TRANSFORMATION INDUCED CYCLIC BEHAVIOR AND FATIGUE PROPERTIES OF NICKEL RICH NiTi SHAPE MEMORY ALLOY ACTUATORS

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Abstract

In the midst of a new emergence of industrial and commercial applications employing multifunctional materials, the design of active structures using Shape Memory Alloys (SMAs) has become predominant. Boeing has recently been working on a new composition for the binary NiTi, Ni_{60}Ti_{40} (wt.%), for actuation applications. The high nickel content leads to the formation of different intermetallics such as Ti_{3}Ni_{4}, Ti_{2}Ni_{3}, and Ti Ni_{3}, resulting in shape memory effect without any cold work. The nickel-rich SMA is used as a bending actuator in opposition to a chevron structure mounted on the nozzle of a jet-engine. This actuator is also called the Variable Geometry Chevron (VGC), can be used to change deformed shapes with temperature variations. The result is different mixing conditions between the hot stream from the nozzle and the cold air flowing around it. The purpose is to reduce noise and vibrations caused by this turbulent flow during take-off and landing. In order to see such design implemented in commercial airplanes, the SMA actuators need to comply with safety standards, the most important being the fatigue life of the component.

In the present work, the transformation induced fatigue behavior of 60Ni40Ti is being investigated. From the loading conditions in service of the VGC, thermally induced constant stress fatigue testing was conducted. A stress range of 100 MPa – 250 MPa was applied to cover the maximum operational applied stresses. The fatigue life results ranged from about 5,000 cycles at 250 MPa load to about 60,000 cycles at 100 MPa. The thermomechanical transformation cycles generated a large amount of irreversible strain. For this study, two key parameters were selected to be investigated, i.e., heat treatment and specimen thickness, in addition to applied stress. The fatigue test results are presented for different values of these parameters and a post failure analysis to identify the micro-mechanisms leading to fatigue failure are discussed.

Introduction

With the purpose of developing new actuation technologies incorporating active materials, including the design of systems providing significant noise reduction of commercial aircrafts, engineers from the Boeing Company have been investigating the use of Shape Memory Alloys (SMAs) as actuators [1]. The SMA is used as a bending actuator and is clamped to a serrated aerodynamic structure placed on the nozzle of a jet engine. The purpose of such a design is to induce actuation in the SMA components, producing an inward deflection of the variable geometry chevrons (VGC). Such deflection causes the hot gases and cold air coming from the rear end of the jet engine to mix with less turbulence and therefore reducing the noise generated during take-off and landing [2].

Two compositions for the binary shape memory alloy (SMA) NiTi were considered and the selected composition corresponds to Ni_{60}Ti_{40} in weight proportions [3]. The selected material differs from the conventional near equiatomic NiTi. The larger proportion of nickel content results in the formation of a second metastable phase in the nickel-rich matrix. The presence of intermetallics contributes to constraining the martensitic transformation that distorts the lattice and results in an alloy exhibiting shape memory effect without the need of any cold work. The formed precipitates are mostly Ti_{3}Ni_{4}, Ti_{2}Ni_{3} and TiNi [4,5]. The formation of the precipitates is usually controlled during a first homogenization treatment while a second aging treatment is applied to adjust the proportion of nickel exchanged between the nickel-rich matrix and the nickel-rich precipitates. This adjustment treatment is proven to be highly effective in adjusting the transformation temperatures as well as the hysteresis [6]. In addition to controlling the transformation temperatures and the hysteresis, the nickel-rich Ni_{60}Ti_{40} demonstrated excellent actuation response in terms of number of cycles to reach stable actuation response as well as creep like behavior upon variable loading [7]. These results made the nickel-rich Ni_{60}Ti_{40} SMA actuators the primary candidate for future experimental testing and applications.

In previous research efforts, development, characterization and modeling of the material were the main objectives with little effort dedicated to the understanding of the materials fatigue behavior [8]. As the nickel-rich SMAs undergo hot rolling followed by homogenization treatment as well as precipitate formation, the maximum transformation strain that these alloys can generate is up to \sim 1.4\% under monotonic thermomechanical loading on ASTM standard test specimens [7,8]. As the knowledge and technology for SMAs used as actuators continues to mature, the understanding and assessment of the transformation induced fatigue behavior has now become pertinent. The need for fatigue life characterization of nickel-rich SMAs for the Boeing VGC application turn into a priority as this structurally active component is subject to one cycle per flight (current airliners usually undergo approximately 20,000 to 70,000 flight cycles). Therefore, the designed VGC can be expected to undergo at least 20,000 to 70,000 cycles during its service life. In order to be integrated into an aerospace structure in the near future, SMA actuators must demonstrate sufficient fatigue life to complete their service life. They also need to account for some safety margin in terms of applied loads and extended number of cycles to failure. Most work done on the

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fatigue properties of SMAs has been carried out on the cyclic pseudoelastic response. It was first performed for small fractions of the maximum transformation strain that can be generated in near equiatomic NiTi SMAs and the number of cycles to failure on the order of hundreds of thousands of cycles was attained [9,10,11]. More recently reported, the biomedical industry has been focusing on the influence of high amplitude cyclic straining of pseudoelastic SMAs with fatigue limits below $10^4$ cycles [12,13]. Under such loading conditions, the classical field of fatigue of metals defines this fatigue behavior as low-cycle fatigue due to a predominant accumulation of irrecoverable deformation. This is due to its very different regime and failure mechanism from the high-cycle fatigue behavior where none or almost no plastic deformation is created [14]. Ni$_{50}$Ti$_{50}$ SMA actuators are no different in terms of high amplitude straining due to full martensitic detwinning induced upon thermal cyclic loading. Large amounts of plastic strain have been reported to be stabilized upon application of the appropriate thermomechanical training [8]. Therefore, it is important to acknowledge the fact that such SMA actuators will develop large amounts of plastic strain in counterpart of being highly competitive in terms of large energy density actuators. Nickel-rich NiTi SMA actuators have not been extensively characterized under cyclic loading taken to failure. This fatigue life characterization becomes a significant challenge to the integration of such promising materials in the first major large scale SMA application. The present work is an effort to investigate the thermomechanical cyclic response and fatigue behavior of nickel-rich NiTi SMA actuators. The purpose is to first estimate the fatigue limits of such alloys under various loading conditions and to understand the failure mechanisms involved in the degradation of the material. In the next section, the experimental setup is introduced with the methodology and the different parameters applied to characterize the fatigue response of the SMA actuators. Following is a description of the fatigue results and a discussion on the modifications undergone by the SMA material upon cyclic loading up to failure in terms of fractography in correlation with the macroscopic fatigue response. The conclusion discusses the first series of observations and concludes with a selected set of test parameters to further investigate this unique material.

Experimental setup and test matrix

The main goal of this study is to test the ability of nickel-rich SMA actuators to sustain a sufficient amount of thermomechanical fatigue cycles within a safety margin prior to certification. Nickel-rich SMA samples were prepared by Boeing engineers as flat dogbone specimens and are used to perform isobaric thermal cycles. The fatigue experimental setup in the Active Materials Laboratory at Texas A&M University allows testing of very small specimens. Therefore, three different narrow cross sections were selected to determine what effects, if any, the size and geometric configuration of the specimens have on their fatigue life. A stress range was selected, which included the working stresses of proposed applications plus some margin of safety. Two different heat treatments were selected, with an additional parameter being the heat treatment environment (vacuum or air).

Experimental setup

The identification of the loading conditions of the SMA components led to the definition of a series of uniaxial isobaric thermally induced fatigue tests (see Fig. 2). The following schematic represents the stress-temperature phase diagram of NiTi SMAs. A typical isobaric loading path is shown with a dashed double-sided arrow and is introduced later as a test parameter. The fatigue test frame consists of a plexiglass bath containing a closed-loop circulating coolant which allows forced fluid convective cooling onto specimens submerged in the bath. The coolant is a waterless solution of ethylene glycol. Heating is achieved through resistive Joule heating using a DC power supply connected to the two ends of the specimen. The specimens are mounted on one end to a fixed point on a rigid aluminum frame while the other end is connected to masses hanging vertically through a pulley system. Displacement measurement is achieved using linear variable displacement transducers (LVDTs) attached to the rigid frame and connected to the free-end of the specimens (see Fig. 1). The displacements of the SMA actuators are recorded through LVDT transducers and the strains in the austenitic and martensitic states are used to define total, plastic and recoverable strains. Thermal loading cycles are achieved at a frequency ~ 0.1 Hz with approximately 2 seconds of heating and 8 seconds of cooling. The advantage with such a design is the capacity to produce thermomechanical fatigue data between 48 hours and one week, on average.

Investigated parameters
The present research effort underlies the analysis of the influence of different parameters on the fatigue response of nickel-rich SMA actuators. The application of thermomechanical cycles is conducted until failure of the SMA specimens is achieved. Therefore, five different parameters were selected to be scrutinized in terms of influence on the stress life response as well as on the level of accumulated plastic strain at failure.

The selected parameters are the constant applied stress, the heat treatment atmosphere, the aging time during heat treatment, the specimen geometry (i.e. specimen thickness) and the alloy composition.

The constant applied stress ranges from 100 MPa to 250 MPa in 50 MPa increments. The heat treatments were performed in two different atmospheres, vacuum or air. The two selected aging times are one hour at 450°C (water quenched) and 20 hours at 450°C (water quenched), after homogenization treatment of one hour at 850°C (furnace cooled). The specimen thickness is a parameter that allows for observation of size effect. The three selected thicknesses were 0.005, 0.01 and 0.015 inches (0.127, 0.254, and 0.381 mm respectively). Finally, two SMAs with two different nickel-rich compositions were chosen to investigate the influence of a small change of nickel content on the fatigue response of the actuators. The corresponding compositions are Ni_{60}Ti_{40} and Ni_{57}Ti_{43}, in weight proportions.

The purpose of this comparative study is to identify the most influential parameters on the fatigue life of Ni rich SMA actuators in order to eliminate any external limiting factors to the intrinsic fatigue response of the SMA material.

### Specimen geometry and test matrix

Due to the geometric constrains of the fatigue test frame and to the targeted high actuation frequency during thermal cycling, small specimens are needed. A flat dogbone geometry was selected, as shown in Fig. 3. The chronology of the parametric study was such that the first investigated alloy composition (Ni_{60}Ti_{40}) was investigated in terms of all other parameters: four applied stress levels, two heat treatment environments and two different aging times with only one aging time in the case of air heat treatment. The resulting test matrix can be seen in Table 1. The second alloy composition is Ni_{55}Ti_{45} and was selected to provide a better understanding and comparative elements in terms of fatigue response. For this alloy composition, two stress levels, one environment, one aging time and one thickness were selected. The corresponding test matrix is represented in Table 2.

### Table 1. Test matrix for Ni60Ti40 SMA composition.

<table>
<thead>
<tr>
<th>Heat Treatment Environment</th>
<th>Aging Time</th>
<th>Specimen Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vacuum Heat Treatment</td>
<td>V-HT 1 - 1 hr 450°C</td>
<td>5 mils (0.127 mm)</td>
</tr>
<tr>
<td></td>
<td>V-HT 2 - 20 hrs 450°C</td>
<td>5 mils (0.127 mm)</td>
</tr>
<tr>
<td>Air Heat Treatment</td>
<td>A-HT 2 - 20 hrs 450°C</td>
<td>5 mils (0.127 mm)</td>
</tr>
</tbody>
</table>

| Applied Stress Level: 100 MPa – 150 MPa – 200 MPa – 250 MPa |

### Table 2. Test matrix for Ni57Ti43 SMA composition.

<table>
<thead>
<tr>
<th>Heat Treatment Environment</th>
<th>Aging Time</th>
<th>Specimen Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air Heat Treatment</td>
<td>A-HT 2 - 20 hrs 450°C</td>
<td>15 mils (0.381 mm)</td>
</tr>
</tbody>
</table>

| Applied Stress Level: 100 MPa – 250 MPa |

### Fatigue tests results

The various investigated parameters are analyzed using different criteria. The first set of results identifies the number of cycles to failure of Ni rich SMA actuators as a function of the applied stress and the second of the accumulated plastic strain at failure. Such results provide a macroscopic understanding of the fatigue behavior. Therefore, fractography is needed to assess the microstructural evolution due to cyclic loading and the post-mortem SMA actuators are investigated using optical and electronic microscopy. An example of post-mortem actuators is given in Fig. 4 (a) with the identification of the fracture surface (Fig. 4 (b)).

### Influence of heat treatment time

One particular property of the nickel-rich Ni_{60}Ti_{40} SMA is that it can exhibit SME without any cold work process. The presence of precipitates favors the storing of elastic energy and therefore the realization of recoverable strains upon martensitic phase transformation. The proportions, the distribution and the composition of the precipitates can be modified/adjusted by...
applying appropriate heat treatments. The heat treatments consist of a homogenization treatment of one hour at 850°C with furnace controlled cooling to allow formation of precipitates followed by an annealing treatment of either 1 or 20 hours at 450°C.

**Stress – life response.** In this first series of tests, the purpose was to determine if a different aging time had any major influence on the stress life response of the nickel-rich SMA actuators. Fatigue limits of ~ 70,000 cycles were identified for stress levels of ~100 MPa and were found to reduce to ~ 4,000 cycles under ~250 MPa applied stress. However, the main result from these experiments is that V-HT1 and V-HT2 gave the same fatigue life in the stress life space. An additional important result is that no size effect was observed, for both V-HT1 and V-HT2.

**Plastic strain accumulation.** The results shown in Fig. 5 did not demonstrate any differences between V-HT1 and V-HT2.

**Post-mortem analysis.** The different aging times not only resulted in different macroscopic behaviors (see Figs. 5 – 7), but also in different microscopic damage. Figures 8 and 9 show SEM fractography of two specimens that failed at ~4,000 cycles under 250 MPa constant stress. Figure 8 represents a specimen, 15 mils thick (0.381 mm) and aged for 1 hour. Large smooth areas can be seen on this fracture surface, indicative of slow fatigue crack propagation followed by a major tear-up area showing the location of the final failure. From the magnified micrographs in Figs. 8 (b) and 8 (c) sharp and straight microcracks, most likely transgranular cracks, which developed through the microstructure, indicate a brittle final failure. However, in Fig. 9, the specimen is 10 mils (0.254 mm) thick, aged for 20 hours and displays small smooth areas where fatigue cracks were initiating and slowly progressing. Figures 9 (b) and 9 (c) show a distinctive pattern with plastic deformation and local tear-up organized in a parallel manner. This process appears to have been biased by the nickel-rich precipitates, which seem to have a stronger influence on the microstructure of Ni₆₀Ti₄₀ when the aging time was 20 hours.

**Figure 5.** Applied stress level vs. number of cycles to failure for Ni₆₀Ti₄₀; comparison between V-HT1 and V-HT2.

**Figure 6.** Accumulated plastic strain vs. applied stress level for Ni₆₀Ti₄₀; comparison between V-HT1 and V-HT2.

**Figure 7.** Accumulated plastic strain vs. number of cycles to failure for Ni₆₀Ti₄₀; comparison between V-HT1 and V-HT2.

Figure 4. (a) Picture of four different fatigue specimens showing random localization of the fatigue failure occurring within the targeted test gauge, (b) schematic showing the area of interest in terms of fractography.
Influence of heat treatment atmosphere

For the next step of this parametric study, vacuum heat treatment 2 (V-HT2) is compared to air heat treatment 2 (A-HT2) for Ni_{60}Ti_{40}. Again all specimen sizes are studied in terms of applied stress versus the number of cycles to failure as well as the accumulated plastic strain versus applied stress and versus the number of cycles to failure.

The result is the observation of a major influence on the fatigue life of thin specimens while larger specimens don’t seem to be so affected. Also, the existence of a consequential oxide layer formed upon heat treatments performed in air is observed and will be discussed later in this work.

Stress – life response. For the first series of specimens heat-treated in high vacuum, the S-N curves didn’t show much difference between in the influence of the different thicknesses on the two different heat treatments. However, the presence of an oxide layer caused the thin specimens (0.005 in., 0.127 mm) to fail prematurely while the thick ones sustained similar number of cycles to failure as the ones heat-treated in high vacuum, as seen in Fig. 11. Due to the presence of a significant
oxide layer, stress correction is necessary to evaluate the actual stress level under which the specimens failed. The results from Fig. 11 take this correction into account; for an oxide layer measured to be 0.001 inch (0.0127 mm) thick. Noticeable fatigue life reduction with thin oxidized specimens failing no more than after 2,000 cycles for applied stresses as low as 100 MPa while thick specimens failed around 15,000 cycles, under an applied stress equal to 150 MPa.

**Plastic strain accumulation.** Figures 12 and 13 compare the previous results for V-HT2 to the ones obtained for A-HT2. The results from Fig. 12 compare the amount of accumulated plastic strain in terms of the applied stress for A-HT2 and for V-HT2 while Fig. 13 shows a similar trend of the accumulated plastic strain attained at failure under both heat treatment environments. The only difference was found to be a larger amount of plastic strain for the tests performed on air heat treated specimens.

**Post-mortem analysis.** The comparison between V-HT2 and A-HT2 is based on optical micrographs and reveals the presence of a significant oxide layer that was formed during the homogenization heat treatment of 850°C for 1 hour, when the atmosphere was selected to be air. The first major result that was found was the influence of air heat treatment on thin specimens. In fact, air heat-treated thin specimens failed before they reached 2,000 cycles for a constant applied stress of 150 MPa compared to nearly 50,000 cycles for heat treatment in vacuum. Figures 14 and 16 show a specimen vacuum heat-treated with no presence of an oxide layer. The microstructure reveals a strong width to thickness aspect ratio with predominant transverse fatigue striations. Figures 15 and 17 point out the width to thickness aspect ratio dependency on the fatigue damage and failure. They also show a consequential oxide layer measured to be ~ 1 mil thick (25.4 µm).

Figures 16 and 17 compare the influence of air versus vacuum heat treatment on the failure of thick specimens. Figures 16 (a) and 16 (b) show the influence of a thicker specimen on the random orientation of the propagating fatigue cracks, and the final tear-up morphology, respectively. Both micrographs are indicative of a bulk behavior. Figure 17 (a) shows a fracture surface with similar oxide layer nearly ~ 1 mil thick, but it is in much smaller proportions on a thick specimen compared to a thin one. The proportion of oxide layer over the specimen thickness at the cross-section is 2/5 on a thin specimen while it becomes 2/15 on a thick specimen. This observation explains the similar fatigue data in the stress life space between thick air heat-treated and thick vacuum heat-treated specimens (see Fig.11). Figure 17 (b) is the characterization of the incompatibility of a brittle oxide layer coating a shape memory alloy and its contribution to surface crack initiation.

![Figure 14: Fatigue striations across the thickness of a thin specimen with transverse cracks indicate a strong dependency on the width to thickness ratio.](image1)

![Figure 15: Identification of transverse cracks surrounded by significant oxide layer with oxide layer measured ~ 0.001 in (25.4µm) formed upon heat treatment in air.](image2)
Figure 16. (a) Thick specimen displaying bulk behavior with randomly oriented fatigue striations, (b) final tear-up – fracture area.

Figure 17. (a) Thick specimen and oxide layer thickness ~ 0.001 in (25.4 µm), (b) presence of a microcrack at the interface oxide/SMA.

Influence of Nickel content

The third series of results is a comparison between two Ni rich SMA alloys: Ni₆₀Ti₄₀ and Ni₅₇Ti₄₃ (wt %). The selected heat treatment was A-HT2.

Stress – life response. The response of the two different alloys in the S – N space from Fig. 18 show a better fatigue limit at high stress levels with N_f ~ 4,000 cycles for Ni₆₀Ti₄₃ while for Ni₆₀Ti₄₀ N_f ~ 2,000 cycles. However, at lower stresses near ~ 150 MPa, the fatigue limit of Ni₅₇Ti₄₃ drops and Ni₆₀Ti₄₀ sustains twice as much life cycles with ~ 15,000 cycles.

Plastic strain accumulation. Similar behavior is observed in terms of accumulation of plastic strain. Both alloys demonstrate stress dependency as seen in Fig. 19 and Fig. 20. Figure 20 also shows that Ni₅₇Ti₄₃ behaves more like Ni₆₀Ti₄₀ with V-HT1, as seen earlier.

Figure 18. Applied stress level vs. number of cycles to failure, comparison between Ni₆₀Ti₄₀ and Ni₅₇Ti₄₃.

Figure 19. Accumulated plastic strain vs. applied stress level, thick specimens, A-HT2: comparison between Ni₆₀Ti₄₀ and Ni₅₇Ti₄₃.

Figure 20. Accumulated plastic strain vs. number of cycles to failure, thick specimens, A-HT2: comparison between Ni₆₀Ti₄₀ and Ni₅₇Ti₄₃.

Conclusions

The purpose of this work was to characterize the fatigue behavior and cyclic response of nickel-rich NiTi shape memory alloy actuators. In the context of a series of scoping experiments, a limited number of test data per condition were selected to investigate a wide range of various parameters in a rather short
amount of time. The understanding of the thermomechanical cyclic and fatigue behavior of the investigated shape memory alloys was based on both the macroscopic response and the post-mortem microstructure of the SMA actuators.

A series of various process and test parameters were selected and their influence was investigated. Parameters such as the applied stress level, the heat treatment time, the heat treatment atmosphere and the variation of nickel content demonstrated a strong influence on the fatigue life of the SMA actuators. With the purpose to select an optimized set of parameters, the best choice for an aerospace application such as the Boeing VGC appears to be Ni60Ti40, heat treated in high vacuum and with an aging treatment time of 20 hours at 450°C. Some of the reasons include a fatigue life ranging between 50,000 to 70,000 cycles at full actuation under a constant stress of 100 MPa, a lower amount of accumulated plastic strain at failure with very little to no applied stress dependency. It was also found that nickel-rich NiTi SMA actuators are very sensitive to the presence of an oxide layer, mostly due to the relatively small geometry of the fatigue specimens. Another interesting result from the post-mortem analysis is a ductile fracture behavior. The microstructure showed the existence of ductile crack formation allowing the material to slowly fail and delay the final failure. Such characteristic behavior can be of high interest in the purpose of monitoring the formation of crack and preventing a catastrophic failure in an aerospace component.

Among top priorities to continue characterizing the fatigue life of nickel-rich SMA actuators, surface effects due the presence of a consequential recast layer are to be investigated. Another important parameter to be investigated is the actuation strain. The fatigue results presented in this work were exclusively performed under full actuation (i.e. cyclic transformation of 100% martensite to 100% austenite). By reducing the amount of actuation strain and still maintaining excellent actuation performances, a fatigue life increase is expected [15].

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