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EVALUATION OF CYCLIC DEFORMATION BEHAVIOR OF LASER-WELDED SHAPE MEMORY NITI ALLOYS AT DIFFERENT WORKING TEMPERATURES

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Abstract

Post-weld heat-treatment (PWHT) has been established as one of the cost-effective ways to improve the functional properties, namely shape memory and super-elastic effects (SME and SE), of laser-welded NiTi alloys. However, the functional performance of the laser-welded joint at different working temperatures has not been explored yet. The purpose of this study is to investigate the effect of different working temperatures on the functional properties of the laser-welded NiTi alloys before and after PWHT by applying cyclic deformation tests. Two laser-welded samples: as-welded and heat-treated sample (after PWHT at 350 °C or 623 K) were tested in this work at room temperature, 50 °C (or 323 K) and 75 °C (or 348 K) respectively. The samples were cyclically loaded and unloaded for 10 cycles up to 4 % strain. The critical stress to induce the martensitic transformation and the residual strain after the cyclic tests were recorded. The results indicate that the heat-treated sample exhibited better functional properties than the as-welded sample at room temperature and 50 °C (or 323 K). However, both the as-welded and heat-treated samples failed in the cyclic tests at 75 °C (or 348 K). These findings are important to determine the feasible working temperature range for the laser-welded NiTi components to exhibit desirable functional properties in engineering applications involving cyclic loading.

Introduction

Since the discovery of the shape memory and super-elastic effects (SME and SE), the near-equiatomic Ni-rich NiTi alloys have been extensively used in various engineering domains, i.e. from implantable biomedical devices and actuators to aerospace applications [1-3]. SME and SE are direct consequences of the reversible martensitic transformation between austenite (high-temperature phase) and martensite (low-temperature phase). SME for which it is named refers to the ability of martensitic NiTi to recover to their original shape in the austenitic state upon heating. SE allows NiTi alloys to sustain up to 8 % strain without permanent deformation. Although NiTi alloys possess these unique functional properties, namely SME and SE, which mark them out from the conventional engineering materials, the difficulty in fabrication, particularly joining with itself and other materials, greatly impedes the design of more complex and efficient engineering products involving NiTi alloys as the core component.

Laser welding is one of the most guaranteed joining processes for NiTi alloys because of its high precision and small heat input, which minimizes the effect of heat distortion and the size of weld zone (WZ) and heat-affected zone (HAZ) [4-7]. However, it is well-documented that the functional properties of the laser-welded NiTi components are degraded to a certain extent due to the microstructural disparity between the WZ, HAZ, and base metal (BM). Certain post-processing treatments are still required to recover the integrity of the laser weld-joint to retain desirable functional properties.

Heat-treatment is commonly used to optimize the functional properties of NiTi alloys by precipitation effects. The precipitates can be appearing in the sequence of Ni₃Ti₃, Ni₃Ti and Ni₅Ti between the heat-treatment temperatures from 350 °C to 800 °C (623 K to 1073 K) [8]. It has been widely recognized that intermediate treatment temperatures, namely 350 °C to 450 °C (623 K to 723 K), have a profound impact on the functional properties of NiTi alloys because of the precipitation of coherent and small-sized Ni₃Ti₃ precipitates. The effect of Ni₅Ti precipitates on the functional properties of the laser-welded NiTi wires in such intermediate treatment temperature range has been comprehensively investigated in our previous work [6, 7]. The experimental findings point to the fact that the laser-welded wires with post-weld heat treatment (PWHT) at 350 °C (623 K) exhibited the most advantageous functional properties than those heat-treated at other temperatures. However, such performance measurements are only valid at room temperature, and whether the good effect of PWHT at 350 °C (623 K) can improve the functional properties...
at higher test temperatures is still unknown. This is the motivation for this study since NiTi engineering components are often operating at higher temperatures.

**Experimental Details**

**Material and Procedure of Laser Welding**

The material used was commercial Ti-55.91 wt % Ni wire of diameter 0.5 mm (purchased from Johnson Matthey). Butt weld was made on two pieces of the NiTi wires using a 100-W CW fibre laser (Model SP-100C-0013, output wavelength 1091 nm, in-focus spot size 46 μm). The process parameters were optimized by using Taguchi experiment (one of Design of Experiments methods) and were obtained as: 72 W laser power, 115 ms welding time, +0 mm defocusing, and 25 L/min of argon flow rate. After laser welding, post-weld heat-treatment (PWHT) was carried out to the laser-welded samples. The PWHT temperature was chosen as 350 °C (623 K). The selection process for the particular PWHT temperature used in this study was detailed in elsewhere [6]. The PWHT was done by keeping the laser-welded samples in the furnace at the target temperature for 3600 s, followed by water quenching. There were two types of samples: as-welded sample and heat-treated sample (which refers to the laser-welded sample after PWHT) being tested and compared in this study. The weldments possessed three distinct and identifiable zones, namely weld zone (WZ), heat-affected zone (HAZ) and base material (BM).

**Cyclic Deformation Tests at Different Temperatures**

The functional behavior of the samples was tested using an Instron tensile machine equipped with a temperature-controlled chamber. The strain rate was 3 x 10^−5 s^−1. Two types of cycle deformation tests were carried out in this study. The samples in the first test were repetitively loaded to 4 % and then unloaded to 5 MPa for 10 cycles, while incremental strains from 0.7 % to 4.0 % were applied to the samples in the second test, i.e. the samples were incrementally loaded from 0.7 %, 1.5 %, 2.5 % and 4.0 % respectively, followed by unloading to 5 MPa. The tests were carried out at three different temperatures: room temperature (25 °C or 298 K), 50 °C (323 K), and 75 °C (348 K).

**Thermal Phase Transformation Measurements**

The phase transformation temperatures, namely austenitic starting/finishing temperature (A_s/A_f) and martensitic starting/finishing temperatures (M_s/M_f) of the WZ and BM in the samples before and after PWHT have been characterized by differential scanning calorimetry (DSC) in our previous work [6], and the results are shown in Table 1.

**Table 1** Phase transformation temperatures of the WZ and BM in the laser-welded samples before and after PWHT

<table>
<thead>
<tr>
<th></th>
<th>WZ (Before PWHT)</th>
<th>WZ (After PWHT)</th>
<th>BM (Before PWHT)</th>
<th>BM (After PWHT)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Austenitic Starting Temperature (A_s)</td>
<td>-20.6 °C (252.4 K)</td>
<td>22.6 °C (295.6 K)</td>
<td>-3.6 °C (269.4 K)</td>
<td>8.3 °C (281.1 K)</td>
</tr>
<tr>
<td>Austenitic Finishing Temperature (A_f)</td>
<td>-5.8 °C (267.2 K)</td>
<td>33.9 °C (306.9 K)</td>
<td>8.8 °C (281.8 K)</td>
<td>28.2 °C (301.2 K)</td>
</tr>
<tr>
<td>Martensitic Starting Temperature (M_s)</td>
<td>-40.5 °C (232.5 K)</td>
<td>5.3 °C (278.3 K)</td>
<td>2.2 °C (275.2 K)</td>
<td>20.6 °C (293.6 K)</td>
</tr>
<tr>
<td>Martensitic Finishing Temperature (M_f)</td>
<td>-52.3 °C (220.7 K)</td>
<td>-4.7 °C (268.3 K)</td>
<td>-7.0 °C (266.0 K)</td>
<td>1.5 °C (274.5 K)</td>
</tr>
</tbody>
</table>

Phase transformation temperatures are the important variable to influence the functional properties of the laser-welded samples. The samples would either exhibit the shape memory effect (SME) or superelasticity (SE) or a combination of them at different test temperatures. SME would occur if the test temperature is below the M_s while SE would occur if the test temperature is above A_s. In the case where the test temperature falls between the M_s and A_f, a combination of SME and SE would occur [9].

**Results and Discussions**

Figure 1 (a-f) shows the cyclic stress-strain curves for the as-welded and heat-treated samples after loading and unloading to 4 % at different cycles (1, 2, 5 and 10) and at different test temperatures (at room temperature, 50 °C and 75 °C) respectively. In the cyclic stress-strain curves, two distinctive features, stress plateau and residual strain, are the important measures for the functional properties of NiTi alloys. From the phenomenal point of view, the stress plateau signifies the stress-induced martensitic transformation (SIMT) process from austenite to martensite (upper plateau) or vice versa (lower plateau). Residual strain is interpreted as the amount of plastic strain left in the samples after the cyclic deformation tests. As a general rule, the smaller the residual strain, the better is the SE.
Figure 1 (a-f) - Cyclic stress-strain curves for the (a-c) as-welded and (d-f) heat-treated samples after loading and unloading to 4% strain for 10 cycles at different test temperatures.
There is one common characteristic which could be observed from the cyclic stress-strain curves. Both the upper and lower plateau stress levels were found to increase with increasing test temperature. But this relationship was only valid between the room temperature and 50 °C. At 75 °C, the as-welded and heat-treated samples fractured in the first cycle without showing the stress plateau. On the other hand, noticeable residual strain can be seen from the samples when testing at room temperature and 50 °C. At room temperature, the residual strain of the as-welded sample was slightly higher than that of the heat-treated sample, i.e. about 0.2 %. However, the difference of residual strain between the as-welded and heat-treated samples increased substantially to about 0.7 % when raising the working temperature to 50 °C. The residual strain of the as-welded sample further increased while that of the heat-treated sample further decreased, compared with the samples tested at room temperature. The aforementioned observations will be discussed in detail in the following subsections.

Effect of Test Temperature on the Stress Plateau

The increase of plateau stress level with increasing test temperature for NiTi alloys can be described by the Clausius-Clapeyron relationship which is expressed by the following equation:

\[
d\sigma/dT = -\Delta H/T \varepsilon_o \tag{1}
\]

Where \(d\sigma\) is the change in plateau stress level, \(dT\) is the change in test temperature, \(\Delta H\) is the latent heat of transformation, \(T\) is the test temperature and \(\varepsilon_o\) is the transformational strain [10]. The \(d\sigma/dT\) values (for the upper and lower plateau stress levels) of the as-welded and heat-treated samples in this work are calculated and tabulated in Table 2.

**Table 2** The \(d\sigma/dT\) values for the upper and lower plateau stress levels of the as-welded and heat-treated samples between room temperature and 50 °C

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>As-welded Sample</th>
<th>Heat-treated Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>(d\sigma/dT) for the Upper Plateau Stress Level</td>
<td>5.7 MPa/°C</td>
<td>4.1 MPa/°C</td>
</tr>
<tr>
<td>(d\sigma/dT) for the Lower Plateau Stress Level</td>
<td>6.4 MPa/°C</td>
<td>7.2 MPa/°C</td>
</tr>
</tbody>
</table>

The calculation results indicate that the \(d\sigma/dT\) values for the as-welded and heat-treated samples were within the typical range reported in literature, i.e. the documented \(d\sigma/dT\) values for NiTi alloys vary between 3 and 20 MPa/°C. In the case of testing at 75 °C, the disappearance of stress plateau before fracture because the critical stress required to trigger the SIMT process became higher than that to cause fracture of the weld-joint. As such, no stress plateau existed in the samples tested at 75 °C.

Effect of Test Temperature on the Residual Strain

The residual strain accumulated in the stress-strain curves after cyclic tests is an indicator to evaluate the deformation behavior of NiTi alloys. The deformation behavior of the laser-welded NiTi alloys before and after PWHT have been explicitly described in our previous work [6]. To briefly summarize, the residual strain in the as-welded sample at room temperature is predominantly contributed by local plastic deformation in WZ and HAZ. These regions would be plastically deformed when straining beyond a particular point prior to the start of the SIMT process (usually between 0.6 % and 0.8 % strain) [6]. The local plastic deformation in the welded regions is signified by the deviation from linearity between 0.7 % and 1.6 % strain in the magnified stress-strain curve for the as-welded sample at room temperature (see Figure 2 below).

![Figure 2 - Magnified stress-strain curve for the as-welded sample at room temperature](image)

In comparison with the as-welded sample, the deformation behavior of the heat-treated sample is more complicated because both the austenite and twinned (or thermally-induced) martensite coexist in the base metal (BM) at room temperature as it lies between the austenite starting (\(A_s\)) and austenite finishing (\(A_f\)) temperatures (see Table 1). On top of the local plastic deformation in the welded regions, detwinning of the twinned martensite in the BM would be the additional source to the residual strain. Detwinning in NiTi alloys is the unique deformation process which is responsible for the shape memory effect (SME). The deformation caused by detwinning is reversible which can be recovered upon subsequent
heating to a temperature above $A_F$ after the deformation. Surprisingly, even though the heat-treated sample contained the detwinned martensite in the BM, its residual strain was still less than that in the as-welded sample after the cyclic tests. This could be explained by the presence of small-sized Ni$_4$Ti$_3$ precipitates which coherently precipitate in the welded regions of the heat-treated sample after PWHT. It is well known that the Ni$_4$Ti$_3$ precipitates can suppress plastic deformation and hence improve the functional properties of NiTi alloys. The presence of Ni$_4$Ti$_3$ precipitates in the welded regions after PWHT can increase the resistance to plastic flow, and hence reduce the portion of residual strain attributed to the local plastic deformation.

As observed from Figure 3 (a-b), the local plastic deformation in the welded regions was well-covered by the red zone (loaded and unloaded from 1.5 % strain) and green zone (loaded and unloaded from 2.5%). It is noteworthy that the sum of residual strain contributed by the red and green zones in the as-welded sample was considerably higher than that in the heat-treated sample. This in turn indicates that the precipitation of Ni$_4$Ti$_3$ precipitates after PWHT can effectively suppress the local plastic deformation in the welded regions. On the other hand, the experimental results also provided strong evidence to support the argument that the residual strain in the as-welded sample was predominantly attributed to local plastic deformation, i.e. the sum of residual strain in the red and green zones contributed to about 83 % of the total residual strain.

The as-welded sample tested at 50 °C showed a significant increase in the residual strain when compared with that tested at room temperature. Moreover, noticeable fluctuations (as pointed by the arrow in Figure 1b) were found in the stress plateau after the first cycle. As described by the Clausius-Clapeyron relationship in the previous section, the plateau stress level increased with increasing test temperature. As such, the plateau stress level of the as-welded sample increased at 50 °C, and raised to the level close to the fracture limit of the weld-joint (630 MPa) (see Figure 1f). It is reasonable to speculate that local plastic deformation would be more severe when approaching to the fracture limit, causing an increase in the residual strain. Furthermore, such aggravated plastic deformation in the welded regions might subsequently interfere with the SIMT process and give rise to the fluctuations in the stress plateau. In contrast, the stress plateau of the heat-treated sample was very stable and no fluctuations can be found throughout the cyclic tests (as shown in Figure 1e). Moreover, the residual strain in the heat-treated sample was much less than that in the as-welded sample. The further reduction in the residual strain at 50 °C was attributed to the vanishing of detwinned martensite in the BM since the test temperature was higher than the $A_F$, i.e. all twinned martensite in the BM would completely transform into the austenite at 50 °C.

**Conclusions**

In this study, the effect of different test temperatures: room temperature (25 °C or 298 K), 50 °C (323 K) and
75 °C (348 K) on the functional properties of laser-welded NiTi samples before and after post-weld heat-treatment (PWHT) were investigated using cyclic deformation tests. The experimental results indicate that the critical stress (or plateau stress level) to induce the martensitic transformation and the residual strain after the cyclic tests was highly associated with the test temperatures. The plateau stress level was found to increase with increasing test temperature. The residual strain of the as-welded sample was found to increase with increasing test temperature from room temperature to 50 °C (323 K), whilst the heat-treated sample showed an opposite relation, i.e. the residual strain decreased with increasing test temperature in the given range. Both the as-welded and heat-treated samples were found to fracture in the first cycle during the cyclic tests at 75 °C (348 K).

References


