Mean strain effects on the fatigue behavior of superelastic Nitinol alloys: An experimental investigation

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Abstract

Experimental fatigue study has been conducted in this paper to investigate the effects of mean strain on the fatigue behavior of superelastic Nitinol in short life regime. Nitinol alloys are used in many applications such as aerospace and bioengineering in which these alloys may be subjected to cyclic loading with mean strains/stresses. For traditional metals, presence of tensile mean strain/stress can significantly reduce fatigue lives. For superelastic Nitinol alloys, however, a more complex behavior is observed under tensile mean strains and ironically some beneficial effects of tensile mean strains have been reported in the literature. This paper aims to experimentally study the effects of tensile mean strains on the fatigue resistance of superelastic Nitinol alloys. Fatigue tests were conducted at room temperature (~24°C) where the material shows superelastic response. Strain-controlled uniaxial fatigue tests with and without tensile mean strains were conducted on standard solid circular specimens. Cyclic deformation and fatigue of the material was studied. Results from this study demonstrates the detrimental effects of mean strain on the fatigue of superelastic Nitinol at least for comparatively large strain amplitudes. Scanning electron microscopy (SEM) was used to investigate the fracture surface and observe the crack initiation sites. Inclusions such as TiC particles located at or near the surface were found to serve as the main crack initiation sites.

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Peer-review under responsibility of CETIM

Keywords: Superelastic; Nitinol; fatigue; mean strain; fractography

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1. Introduction

Nitinol, an almost equiatomic alloy of nickel and titanium, exhibits superior properties such as superelasticity and shape memory effects, resistance to corrosion, strength and ductility among shape memory alloys. Popularity of Nitinol in various industries is resulted from its unique properties such as high energy dissipation, vibration absorption, good resistance to environmental attacks and nonmagnetic properties [1]. Following the successful performance of the shape memory alloys in the F-14 fighter jets, these alloys are frequently used in more aerospace applications. Actuators, structural connectors, sealers, vibration dampers and inflatable structures are more examples of applications of Nitinol alloys in the aerospace industry. Nitinol has also been used in multiple parts of spacecraft such as hydraulic lines, landing gears, rotors, inlets, flap edges, vortex generators, winglets and wings [2]. In addition to the aerospace applications of Nitinol, this material has been widely used in other fields such as biomedical, automotive and civil engineering. Endovascular stents, endodontic files, vena cava filters and Nitinol implants are some examples of this material in biomedical applications. Nitinol elements have also been used as an energy dissipating tool for shock and seismic applications [3,4].

In most of its applications the Nitinol component is under cyclic loads, and thus, the fatigue behavior of this material needs to be carefully determined. In some applications such as intravascular stents, the component is under a combination of constant (i.e. mean) and alternating strains/stresses. Therefore, understanding the fatigue behavior of Nitinol under nonzero mean strain loading condition is of great interest to many industries. Such an understanding can also facilitate further fatigue analyses under variable amplitude loads [1].

Several studies [1,5,6] have investigated different aspects of property deterioration in Nitinol alloys. However, due to the complexities in mechanical behavior of this material, the fatigue behavior of Nitinol is not yet clearly understood. Compared to other metals, Nitinol can tolerate higher strain amplitudes prior to failure. For example, Nitinol may endure cyclic strain amplitudes ranging from 4% to 12%, whereas for most other metals the total tolerable strain before failure is usually around 1% for the same number of load/deformation cycles. One of the challenges in evaluating the fatigue behavior of Nitinol arises from formation of stress-induced martensite under mechanical loading. Therefore, classical fatigue theories may not be directly applicable to superelastic Nitinol alloys specifically for nonzero mean strain cyclic loads [7].

Previous studies [5,8] reported that unlike other metallic materials, tensile mean strain does not necessarily reduce the fatigue resistance of Nitinol alloys. Experimental works in the literature have demonstrated that for a range of

Nomenclature

| Symbol | Definition |
|--------|------------|
| \(2N_f\) | number of reversals to failure |
| \(E_A\) | austenite modulus |
| \(E_M\) | stress-induced martensite modulus |
| \(N_f\) | number of cycles to failure |
| \(R_e\) | minimum to maximum strain ratio in cyclic loading |
| \(\varepsilon_a\) | strain amplitude |
| \(\bar{\varepsilon}_a\) | equivalent strain amplitude |
| \(\varepsilon_{\min}\) | minimum strain in cyclic loading |
| \(\varepsilon_{\max}\) | maximum strain in cyclic loading |
| \(\varepsilon_{As}^{AM}\) | A → M start strain |
| \(\varepsilon_{Af}^{AM}\) | A → M finish strain |
| \(\varepsilon_{Af}^{MA}\) | M → A finish strain |
| \(\sigma_{As}^{AM}\) | A → M start stress, a.k.a. plateau stress |
| \(\sigma_{Af}^{MA}\) | M → A finish stress |
| \(\sigma_{\max}\) | maximum stress in cyclic loading |
tensile mean strains, the fatigue life of the Nitinol alloys increases by increasing tensile mean strain [5,8]. Pelton and co-workers [5] studied the fatigue behavior of diamond-shape components, as a representation of Nitinol stents, under uniaxial tension-tension test. The strain distribution in the component then was calculated using nonlinear finite element analysis (FEA). Results from [5] show that for a certain run-out life such as $10^6$ or $10^7$ cycles, the alternating strain of the failed specimen does not continuously decrease with increasing mean strains. Similar results were obtained by Tolomeo et al. [9]. Such a behavior is not typical for other metallic materials. Morgan et al. [8] also studied the effects of mean strain on the fatigue life of Nitinol and reported longer fatigue lives for specimens with 6% mean strain compared to specimens with 4% and 2% mean strain for the same strain amplitudes. These experimental observations indicate that the classical formulation for considering the effects of mean strain/stress, such as Goodman relation, are not applicable to Nitinol alloys. However, there is not currently a proposed method in the literature to formulate the fatigue behavior of Nitinol in presence of mean strains. In this paper, uniaxial fatigue behavior of superelastic Nitinol under strain-controlled cyclic loads were experimentally analyzed. Several strain ratios ($R_\varepsilon$) are considered in the experimental program to analyze the effect of various levels of mean strain on the fatigue behavior. Fractography using scanning electron microscopy (SEM) is also performed to determine the inclusions/impurities responsible for the crack initiation.

2. Materials and Testing

Material used in this study is a nominal Ni$_{50.8}$Ti$_{49.2}$ at. % alloy that was purchased in form of straight bars with 10 mm diameter. As-received Nitinol bars were machined to a standard dogbone specimen shape with 7mm diameter at the gage section according to ASTM standard [10], as shown in Fig. 1. Machined specimens then were heat treated at 550 °C for 1 minute in salt bath and then water quenched in iced water to get a superelastic material at room temperature. Appropriate heat treatment to get fully superelastic (up to 3% strain) Nitinol was determined by trial and error for different combinations of annealing temperature, time, environment, and the cooling process and based on the recommendations by Pelton et al. [11]. All the tests were conducted in air at room temperature (~ 24 °C) where the stress-strain response of the material displays superelastic response.

Monotonic tests were conducted in strain-controlled condition up to 6.5% strain using Instron 5882 electro-mechanical tensile machine. Fatigue tests were conducted in strain-controlled conditions at several strain amplitudes (i.e. $\varepsilon_a = 0.4\%$, 0.5%, 1%, 1.1%，1.25% and 1.5%) and three strain ratios ($R_\varepsilon = -1$, 0, and 0.5). MTS 810 uniaxial servo-hydraulic fatigue testing machine was used to apply the cyclic loads. Strain values were measured and
controlled using an MTS uniaxial extensometer with a gage length of 15 mm. For all the tests, the strain rate was held constant ~ 0.1 mm/mm/s to avoid a temperature increase in the specimen, and thus, tests were conducted at different frequencies with respect to the strain amplitude. All the specimens were mechanically polished with sand papers starting from a rough sand paper (grit #320) to a very fine (grit #4000) sand paper to minimize the effects of surface flaws on the fatigue resistance.

3. Results and Discussions

3.1. Monotonic test

Fig. 2 illustrates the tensile stress-strain response of the material, indicating a perfect stress plateau for a strain level between 1% to 3.4%. Material exhibits different moduli in austenitic and stress-induced martensitic regions. Austenite modulus \( E_A \) of the material was calculated to be 73 GPa, while the stress-induced martensite modulus was shown to be ~22 GPa. A plateau stress region in which a significantly large strain range (~2.4%) was recorded for a constant stress, \( \sigma_{AM} = 515 \text{ MPa} \). Monotonic material properties such as the upper and lower plateau stresses and modulus of elasticity are presented in Table 1.

![Fig. 2- Tensile stress-strain response of Nitinol from two specimens](image)

| Property                           | Value   |
|------------------------------------|---------|
| Austenite modulus, \( E_A \)       | 73 GPa  |
| Stress-induced martensite modulus, \( E_M \) | 22 GPa  |
| A→M start stress, \( \sigma_{AM,s} \) | 515 MPa |
| M→A finish stress, \( \sigma_{MA,f} \) | 400 MPa |
| A→M start strain, \( \varepsilon_{AM,s} \) | 1.0%    |
| A→M finish strain, \( \varepsilon_{AM,f} \) | 3.4%    |
| M→A finish strain, \( \varepsilon_{MA,f} \) | 0.6%    |

3.2. Cyclic deformation behavior

The deformation response for the first cycle of fatigue tests at different strain levels and ratios are presented in Fig. 3. Data obtained from the tension and compression cyclic tests shows a stress-induced martensite starting strain
of about 1%. As can be seen from Fig. 3a, the material exhibits perfect superelasticity in both tension and compression. A significant asymmetry in tension and compression stress plateaus can be observed from this figure similar to the observations in other studies [12]. That is, the plateau stress in tension, holds a value about 515 MPa, while in compression the plateau stress is approximately 40% higher about 700 MPa. Moduli of elasticity of tension and compression are very close and approximately 73 GPa.

One of the consequences of the tension-compression asymmetry in fatigue testing of Nitinol is the fact that there is always a compressive mean stress applying on the material in fully reversed strain-controlled tests at higher strain amplitudes. Thus, for strain amplitudes where the material enters the stress-induced martensitic region, fully reversed strain-controlled tests are not fully reversed in stress-based fatigue analysis [1]. That being said, a mean stress/strain model for fatigue evaluation of the Nitinol should also be able to capture the effects of tension-compression asymmetry.

Fig. 3- First cycle stress-strain response of the Nitinol from different specimens at different strain amplitudes and various strain ratios: (a) $R_{ε}= -1$, (b) $R_{ε}= 0$, and (c) $R_{ε}= 0.5$

Fig. 4- Cyclic stress-strain response of Nitinol at different cycles of loading and various strain ratios: (a) $R_{ε}= -1$, (b) $R_{ε}= 0$, and (c) $R_{ε}= 0.5$
compression asymmetry in the material.

For compressive strain amplitudes larger than 1.5%, the possibility of buckling was high, as a result maximum achievable strain level in fully reversed tests was limited to 1.5% to avoid buckling in the specimen. Larger strain amplitudes for fully reversed fatigue tests can be studied by employing larger diameter gage section and smaller extensometer to reduce the length-to-diameter ratio at the gage section. Figures 3b and c present the stress-strain response of the first cycle for tension-tension specimens with \( R_e = 0 \) and 0.5, respectively. As can be seen in Fig. 3b, material is capable of recovering strain amplitudes as large as 3% completely with almost no residual strain. Fig. 3c also includes first half cycle (loading) of the second cycle. This plot shows that the slope (i.e. modulus) of the loading portion of the second cycle in presence of mean strain/stress is not similar to the first cycle and seems to hold a value between the loading and unloading moduli. \( M \rightarrow A \) finish stress and strain values, \( \sigma_{MA,f} \) and \( \epsilon_{MA,f} \), respectively, as listed in Table 1 were measured from the unloading portion of a \( R_e = 0 \) test as can be seen in Fig. 3b.

Similar to other materials, Nitinol exhibits different behavior under cyclic and static loads. Accumulation of residual strains/stresses, reduction in the martensite inducing stress level (i.e. \( \sigma_{AM,s} \)), and thinning of the hysteresis loop are some of the changes, previously reported in the literature [13,14]. Cyclic stress-strain response at different cycles of some of the specimens tested in this study are presented in Fig. 4. As seen in this figure for a tension-compression test (Fig. 4a), with increasing number of cycles, the \( A \rightarrow M \) start stress, \( \sigma_{AM,s} \), decreases slightly and the stress corresponding to the maximum strain (1.5% in this figure) increases to some extent. As a result a clear stress plateau, similar to what was observed in the first cycle, is no longer noticeable after a limited number of cycles. Such changes in behavior are even more pronounced in presence of mean strains/stresses as illustrated in Figures 4b and c. The area encompassed by loading and unloading lines decreases with increasing number of cycles for all the tests until the stress-strain response reaches a stable state. Fig. 4 also indicates that the stress range acting on the material during a strain-controlled cyclic load for all the strain ratios progressively increases from the first cycle of loading up to the stable response of the material. This change in the stress range depicts the cyclic strain hardening in the material. Similar to what stated before for \( \sigma_{AM,s} \) and the stress corresponding to the maximum strain, the amount of change in the stress range is more significant in presence of mean strains/stresses which can be an indicator of a larger cyclic hardening as well as larger mean stress relaxation in the material. Unlike other studies, the amount of residual martensite in the material at zero strain (i.e. residual stress in strain-controlled tests) was not considerable as can be seen in Figures 4a and b. However, for specimens tested at higher strain levels and volume fraction of the stress-induced martensite in the material, Fig. 4c, some residual martensite may have been accumulated, causing a significant increase in the stress range, by reducing the stress level at 2% strain, and mean stress relaxation. Comparing response of the specimen in Fig. 4c with Fig. 2 reveals the specimen (i.e., the one used to generate Fig. 4c) had entered the fully martensitic region. Therefore, it seems that martensitic volume fraction of the material plays a key role in the amount of cyclic hardening, residual martensite and mean stress relaxation in the material.

As mentioned before, some studies have reported the residual martensite and deformation slips as the causes for the residual strain in the superelastic Nitinol under cyclic loads [14]. In strain-controlled test the residual martensite results in residual stress, instead of residual strain. As can be seen in Fig. 4b, the stress corresponding to the starting point of \( A \rightarrow M \) transformation drops in cyclic loading under tensile mean strain. However, there is no residual martensite in the material at the corresponding cycle. This may indicate that either the residual martensite is not causing this drop or it is not responsible for residual stress. Further microstructural investigations are required to determine the changes in the material that are responsible for the drop in \( A \rightarrow M \) start stress under cyclic loads.

### 3.3. Fatigue behavior

Strain-life diagrams for several strain ratios plotted in Fig. 5 illustrate a significant effect of mean strain on fatigue of superelastic Nitinol. The data point shown by an arrow was failed at the grip meaning that the actual fatigue life of the specimen is longer than what is shown on the figure. Strain-life fatigue data from fully reversed fatigue tests indicate a significant increase in the fatigue life for strain amplitudes lower than 1%. This strain amplitude approximately corresponds to the \( A \rightarrow M \) start strain, \( \epsilon_{AM,s} \), implying a significant detrimental effect of stress-induced martensite on the fatigue resistance of superelastic Nitinol. Similar observations on considerable longer lives for strain amplitudes in linear elastic region have been reported in the literature [15,16].
According to Fig. 5, fully reversed strain-life fatigue data for superelastic Nitinol in low-cycle regime ($\varepsilon_a > \varepsilon_{AM,s}$) can be expressed with a straight line in a log-log plot, implying a power law relation between the strain amplitude and the fatigue life, in terms of the number of reversals to failure, $2N_f$. This relation has been referred to as Coffin-Manson-type relation [13] and can be generally expressed as:

$$\overline{\varepsilon}_a = \varepsilon_f' (2N_f)^\beta$$

where $\overline{\varepsilon}_a$ is the equivalent strain amplitude for a fully reversed test that has the same number of cycles to failure as the nonzero mean strain test. $\varepsilon_f'$ is the fatigue strength coefficient and $\beta$ is the fatigue exponent. The fatigue data for nonzero mean strain tests, presented in Fig. 5, show a significant reduction in fatigue life compared to fully reversed experiments with the same strain amplitude. Fig. 5 also indicates a linear relation between the strain amplitude and the fatigue life for all the different $R_\varepsilon$ values within the life regime investigated in this study ($N_f < 10^5$). Further investigations, employing more comprehensive experimental data, is ongoing by the authors to develop a model to formulate the effect of mean strain on the uniaxial fatigue behavior of superelastic Nitinol.

Classical fatigue models for the effects of mean strain such as Goodman [17] or Smith-Watson-Topper [18] may not be directly applicable to Nitinol since the material exhibits a comparatively large plateau region. For strain ranges on the stress plateau the maximum stress remains constant while the strain can oscillate between two values. Cyclic straining of the material in this situation may result in a significant amount of residual martensite that causes the fatigue failure. Therefore, a suitable mean strain/stress correction model should be able to consider the effects of both mean strain and stress. Goodman relation is basically a stress-based model that considers only the effects of mean stress on the fatigue behavior. Smith-Watson-Topper (SWT) parameter, $\varepsilon_a\sigma_{max}$, includes effects of maximum stress and alternating strain on the fatigue resistance. For Nitinol alloys, as mentioned before, the maximum stress remains constant for a large range of strain. Thus, SWT parameter yield similar values for strain-controlled loading with the same strain amplitude, $\varepsilon_a$, and various mean strains, indicating similar fatigue life. However, experimental results from this study show different fatigue lives for specimens with the same strain amplitude, tested at different mean strain levels.

3.3.1. Fractography

Scanning electron microscopy (SEM) was used to investigate the fracture surface and study the crack initiation sites. Fractography under SEM indicates a small crack growth region for most of the specimens. Fatigue cracks appear to initiate mainly from impurities and inclusion particles such as voids and TiC particles as can be seen in
Fig. 6. Size and location of the inclusion particles are important factors that significantly influence the fatigue resistance of Nitinol. Stress concentration around the inclusion particles can induce a large amount of martensite in those areas causing a local residual martensitic region. Accumulation of the residual martensite and deformation slips around the inclusion particles under cyclic forces/deformations may result in crack initiations in those areas. Several microcracks were also observed in the fracture surface of specimens as illustrated in Fig. 6 and Fig. 7. For longer fatigue lives, subsurface inclusions located near the surface of the specimen were found to be the main cause for crack initiation. In addition, surface flaws were also observed to serve as the crack initiation site in some specimens.

4. Summary and Conclusion

Several fully reversed and nonzero mean strain fatigue tests have been conducted on the superelastic Nitinol alloys to investigate cyclic deformation and fatigue behavior of this material. Based on the experimental observations, the following conclusions can be made:
1. Mean strain has significant effects on the strain-controlled fatigue behavior of superelastic Nitinol. Despite the findings from other studies regarding some beneficial effects of tensile mean strain, results from this study indicate a detrimental effect of mean strain on the fatigue behavior of the superelastic Nitinol for the strain amplitudes and mean strains considered in this study. There is a need for more investigations involving various combinations of mean strain and strain amplitude to replicate the beneficial effects of mean strain on fatigue behavior.

2. Classical fatigue models for the effects of mean strain/stress such as Goodman and Smith-Watson-Topper are not directly applicable to the superelastic Nitinol. This is mainly because of the presence of the stress plateau where stress remains constant for a large range of strains.

3. Cyclic strain hardening and mean stress relaxation were observed for superelastic Nitinol specifically for tests with larger maximum strain, $\varepsilon_{\text{max}}$, where stress-induced martensite exists in the larger volume fraction of the material.

4. Volume fraction of the stress-induced martensite was found to be a key parameter affecting the fatigue behavior of the superelastic Nitinol and should be considered in the modeling of mean strain/stress effects.

5. Tension-compression asymmetry may affect the fully reversed strain-controlled fatigue response of the superelastic Nitinol since this asymmetry results in a compressive mean stress for larger strain amplitudes ($\varepsilon_m > \varepsilon_{\text{AM,s}}$).

6. Size, location and distribution of the voids and inclusion particles are important factors that affect the fatigue resistance of the superelastic Nitinol. Inclusion particles and voids have been shown to be the main crack initiation sites in the fatigued specimens.

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