and as stringers along the wire drawing direction. Combined inclusion/pore area percentages ranged from 0.08% ± 0.04% (HP) to 1.44% ± 0.26% (SP) when measured with scanning electron microscopy. Plasma focused ion beam (PFIB) serial sectioning and x-ray microscopy (XRM) were used to characterize the combined NMI/pore volume percentages yielding 0.09% (HP) and 0.47% (SP) for PFIB and 0.001% (HP) and 0.11% (SP) for XRM. Variability in the percentages was attributed to both the area/volume characterized due to the technique limitations and the ability to clearly resolve the individual non-metallic inclusions from the pores and matrix material.

Microindentation hardness measurements showed gradients across the wire diameter and were compared to strength and fatigue behavior. The center-line measurements averaged 359 ± 6.4 HK (HP) and 329 ± 7.2 HK (SP) while mid-radius values were similar for both at 366 ± 5.1 HK and 361 ± 6.0 HK, respectively. Higher tensile and upper plateau strengths were observed in SP (1573 MPa and 515 MPa, respectively) than HP (1420 MPa and 392 MPa, respectively) with greater reduction in area for HP (92.2%) vs. SP (80.2%). Fatigue enhancement was observed in HP wires in flex bending fatigue at strain amplitudes (ε_a) from 0.67-11% with a run-out at 10^6 cycles and rotating bending fatigue (ε_a 1.2-1.5%) though run-out at 10^8 cycles. Fractography showed crack initiation attributed to a pore or inclusion at the surface or sub-surface with fatigue initiation feature areas ranging from submicron to nearly 20.4 μm^2. Characterization techniques showed that HP contained less large-dimension NMIs/pores than SP leading to less potential stress concentrations and points for crack initiation. These differences in microcleanliness were reflected in the fatigue results where the HP exhibited enhanced fatigue resistance in both low cycle fatigue and high cycle fatigue regimes [5].

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[5] The authors would like to acknowledge DARPA HAPTIX N66001-15-C-4014, Arthur P. Armington Professorship, Confluent Medical, ASM International, Thermo Fischer Scientific, Zeiss Microscopy, ORS, UCONN CAMMA, and CWRU AMMRC & SCSAM for their support

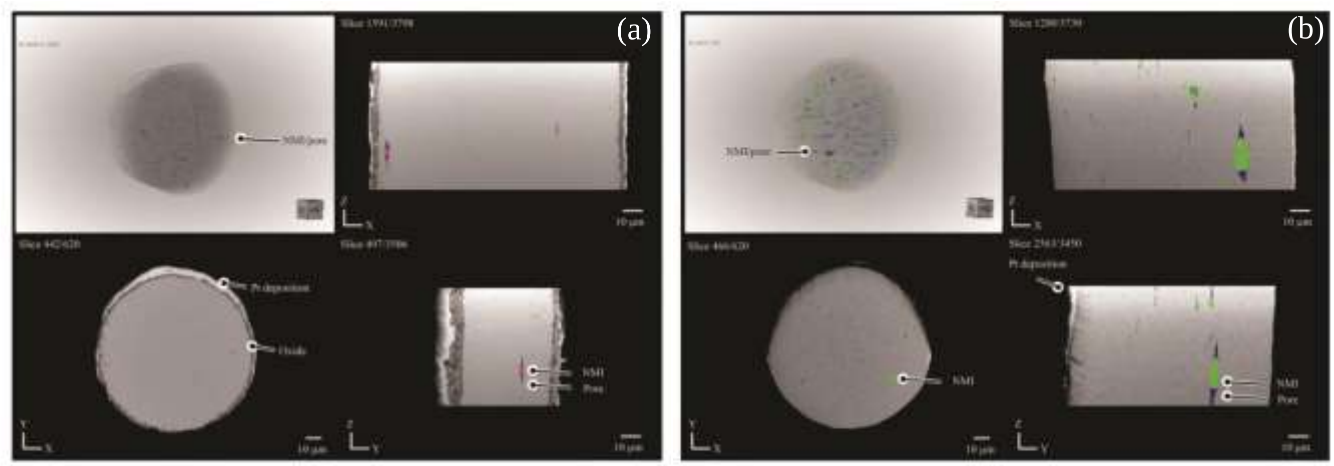

Figure 1. PFIB 3D reconstruction of (a) HP Nitinol wire showing the segmentation process. The colored features represent NMIs (pink) and pores (blue) and (b) SP Nitinol wire showing the same segmentation process and colored features representing NMIs (green) and pores (blue).

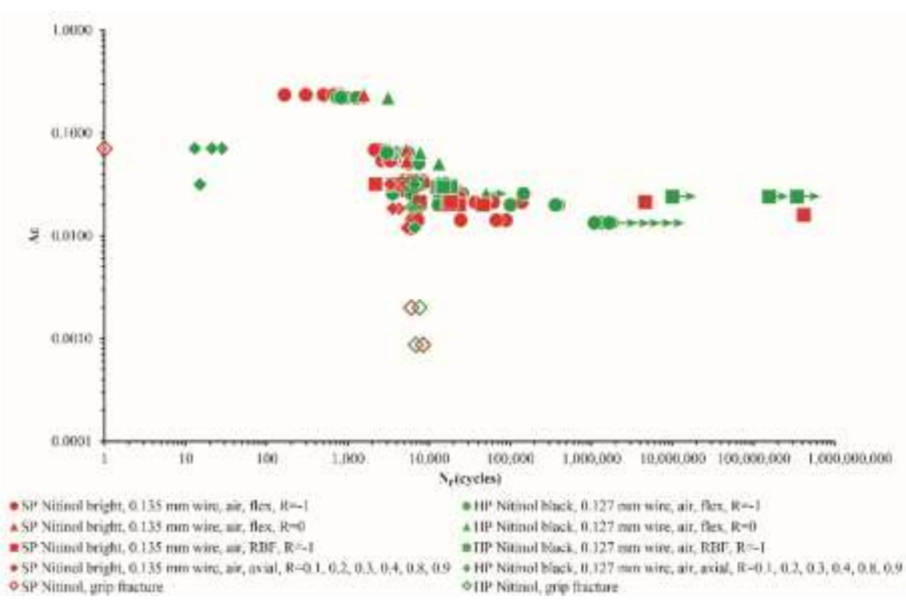

Figure 2. Comparison of all HP Nitinol and SP Nitinol fatigue data representing axial fatigue for R = 0.1, 0.2, 0.3, 0.4, 0.8, and 0.9, flex bending fatigue R = 0 and R = -1, and rotating bending fatigue with data points defined as run-out denoted with right-facing arrows and specimens that broke at/near the grip indicated with open diamonds