THE EFFECTS OF NOTCHES AND GRAIN SIZE ON TRANSFORMATIONS IN NITINOL

Paul E. Labossiere* and Kenneth E. Perry**

*University of Washington, Seattle, WA, 98195-2600 **ECHOBIO, 579 Azalea Avenue NE, Bainbridge Island, WA 98110

ABSTRACT

Over the past several years, NiTi shape memory alloys have seen a tremendous increase in applications, which exploit the materials' ability to repeatedly recover inelastic strains up to 8 percent. Despite our solid understanding of the relationship between the deformation modes and the transformations in NiTi, we do not have a clear understanding of the fracture initiation processes. In this paper, we present a research methodology to characterize the effects of crystallographic orientation and grain size on the deformations and transformations in Nitinol. The approach involves a combination of full-field strain measurements using Moiré interferometry and finite element modeling. We explore the effects of localized stress raisers such as notches and grain boundaries, and discuss implications for medical device design.

INTRODUCTION

Recently, superelastic Nitinol has been used for many commercial applications ranging from eyeglasses to cell phone antennae. Nitinol is also attractive for many medical applications because not only can the material sustain large deformations (strains up to 8 percent) that are fully recoverable, it also exhibits excellent biocompatibility and corrosion resistance. Addressing reliability, in terms of fatigue and fracture, are of major concern for these applications (see for example: Vaidyanathan et al., 2000; <u>McKelvey</u> and Ritchie, 2001). Traditional fracture mechanics is not suitable for two reasons: a) component feature dimensions are typically small (on the order of several grain diameters); and b) highly nonlinear constitutive relation. Furthermore, characterization is complicated by the multiaxial nature of the stress fields inducing martensitic transformations near stress raisers such as corners. Thus, the best that one can do is to take a more experimental approach in order to characterize component reliability; however, this experimentation alone is both time consuming and costly and does not lend itself well to the design process.

Here we seek to better our understanding of Nitinol by observing full-field strains and local transformations in the vicinity of a rounded notch. The approach consists of employing phase shifted Moiré interferometry in combination with detailed finite element analyses of a standard compact tension (CT) specimen. We discuss the limitation of FEA and propose approaches to develop more accurate modeling capabilities.

EXPERIMENTAL METHODOLOGY

Moiré interferometry is a well established photomechanics method for producing fringe patterns representing relative in-plane deformation of a sample (Post et al., 1994). Diffraction gratings are replicated on the sample surface, which is then observed in a two-beam interferometer as illustrated in Fig. 1. The pair of primary diffracted wavefronts are collected by a lens and imaged as an interference fringe pattern. The fringes formed represent relative in-plane surface displacements for any given loading condition. The difficulties associated with using this technique include vibration isolation, phase extraction, and post processing.



Figure 1. Schematic of Moiré interferometry technique.

The grating serves to provide an optical encoding of the in-plane strain fields. It is desirable to have a grating that is sufficiently thin so as to minimize smearing of the strains and have negligible influence on the mechanical behavior of the specimen. Gratings are replicated by a molding process using epoxy and a master grating. Several epoxy manufacturers and types were tested in order to minimize the aforementioned negative effects and to provide a replicated diffraction grating with sufficient elastic properties to cover the deformation range of interest (zero to ten percent strain). Compact tensions specimens, t = 12.5mm, notch diameter = 0.5mm, notch length = 7.1mm with a superelastic heat treat ($A_f = 10C$) are tested under opening mode loading at room temperature. A custom, four beam fiber optic interferometer with dual field piezoelectric phase shifter is used to acquire fringe patterns. A photograph of the apparatus is shown in Fig. 2. Both the load and crosshead displacement were monitored throughout each test using a load cell and LVDT, respectively.



Figure 2. Photograph of the experimental apparatus and close-up of mounted specimen.

Phase shifting is a technique of acquiring and processing fringe patterns that extends conventional interferometric measurements (Perry and McKelvie, 1993; Perry, 1996). Three or more fringe pattern images are digitized in rapid succession with a known optical phase shift introduced between frames. These images represent an oversampling of the Moiré data of interest and can be processed to generate a

noise-reduced image that inherently preserves fine spatial detail. This is particularly important in the case of fringe patterns involving discontinuous displacement fields. Figure 3 shows typical wrapped fringe patters of the horizontal (opening mode) and b) vertical in-plane displacement fields at low load prior to any detectable martensitic transformation.

Figure 3. Images of wrapped Moiré fringe patterns of the a) horizontal (opening mode) and b) vertical displacement fields near the notch at low load prior to martensitic transformation.

NUMERICAL ANALYSIS

We adopted the approach of Boyd and Lagoudas (1996) for constitutive models for shape memory and superelastic materials based on first principles. In their approach, the second law of thermodynamics is written in terms of the Gibbs free energy where strain, temperature and martensite volume fraction are included as state variables. An evolution equation for the martensite volume fraction is derived from a dissipation potential and the effective transformation surfaces are evaluated as functions of the state variables. 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 material subroutine for the commercial finite element code ABAQUS v6.2-5. Figure 4 shows a typical uniaxial stress-strain behavior of superelastic Nitinol demonstrating the difference in the activation stress for the phase transformation from austenite to martensite and from martensite to austenite. Accurate characterization of the material behavior becomes increasingly important, especially as component feature size is reduce to same order of the grain diameter and difficulties arise due to instabilities associated with the martensite and twin boundary mobility (Liu and Yang, 1999).



Figure 4. Uniaxial stress-strain relation implemented as a user define material model and used in FEA.

A three-dimensional finite element model taking advantage of symmetry was constructed of the specimen geometry. Preliminary two-dimensional models indicated that the geometry was such that neither plane stress nor plane strain assumptions were applicable. The plane stress model greatly over predicted the magnitude of the vonMises stress at the notch, whereas the plane strain model resulted in an under prediction. Furthermore the extent of the region undergoing a phase transformation was poorly predicted in both cases. This is due to the fact that a complex multiaxial stress state exists at the tip of the notch where a relatively large hydrostatic (or dilatational) stress component exists. The martensitic phase transformation is not induced by this stress component, but rather it is induced by the deviatoric (or distortional) stress component.

Figure 5 shows the FEA solution for the vonMises stress, maximum principle strain and the volume fraction of transformed martensite on the surface at the tip of the notch at moderate load once some local

martensitic transformation has occurred. Note the left boundary represents a plane of symmetry in the model. Three distinct regions can be identified in these plots. Far from the notch, no martensite has formed (the volume fraction of martensite of Fig 5c) is zero) and the material is in the lower linear portion of the stress-strain curve of Fig. 4. Closer to the notch where martensite has formed (nozero in Fig 5c), the vonMises stress of Fig. 5a) is relatively constant and the material is on the upper plateau of the stress-strain relation of Fig. 4. Closer to the notch, the strains of Fig 5b) are greater than 6 percent and the ratio of martensite is unity indicating a fully transformed structure and the vonMises stress shows a rapid increase in this region.



Figure 5. a) VonMises stress, b) maximum principle strain, and c) volume fraction of martensite ahead of the notch in superelastic Nitinol at moderate load showing local martensitic transformation.

FEA AND EXPERIMENTAL RESULTS

A) Comparison of FEA and experimental load vs. pin displacement results

We investigated two sets of specimen orientations: one in which the notch was oriented parallel to the similar texture, these specimens have a slightly different microstructure in that the grains are elongated in the rolling direction and hence should display slightly different responses. Note that it is reasonable well known that the material behavior depends significantly on the crystallographic orientation as shown by experiments on single crystal NiTi (Gall et al., 2002). Figure 6 shows a typical pin load versus pin displacement measurement for specimens with a notch oriented parallel to the rolling direction. The open circles denote experiments and the Solid lines denote the FEA solution. There is good agreement between the experiments and the FEA during the loading portion of the curve; however, discrepancies arise during the unloading phase. Some of this may be attributed to relaxation of the load frame and flexure at the pin loading points. The load displacement results, in conjunction with fringe patterns recorded at successive points along the loading and unloading paths, were used to validate the modeling of Nitinol with our material model.



Figure 6. Typical pin load versus pin displacement for various load and unloading paths. The solid lines denote the FEA solution and the open symbols denote experimental values.

B) Comparison of FEA and experimental full-field measurements

The full-field strain measurements from phase shifted Moiré interferometry allow us to further our understanding of the role of transformations on the material response. Figure 7 shows key wrapped fringe patterns during a loading, unloading cycle after martensitic transformation Images prior to the onset of martensitic transformation were shown earlier, see Figs 3a) and b). Figures 7a) and b) show the fringe patterns of the opening mode displacement fields and the corresponding strains, respectively, at the onset of martensitic transformation which occurs in individual grains near the tip of the notch. Note the presence of localized strain concentrations. The experimentally determined strains indicate localized concentrations of approximately two percent which agrees well with uniaxial stress-strain behavior. Figure 7c) shows the fringe patterns at maximum load showing a large transformation region, as evidenced by the loss of fringe coherence at the notch tip.. Figure 7d) shows the fringe patterns partway unloaded below the initial transformation load showing retained transformations. Note that these retained transformed regions are local stress raisers and are potential sites of fatigue crack formation. As feature size decreases, the effects of the individual transformed grains become more significant. Figure 7e) shows the fringe pattern when the specimen is nearing fully unload indicating that the transformations have reversed to the austenitic phase, which is a characteristic specific to superelastic Nitinol.



Figure 7. Fringe patterns of the opening mode displacement fields at a) partial loading and b) the corresponding strain field c) full loading, d) partial unloading below the initial transformation load, and e) nearing full unloading.

Figure 8 shows a comparison between the FEA prediction of the martensite volume fraction and the corresponding phase shifted Moiré fringe pattern for the opening mode. In the image on the right, areas near the notch tip where the FEA predicts greater that 0.9 martensite volume fraction, have clearly undergone stress induced transformation, although not in a smooth symmetric manner as the FEA prediction. Further from the notch, as the martensite volume fraction decreases, the fringes become more distinguishable. Near the notch where transformations have occurred, the diffraction grating encoding the surface strains becomes deformed beyond the limits of information theory. In other words, the grating looses coherency at some strain value. Based on our measurements, this occurs at approximately 0.4 volume fraction martensite. The key point is that the correlation is very good. Even though the experiment is less symmetric and smooth, the FEA still predicts the general trends, including average relative magnitudes as observed through the experiments.



Figure 8. Comparison of martensitic transformation zone from FEA using user defined material subroutine (left) and phase shifted Moiré fringe patterns (right).

DISCUSSION

Phase-shifted Moiré interferometry is an excellent technique for producing detailed displacement and strain fields in superelastic materials. The method is capable of extremely good strain resolution even in the presence of large deformations resulting from stress induced martensitic transformations. The displacement and strain data available through Moiré interferometry is ideal for verifying finite element models and for guiding the development of more comprehensive material models to improve component processing and mechanical performance predictions. These improvements include better modeling of the transformation effects during unloading, transformation zone size and volume fraction, and multiaxial loading effects.

ACKNOWLEDGEMENTS

The authors would like to thank Xiao-Yan Gong at Nitinol Devices and Components (NDC) for providing specimens and Eric Steffler, Vance Deason, Randy Lloyd, Kevin Kenney, Neal Boyce and Tom Walters from the Idaho National Engineering and Environment Laboratory (INEEL) for providing their expertise and resources for the Moiré interferometry experiments.

REFERENCES

Vaidyanathan, R., Dunand1, D.C., and Ramamurty U., 2000, Fatigue crack-growth in shape-memory NiTi and NiTi–TiC composites. *Materials Science and Engineering*, Vol. A289, pp. 208–216.

McKelvey, A.I., and Ritchie, R.O., 2001, Fatigue Crack Growth Behavior in Superelastic and Shape Memory Alloy Nitinol. *Mettalurgical and Materials Transactions A*, Vol. 32A, pp. 731-743.

Post, Han and Ifju, 1994, High Sensitivity Moire, Springer, Berlin.

Perry, K.E. and McKelvie, J., 1993, A Comparison of Phase Shifting and Foorier Methods in the Anlaysis of Discontinuous Fringe Patterns. *Optics and Lasers in Engineering*, Vol. 19, pp. 269-284.

Perry, K.E., 1996, Delamination and Damage Studies of Composite Materials Using Phase-shifting Interferometry, *Optics and Lasers in Engineering*, Vol. 24, pp. 467-483.

Liu, Y., and Yang, H., The Concern of Elasticity in Stress-Induced Martensitic Transformations in NiTi. *Materials Science and Engineering*, Vol. A260, pp. 240-245.

Boyd, J.G., and Lagoudas, D.C., 1996, A Thermodynamic Constitutive Model for Shape Memory Metals Part I. The Monolithic Shape Memory Alloys. *International Journal of Plasticity*, Vol. 12, pp. 805-842.

Gall, K., Dunn, M.L., Liu, Y., and Labossiere, P.E., 2002 Micro and Macro Deformation of Single Crystal NiTi. Journal of Engineering Materials and Technology, Vol. 124, pp. 238-245.