Phase Transformations in Nitinol and Challenges for Numerical Modeling

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Abstract

Over the past several years, NiTi alloys have seen an increased use in medical devices where components are compressed to catheter dimensions, delivered through tortuous anatomy and deployed in various diseased locations within the human body. Nitinol is well suited to the design constraints imposed by such conditions, however, only modest progress has so far been made regarding long term predictions of the structural integrity of such components. Challenges in predicting long term reliability of superelastic NiTi medical components include the consequences of manufacturing tolerances, uncertainty with regard to in vivo loading and the inherent complexity of superelastic material behavior. In this article, we focus on the latter challenge and discuss ongoing research to characterize the effects of phase transformation and accurately obtain calibrated material constitutive relations for polycrystalline Nitinol materials. Our approach involves a combination of full-field strain measurements using phase shifted Moiré interferometry and the development of improved finite element methodology to account for various material behaviors and simulated conditions. Phase shifted Moiré interferometry provides unprecedented accuracy in the measurement of strains and displacements in the vicinity of stress risers such as notches and can be used to guide and verify finite element models for implantable superelastic devices.

Introduction

Nitinol has become a material of importance in medical applications as it allows designers to overcome a wide range of issues related to the miniaturization of medical devices and the increasing trend for less invasive, and therefore less traumatic procedures across medical specialties. Applications using Nitinol include self-expanding stents, graft support systems, filters, baskets and various other devices for minimally invasive interventional and endoscopic procedures. The primary feature responsible for this widespread use is the stress-induced reversible phase transformation responsible for recoverable macroscopic strains of 8% or more and has been the subject of much study over the years.

The use of Nitinol for implantable medical devices is not without its challenges. Intrinsic to the material is a lower fracture toughness and faster crack growth rates than conventional engineering materials [1]. Another challenge is the measurement of key properties such as elastic moduli which is made difficult by the contributions of stress-induced martensite and martensite reorientation during testing.

In the face of these challenges, Finite Element Analysis (FEA) has proven useful in the design and development process as it allows designers to predict and optimize critical performance parameters, such as force exerted by a stent on a vessel wall, and can correlate design geometry and guide accelerated test methods. Critical to the application of finite element methodology are meaningful material property inputs and confidence in the relationship between the analysis outputs and the real world.

Recently, the availability of constitutive models for Nitinol has improved, but in light of present experimental evidence, interpretation and use of these models requires caution. Here we present a methodology to guide future advancements of theory and practice and the corresponding evidence that suggests the notion that strains in excess of 2 percent may be erroneous, at least at the material level.

Moiré Interferometry

Moiré interferometry is a well established photomechanics technique for characterizing detailed material response [2]. Diffraction gratings are replicated on the sample surface which is then observed in an interferometer as shown schematically in Figure 1, to reveal fringe patterns representing in-plane surface strains for any applied loading condition. Phase shifting is a technique of acquiring and processing fringe patterns that extends conventional interferometric measurements through over-sampling and automated image processing [3].

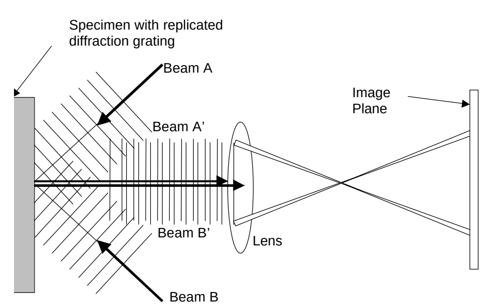

Figure 1: Schematic of Moiré interferometry technique.

The technique is used on two specimen configurations as shown in Figure 2. Both specimen types are made from Nitinol with an A_f below room temperature; however the dogbone spoecimens were made from material from two different sources and are refered to as material X and material Y. The specimens are tested in a custom built load frame with data collection for load using a load cell, cross-head displacement using an LVDT and strain using a custom built extensometer.

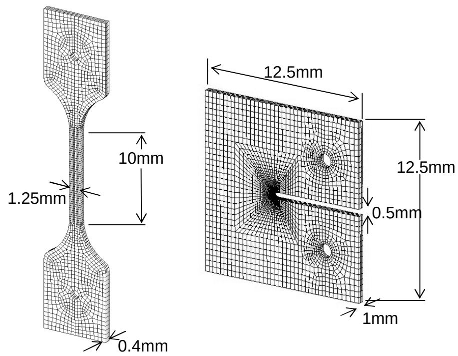

Figure 2: Schematic of FEA meshes of the dogbone and compact tension (CT) specimens showing dimensions.

Figure 3 shows a representative image of the phase shifted Moiré interferogram of the opening mode displacement fields of the CT specimen at partial loading. Close to the notch tip large deformations obscure the clarity of the fringes and indicate the extent and character of stress induced martensite transformation. Here gratings were replicated by the typical molding process using epoxy and a master grating (shown in the left half of Figure 3); however, in order to improve resolution and negate the effects of grating thickness which results in smearing, gratings were also created using photoresist spun directly onto the surface of the specimen and exposed to a master (shown in the right half of Figure 3). The photoresist grating method results in an extremely thin grating and provides significant improvement to the accuracy of the actual specimen displacements. This is extremely critical for large displacement gradients (i.e. large strains) as is the case for superelastic Nitinol.

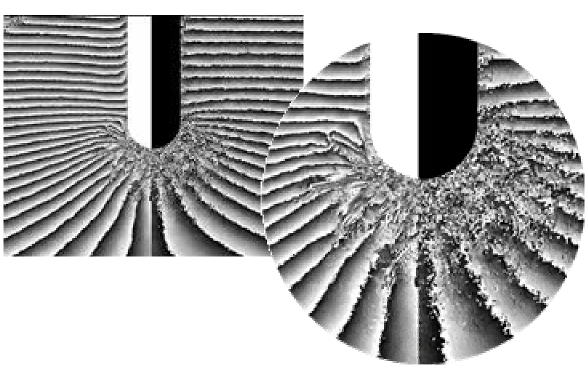

Figure 3: Phase shifted Moiré interferograms and close-up view of the opening displacements of the CT specimen at partial load using epoxy replicated gratings (left half) and using photoresist gratings (right half).

Figure 4 shows a representative series of images of the phase shifted Moiré interferogram of the vertical displacement fields of the dogbone specimen made from material X with increasing tensile load. The two images on the right exhibit localization as Lüders-type bands. This is consistent with the thermomechanical measurements and observations made by Tan et al., [4] and others. Figure 5 shows the corrsponding stress-strains behavior with strains calculated from the phase shifted Moiré interferograms along with the far-field measurements made using the extensometer and load cell.

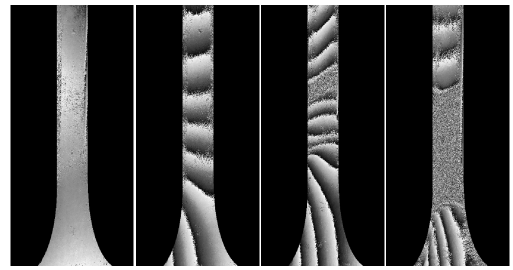

Figure 4: A representative series of phase shifted Moiré interferograms showing a Nitinol dogbone specimen made of material X experiencing increasing tensile loading.

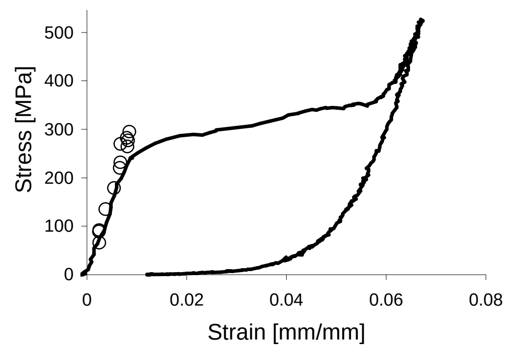

Figure 5: Far-field stress-strain measurements (solid line) and local strain measurements calculated from the Moiré interferograms for the dogbone made from material X.

It is interesting to note that in Figure 5, the only strain values that could be calculated from the phase shifted Moiré interferograms were within the linear elastic region of the Austenite phase of the material. No values could be computed in the region undergoing a phase transformation due to severe loss of coherence of the gratings. The transformation was completely reversible; however, as the band reduced in size and vanished as the applied loading was removed, although this occurred at a significantly lower load.

Figure 6 shows a representative series of images of the phase shifted Moiré interferogram of the vertical displacement fields of the dogbone specimen made from material Y with increasing tensile load. Figure 5 shows the corrsponding stress-strains behavior with strains calculated from the phase shifted Moiré interferograms along with the far-field measurements made using the extensometer and load cell.

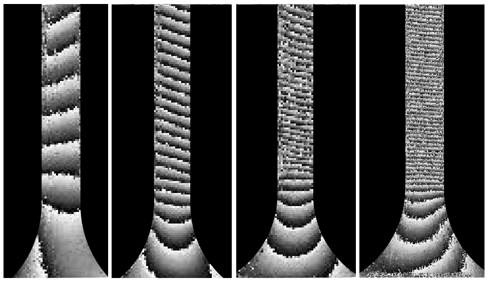

Figure6: A representative series of phase shifted Moiré interferograms showing a Nitinol dogbone specimen made of material Y experiencing increasing tensile loading.

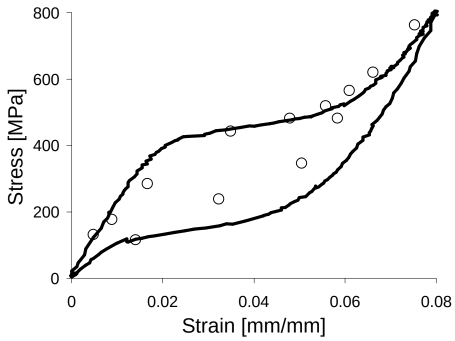

Figure 7: Far-field stress-strain measurements (solid line) and local strain measurements calculated from the Moiré interferograms for the dogbone made from material Y.

It is aparent that the behavior of material Y is significantly different from that of material X and there is no localization or Lüders-type bands forming in the gage section. In material Y, stress induced martensite transformation appears to occur microscopically and uniformly distributed along the length of the gage section of the specimen. If one considers only the far-field stress-strain behavior (the solid lines of Figure 5 and 7), however, one may conclude that the materials simply differ in terms of their A_f temperatures.

It is interesting, however, that if we then go back to the CT specimen (also made from material X) and shown in Figure 3 and examine the transformation zone at the tip of the notch in more detail, the formation of a Lüders type band is not directly apparent. Here there is no nucleation and propagation of the martensite phase as observed in the dogbone specimen made from the same material, rather we see what appears to be stress induced martensite transformation occuring at the microscopic level, on the order of 10 microns or less. This suggests that the difference between uniform and localized Lüders-type deformation is not only related to the material, but related also to geometric effects as is the case for the CT specimen. Here the near-field strains at the tip of the notch are controlled by the far-field elastic strains thereby preventing large scale transformations from occuring, but rather distributed microscale transformations occur.

Upon unloading, we see something quite remarkable. Figure 8 shows the phase shifted Moiré interferogram at the tip of the notch of a CT specimen where a distinct band separating the notch tip from the rest of the specimen can be identified. This band, or jump in the fringe pattern represents the persistence of a significant displacement discontinuity and suggests the same phenomenon Lüders-type deformations as that observed in the dogbone specimen made from material X. The band, however, and all the fringes disappeared as the loading was further decreased indicating full reversal of the stress induced martensite transformation.

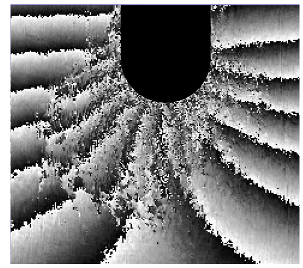

Figure 8: Detailed view of a phase shifted Moiré interferogram of a transformation zone in polycrystalline NiTi in the vicinity of the notch when the sample is partially unloaded.

Finite Element Analysis

We adopted a thermodynamic approach following Boyd and Lagoudas [5] and Wollants et al., [6] for constitutive models for shape memory and superelastic materials based on first principles. This approach also allows for different temperature dependant elastic properties for austenite and martensite and accommodates both mechanical and thermal loading. The material constitutive relation was implemented as a user defined subroutine for the commercial finite element code ABAQUS.

Figures 9a) and b) show comparisons between the FEA prediction of the martensite volume fraction and the corresponding phase shifted Moiré interferograms for the CT specimen and the dogbone specimen made from material X, respectively.

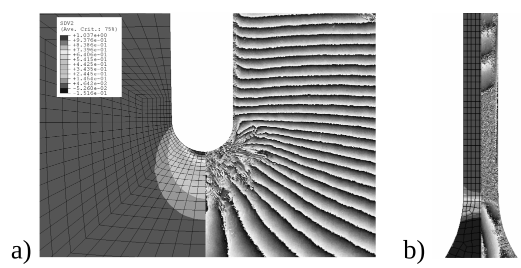

Figure 9: Comparison of martensitic transformation zone from FEA using user defined material subroutine (left) and actual phase shifted Moiré interferograms (right) for a) the CT specimen, and b) the dogbone specimen.

In the image on the right of Figure 9a) for the CT specimen, areas near the notch tip where the FEA predicts greater that 90 percent martensite volume fraction, have clearly undergone stress induced martensite transformation where the diffraction grating encoding the surface strains becomes deformed beyond the limits of information theory, although not in a smooth symmetric manner as the FEA prediction which is based on a homogeneous continuum representation of the material. Further from the notch, as the martensite volume fraction decreases, the fringes become more distinguishable. Based on our measurements, this occurs at approximately 40 percent volume fraction martensite. For the dogbone specimen, however, there is significantly more difference between the results obtained using FEA and the experimental measurements. Again, the FEA predicts smooth results over the gauge length of the specimen, whereas, the Moiré fringe patterns show a localized phenomena in the form of a Lüders-type band of phase transformation.

Relevance to Medical Device Fatigue

Of paramount concern for the use of Nitinol in implantable medical devices is the issue of material fatigue. Numerous investigators have examined the cyclic failure of Nitinol alloys for different specimen shapes such as bars, wires, and plates. In general, Nitinol does extremely well in a strain- or displacement-controlled fatigue, but does less well in a stress-controlled fatigue environments [7]. Early available fatigue data for superelastic Nitinol using rotary bend testing of wire [8] provided a baseline for medical device design and characterization. Others noted the dramatic effect that mean strain has on the fatigue behavior of Nitinol [9].

While these studies provided tremendous insight into the behavior of Nitinol under cyclic fatigue loading, many questions remained regarding the effects of processing, sample geometries, metallurgical texture and loading modes. More recent testing [10-13] and particularly [7] confirm the importance of carefully reproduced fatigue test conditions and demonstrate the complex relationship between mean and alternating strain and fatigue life for Nitinol. This complex relationship is contrary to the behavior typically observed in conventional engineering materials [14].

The current results indicates that phase transformations in Nitinol do not follow a continuum description of nucleation and propagation. This suggests that FEA predictions of strain need to be interpretted carefully in circumstances where phase transformation is expected. While we have no evidence for a toughening mechanism associated with stress induced martensite formation, the data does support stress shielding and redistribution effects around concentrations. Furthermore, since Nitinol is a two-phase material, the fatigue performance cannot be interpreted in the traditional continuum sense, but rather by considering the roles of the two phases present in the structure.

Conclusions

These results demonstrate the importance of accurate modeling of the transformation behavior in NiTi. Localized stress induced martensite transformations and the polycrystalline nature of the material can result in local strain raisers which can strongly influence overall fatigue performance. Accurate characterization of the material behavior becomes increasingly important as component feature size is reduced as in the case of implantable medical devices. Lastly, Moiré interferometry is an instrumental experimental technique enabling for the development and verification of more accurate modeling capabilities.

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