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Fabrication of a novel laser-processed NiTi shape memory microgripper with enhanced thermomechanical functionality

Matthew Daly¹, Andrew Pequegnat¹, Yunhong N Zhou¹ and Mohammad I Khan²

Abstract
The thermomechanical properties of nickel-titanium shape memory alloys have sparked significant research efforts seeking to exploit their exotic capabilities. Until recently, the performance capabilities of nickel-titanium devices have been inhibited by the retention of only one thermomechanical response. In this article, the application of a novel laser-processing technique is demonstrated to create a monolithic self-positioning nickel-titanium shape memory microgripper. Device actuation and gripping maneuvers were achieved by thermally activating processed material regions which possessed unique phase transformation onset temperatures and thermomechanical recovery characteristics. The existence of each thermomechanical material domain was confirmed through differential scanning calorimetry analysis. Independent thermomechanical recoveries of each embedded shape memory were captured using tensile testing methods. Deployment of each embedded shape memory was achieved using resistive heating, and in situ resistivity measurements were used to monitor progressive phase transformations.

Keywords
nickel-titanium, laser processing, microgripper, thermomechanical behavior, shape memory effect, shape memory alloys, smart materials

Introduction
With the surging demand for microsystems technology in the aerospace and biomedical industries, a significant need has developed for elegant smart material mechanisms to replace conventional electromechanical systems. Nickel-titanium (NiTi) shape memory alloys (SMAs) are considered a top candidate for a variety of next-generation smart material applications because of their excellent combination of mechanical properties and biocompatibility (Duerig et al., 1999; Fu et al., 2001; Hartl and Lagoudas, 2007). While NiTi has enjoyed relative success in many Microsystems devices, future technologies demand more precise control over its functional thermomechanical properties, namely the shape memory effect.

As in other SMA systems, the mechanism responsible for shape memory behavior in the NiTi system is a reversible thermoelastic austenite–martensite phase transformation. Given that the phase transformation onset temperatures in NiTi alloys are extremely sensitive to alloy composition and processing history (Miller and Lagoudas, 2001; Tang, 1997), efforts to create application-specific NiTi alloys have proven to be problematic. Furthermore, the functionality of conventional NiTi alloys is limited by the retention of only one shape memory response. A novel material processing protocol is therefore required to overcome the performance limitations of traditional NiTi fabrication technologies.

Recent investigations have shown that exposure to high-density laser energy can alter thermomechanical...
behavior in NiTi by shifting phase transformation onset temperatures within the processing region (Daly et al., 2011, 2012; Khan and Zhou, 2010a; Pequegnat et al., 2011). This technique, referred to as multiple memory material (MMM) technology, can be used to locally tune the thermomechanical response of conventional NiTi SMAs (Khan and Zhou, 2010b, 2011). Laser processing with MMM technology therefore permits the localized embedment of unique shape memory responses within a monolithic NiTi structure; thus, enhancing the functionality of NiTi by augmenting its thermomechanical characteristics. The purpose of this study is to implement laser processing to fabricate a novel NiTi-based microsystems device with enhanced thermomechanical functionality.

Given the complicated path trajectories demanded from smart material microsystems technologies, monolithic fabrication of NiTi devices is usually not practical and SMA components are often integrated into hybrid packages (Kohl, 2004). For example, several hybrid designs exist for NiTi microgripper heads. Lee et al. (1996) have developed a passively biased SMA microgripper by sputtering a NiTi film onto a silicon substrate and Kohl et al. (2000) created an integrated antagonistic SMA microgripper through laser micro-machining of a NiTi thin sheet. Although each of these designs achieves gripper actuation through exploitation of the shape memory effect, device performance is inherently limited by their reliance on external systems to position the microgripper head during gripping operations. In order to eliminate the need for external positioning systems, presented in this study is a monolithic laser-processed NiTi microgripper capable of self-positioning. Actuation of positioning segments and the device gripper head is achieved through the sequential activation of unique thermomechanical regimes. The added thermomechanical functionality of the laser-processed NiTi eliminates the need for external positioning systems and greatly reduces the complexity of the NiTi microsystems device. To the author’s knowledge, this study represents the first example of a monolithic NiTi microgripper that possesses multiple recoverable thermomechanical characteristics.

Experimental

Commercially available 410 μm diameter NiTi wire with a nominal composition of 50.8 at.% Ni and 49.2 at.% Ti was used for microgripper construction. Prior to laser processing, the surface oxides were removed using a hydrofluoric–nitric acid etching agent; uniformly reducing the diameter of the wire to 380 μm.

Laser processing was achieved using a Miyachi Unitek pulsed neodymium-doped yttrium aluminum garnet (Nd:YAG) laser system (Model LW50 A) which produced a 1.06 μm wavelength beam and possessed a nominal postoptic spot size of 600 μm. In order to process larger lengths of NiTi wire, a custom fixture was designed to advance the wire between laser pulses. Processing parameters were selected to locally embed three unique thermomechanical responses within the as-received NiTi alloy, as per the previous study (Khan, 2010). In each laser-processing schedule, full melting of the base metal was achieved. The two lower temperature thermomechanical material domains were designed to actuate the positioning segments of the microgripper and the higher temperature shape memory was used to close the microgripper head. In order to avoid contamination during laser-processing operations, argon shielding was provided to the processing region at a flow rate of 0.42 m³/h (15 ft³/h). After laser-processing operations, the wire was resistively shape trained using a Sorensen XG series 33-25 programmable direct current (DC) power supply. Figure 1 illustrates the processing dimensions of the NiTi wire and microgripper shape set geometry.

In order to confirm the embedment of independent thermomechanical material domains within the laser-processed NiTi microgripper, differential scanning calorimetry (DSC) analysis was used to capture changes in phase transformation onset temperatures in the NiTi samples. DSC scans were performed using a Thermal Analysis Q2000 system equipped with a refrigerated cooling system. As-received and laser-processed samples were prepared for comparison. Given the high power densities associated with laser processing, a heat-treated wire specimen was also analyzed to ensure that changes in phase transformation onsets were not the result of localized annealing of the as-received material. Sample data was collected using a modified ASTM
F2004-05 testing protocol. Heat flow was measured at a controlled heating and cooling rate of 5°C/min, and austenite and martensite start and finish temperatures (As, Af, Ms, and Mf, respectively) were defined following the ASTM standard.

Thermomechanical recovery of the laser-processed NiTi samples was assessed using an Instron model 5548 microtensile machine with a ±0.5 μm measurement accuracy. Tensile samples were loaded at room temperature (20°C) under an ASTM F2516-07 testing protocol at a rate of 1.0 mm/min to 6% strain and then subsequently relaxed. Upon complete unloading, laser-processed samples were heated in situ to above Af in an environmental chamber in order to activate each embedded thermomechanical response. A zero load condition was maintained at the crossheads and displacement feedback was continuously recorded in order to assess material recovery. The measured displacement was then normalized against the final length of the NiTi wire and then correlated with collected temperature data to track shape memory recovery. Temperature measurements were recorded using a thin film resistance temperature detector (RTD) with an uncertainty of ±1.3°C.

Actuation of the laser-processed NiTi microgripper was achieved by resistive heating methods that were controlled using a National Instruments PXI-1031 data acquisition module equipped with a RTD. In order to assess phase transformations in situ, online resistivity measurements were captured by monitoring applied voltage and current loads during microgripper actuation. In order to eliminate high-frequency signal noise from alternating current (AC) line voltages, the collected thermomechanical and resistivity data were filtered using a low-pass Butterworth algorithm (Bianchi and Sorrentino, 2007).

Results and discussion
Detection of embedded thermomechanical behavior

The collected DSC scans and corresponding phase transformation onset temperatures are shown in Figure 2 and Table 1, respectively. Examination of the as-received sample showed a single diffuse subambient phase transformation. The broad transformation range and low hysteresis were typical of trained NiTi and are commonplace in commercially available material (Tam, 2010). As shown in Figure 2(b), the effects of heat treatment on the as-received material have removed prior material training and re-established a thermal response consistent with solutionized NiTi (Grossmann et al., 2008). DSC scans of the microgripper components (Figure 2(c) to (e)) revealed subambient heat flow peaks which were attributed to transformations from retained base metal. As shown in Figure 3(a), a portion of the NiTi sample was not melted during laser pulsing operations due to the conical geometry of the processing volume. Optical microscopy of an isolated laser pulse revealed a recrystallized zone approximately 100–200 μm in width (Figure 3(b)). A thermal response typical of annealed NiTi was therefore not unexpected in the laser-processed samples. The slight distortions detected in the base metal phase transformation dynamics were likely due to the relatively small volume fraction of retained base metal in the DSC sample.

Table 1. Phase transformation temperatures of the NiTi base material and laser-processed microgripper components (°C)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ms</th>
<th>Mf</th>
<th>As</th>
<th>Af</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-received</td>
<td>19</td>
<td>−29</td>
<td>−26</td>
<td>20</td>
</tr>
<tr>
<td>Heat treated</td>
<td>−20</td>
<td>−30</td>
<td>−9</td>
<td>3</td>
</tr>
<tr>
<td>First segment</td>
<td>20</td>
<td>−6</td>
<td>21</td>
<td>45</td>
</tr>
<tr>
<td>Second segment</td>
<td>52</td>
<td>15</td>
<td>48</td>
<td>89</td>
</tr>
<tr>
<td>Gripper head</td>
<td>63</td>
<td>33</td>
<td>71</td>
<td>95</td>
</tr>
</tbody>
</table>

Figure 2. DSC scans of the (a) as-received NiTi, (b) NiTi heat treated at 800°C for 300 s and components of the laser-processed NiTi microgripper: (c) first segment; (d) second segment; and (e) microgripper head.
In addition to transformation peaks from retained base metal, results from DSC testing captured three additional thermal responses which were absent in the as-received and heat-treated NiTi samples. The effects of laser processing, therefore, embedded three unique thermal behaviors within the single NiTi wire—allowing for independent shape memories to be recovered upon heating samples above their respective $A_f$ temperatures. A slight recovery overlap existed, however, between the two higher temperature laser-processed NiTi samples, which requires further characterization to determine the impact on microgripper performance.

**Thermomechanical response of the laser-processed microgripper**

Results from tensile testing of individual NiTi microgripper components indicated that in addition to unique thermal behavior, the laser embedded material domains possessed different mechanical properties. As shown in Figure 4(a) to (c), each of the three active microgripper components exhibited unique stiffening characteristics. As the most thermally stable martensite at room temperature, the microgripper head exhibited the largest resistance to detwinning, which was consistent with the trends reported by Miyazaki et al. (1981) for solutionized NiTi.

In order to evaluate the combined mechanical responses of microgripper components, a NiTi sample processed to the microgripper dimensions was prepared. As shown in Figure 4(d), the microgripper underwent a multistage detwinning when loaded. At stresses below $\sigma_{Dt1}$, the mechanical response was linear elastic. In the load range of $\sigma_{Dt1} < \sigma < \sigma_{Dt2}$, a detwinning of the first microgripper positioning segment occurred. Subsequent detwinning of each of the laser-processed microgripper components occurred, which was evident from changing inflections along the microgripper tensile response curve.

Identified by its characteristic serrated Lüders-like mechanical deformation (Sittner et al., 2005), a pseudoelastic response from the retained base metal was visible in each of the stress–strain curves presented in Figure 4(a) to (d). In comparison to the pseudoelastic behavior of virgin base metal (Figure 4(e)), the pseudoelastic plateau stress for each of the laser-processed samples was approximately 100 MPa lower. This reduction in stress was attributed to recrystallization of the base metal during laser processing (Figure 3(b)) and has been observed in a previous study by Khan et al. (2008). Although a defined return plateau was not visible in the laser-processed samples, according to a study completed by Liu and Galvin (1997), martensite variant accommodation can stabilize the stress-induced phase
above the $A_f$ temperature. Therefore, the retention of stress-induced martensite upon unloading of laser-processed NiTi samples was not unexpected. Further investigation is required, however, to better understand the effects of laser processing on microstructure and corresponding thermomechanical response in retained base metal.

Figure 5(a) provides the captured data for the thermomechanical recoveries of the laser-processed tensile specimens shown in Figure 4. As predicted by DSC results, each microgripper component exhibited an independent recovery at different temperatures. While there was a slight overlap between recoveries of the second and third thermomechanical responses, the lower temperature material domain (second microgripper segment) recovered by approximately 90% before activation of the microgripper head. Temperatures corresponding with the onset of thermomechanical recovery ($A_s$) correlated very well with the phase transformation onset temperatures determined from DSC testing. The $A_f$ temperatures, however, were slightly higher than anticipated. This discrepancy can be explained by thermal sinking to the tensile grips. A complete recovery was therefore slightly hindered in portions of the laser-processed samples that were in close proximity to the gripping point.

As shown in Figure 5(b), the recovery of the laser-processed NiTi microgripper occurred in three stages. Upon heating to temperatures above $A_{s1}$, the specimen underwent recovery of the first laser-processed positioning segment. Further heating sequentially activated the remaining embedded thermomechanical behaviors. Actuation of the microgripper head was identified from the data set by the change in inflection of the microgripper’s thermomechanical response and comparison to the individual responses provided in Figure 5(a). In contrast to its individual response, recovery in the laser-processed microgripper specimen correlated well with DSC results, which was attributed to the processing dimensions of the tensile specimen (Figure 1(a)). Since the microgripper head segment was not in direct contact with the tensile grips during thermomechanical characterization, thermal sinking did not occur.

It was initially anticipated that the microgripper head would account for 50% of the overall thermomechanical recovery because of its proportionally larger processing dimensions. Since the amount of detwinned martensite in NiTi determines the magnitude of shape memory recovery (Miyazaki et al., 1984), and considering the different stiffnesses of laser-processed microgripper components, a lessened response from the microgripper head was understandable. In contrast, the thermomechanical response of the first microgripper segment represented approximately 45% of the overall microgripper recovery, despite being only 25% of its active length. Future studies are planned to further investigate the complex mechanical relationships between laser-processed material domains.

**Deployment of the laser-processed microgripper**

Using the DSC and thermomechanical recovery results as a guideline, a heating profile was designed to sequentially activate each of the three processed microgripper components. Figure 6 provides photographs of each of the four positions achieved by heating the laser-processed microgripper along with the measured wire temperature and electrical resistivity profiles. As anticipated, recorded temperatures did not explicitly match with the DSC data because of external loads imposed on the device from fixturing and also from the heat-sinking effects of the temperature sensor. In order to confirm sequential phase transformations in the laser-processed NiTi microgripper, optical observations were correlated with in situ resistivity measurements. According to the trends reported by Kakeshita et al. (1998), sudden increases in the bulk resistance of solution-treated NiTi are indicative of a martensite to austenite phase change. While it was expected that the NiTi material would show some increase in resistivity due to heating, the increase in the magnitude of resistance in the microgripper could not be explained by joule heating alone. The captured resistivity

![Figure 5](https://example.com/figure5.png)

**Figure 5.** (a) Thermomechanical recovery of each microgripper component and (b) the combined recovery of the laser-processed NiTi microgripper.
measurements and photographs therefore confirmed the localized phase changes and sequential activation of embedded thermomechanical domains in the laser-processed microgripper.

While this study has demonstrated the enhanced capabilities of a simple NiTi microgripper, it is anticipated that laser processing can be used to embed added functionality in the more elaborate microgripper designs currently available (Kohl et al., 2000; Lee et al., 1996). Furthermore, it is expected that the pseudoelastic response of the retained base metal can be exploited to deliver a reversible thermomechanical recovery. A detailed investigation of the energy storage capabilities of retained pseudoelasticity is therefore planned for future research.

**Conclusion**

In this study, a novel NiTi microgripper capable of self-positioning was fabricated through the application of laser processing with MMM technology. The effects
of laser processing removed the prior training of the as-received alloy and considerably altered its thermomechanical characteristics. Results from DSC testing showed three independent material domains with unique austenite onset temperatures of 21°C, 48°C, and 71°C, respectively. The three material domains were locally embedded as the active components of a NiTi microgripper. The two lower temperature domains were utilized as the self-positioning microgripper segments, while the higher temperature domain actuated the microgripper head. Mechanical testing revealed three separate thermomechanical behaviors in the laser-processed microgripper which corresponded to the independent shape memory recoveries of each embedded material characteristic. Resistive heating of the NiTi microgripper permitted a visual confirmation of the sequential thermal activation of each laser-processed microgripper component.

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