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Research paper

Impact of thermomechanical texture on the superelastic response of Nitinol implants

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ABSTRACT

The phenomenon of superelasticity in near-equiatomic NiTi, which originates from a first-order martensitic phase transition, is exploited in an increasing number of biomedical devices, most importantly endovascular stents. These stents are often manufactured from microtubing, which is shown to be highly textured crystallographically. Synchrotron X-ray microdiffraction provided microstructural, phase, and strain analysis from Nitinol tube sections that were deformed *in situ* along longitudinal, circumferential, and transverse orientations. We show that the large variation in the superelastic response of NiTi in these three tube directions is strongly influenced by the path that the martensitic transformation follows through the microstructure. Specifically, in severely worked NiTi, bands of ⟨100⟩ grains occur whose orientation deviates markedly from the surrounding matrix; these bands have an unusually large impact on the initiation and the propagation of martensite, and hence on the mechanical response. Understanding the impact of these local microstructural effects on global mechanical response, as shown here, leads to a much fuller understanding of the causes of deviation of the mechanical response from predictions and unforeseen fracture in NiTi biomedical devices.

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1. Introduction

Deployment of self-expanding stents, manufactured from near-equiatomic NiTi (Nitinol) is an effective, lower risk therapy alternative to surgery and represents a major therapy in the fight against cardiovascular disease (Schillinger et al., 2006). Due to the first-order phase transition from cubic austenite (B2) to monoclinic martensite (B19') phase, Nitinol can undergo several times larger reversible deformation than conventional biomaterials such as stainless steel or

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titanium. This feature makes Nitinol optimally compliant to withstand large crimp and deployment strains (up to 10%) as well as cyclic deformations experienced by the peripheral vessels, while maintaining vessel patency. Indeed, such superelasticity in Nitinol has been exploited in many other biomedical applications, from endodontic files to spinal prostheses (Duerig et al., 1996).

Nitinol biomedical devices are manufactured from wires, thin rods, sheet, strip or thin-walled tubes, all of which are formed by a series of hot and cold working operations from cast ingots to final shape. The multistage thermomechanical processes impart deformations that significantly affect the microstructure (grain size, defect density, and crystallographic orientation). For example, there is a million-fold reduction in the cross sectional area in fabricating a 0.5 mm diameter wire from the original (500 mm diameter) as-cast ingot. As such, the grain refinement and crystallographic texture formation resulting from these manufacturing processes can be considerable.

Fig. 1 shows two versions of a generic stent pattern (http://nitinoluniversity.com/open-stent-design/) laser cut from thin-walled Nitinol tubes. The as-cut pattern (a) is from a 2 mm diameter tube (the so-called "closed configuration"), whereas the pattern in (b) is from an 8 mm diameter tube ("open configuration").[2] After mechanical expansion of both stents to 10 mm diameter and "shape setting" (stress relief at ∼500 °C), the stent that was laser machined in the closed configuration (c) is macroscopically identical to that of open configuration stent (d). Note, however, that the orientation of the tube axis, and hence the drawing direction (indicated by the arrow) relative to the major axis of the struts in the open and the closed configuration of stents, are significantly different. As demonstrated by Pelton et al. (2008), the radial pressure exerted by a Nitinol stent is proportional to the mechanical properties of the individual "V" sections of the final stent geometry. Though still poorly understood, the influence of microstructural orientation on the superelastic response of NiTi is known to be significant (Robertson et al., 2006). Consequently, differences in crystallographic texture for macroscopically identical stents may result in stents experiencing markedly different cumulative radial forces and fatigue properties.

Cardiac cycles and musculoskeletal motions subject biomedical devices to millions of complex deformations. For example, a stent deployed in the superficial femoral artery (SFA) undergoes severe multiaxial displacements from pulsatile motion (ca. 4×10^7 cycles annually) plus up to 60% rotation and up to 20% contraction (at a rate of ca. 1×10^6 cycles annually) as the leg is bent during a walking cycle (Cheng et al., 2006, 2010). To design a device to withstand these frequent and severe deformations, finite element analytical (FEA) models of complex structures such as endovascular stents are generated by creating a fine elemental mesh of the geometry (Rebelo and Perry, 2000). The models attempt to incorporate the highly nonlinear mechanical response of NiTi from global monotonic stress–strain relationships. But as the microstructural and textural effects alluded to above are still poorly understood, they are ignored in current commercially available computational design models; this leads to predictions that are only qualitative and deviate significantly from the actual response at large deformation, as demonstrated previously (Mehta et al., 2007). Occasionally, therefore, an implant unexpectedly fractures *in vivo*, resulting in loss of vessel patency, and requires a major invasive surgery (Pelton et al., 2008).

X-ray microdiffraction (μXRD) from a synchrotron radiation source was the primary tool used to characterize the microstructural features, including grain size and grain orientation. This technique is the only method with sufficient spatial resolution (<10 μm) to allow detailed analysis of a 'bulk' specimen on the order of a stent strut. Furthermore, this technique provides the highest resolution strain gauge that can distinguish among different modes of strain accommodation, *i.e.,* elastic, plastic, or phase transformational strain.

Consequently, the goal of the investigation reported here is to develop a deeper understanding of the role of microstructure and texture on the martensite phase transformation and superelastic response.

2. Material and method

Medical-grade $Ni_{50.8}Ti_{49.2}$ tubing (5.5 mm OD with 0.3 mm wall thickness) was laser machined along the longitudinal axis. These tube sections were then shaped into flattened "sheets" by a two-step process at 500 °C. This flattening process induced approximately 10% strain into the material, comparable to the expansion strains used during forming endovascular Nitinol stents from laser-machined tubes (Pelton et al., 2000), but insignificant in comparison to the strain introduced on formation of the tube from the ingot. Micro-dogbone-shaped tensile specimens were then laser machined from these flattened sheets at three different orientations to the Nitinol tube drawing axis: 0°, (longitudinal), 45° (transverse), and 90° (circumferential); see Fig. 2 inset for a photograph of a dogbone. The dogbone specimens were given an additional heat treatment of 700 °C for five minutes to obtain a nominal austenite grain size of about 10–20 μm. The larger grain size was necessary to permit the 1 μm X-ray spot size to resolve the grain-by-grain transformation. These thermal treatments resulted in an austenite finish temperature of 15 °C, as measured by the bend-free recovery method in accordance with ASTM F 2082 (2006). After electropolishing, these dogbones had a gauge section of 800 × 200 × 200 μm.

Global force–displacement curves were obtained at room temperature from dogbone specimens machined in the three different orientations, using a 2.3 kg_f maximum force capacity displacement-controlled rig. The measurements were performed at room temperature on the same *in situ* rig used for the microdiffraction measurements. The rig grips had 2 mm diameter alignment pins to hook the corresponding machined alignment holes in the flared grip section of the dogbone. The rig displacement was gradually

[2] The terms "open" and "closed" configuration refer to the manner in which the stents are manufactured and should not be confused with "open" and "closed" cell geometry verbiage used in stent design.