Available online at www.sciencedirect.com
ScienceDirect
Acta Materialia 57 (2009) 5892–5897
Acta MATERIALIA
www.elsevier.com/locate/actamat

Direct physical evidence for the back-transformation of stress-induced martensite in the vicinity of cracks in pseudoelastic NiTi shape memory alloys

S. Gollerthan [a], M.L. Young [a,*], K. Neuking [a], U. Ramamurty [b], G. Eggeler [a]

[a] *Institut für Werkstoffe, Ruhr-Universität Bochum, Universitätsstraße 150, D-44801 Bochum, Germany*
[b] *Department of Materials Engineering, Indian Institute of Science, Bangalore 560012, India*

Received 7 April 2009; received in revised form 27 April 2009; accepted 7 August 2009
Available online 18 September 2009

Abstract

Crack loading and crack extension in pseudoelastic binary NiTi shape memory alloy (SMA) miniature compact tension (CT) specimens with 50.7 at.% Ni (austenitic, pseudoelastic) was investigated using infrared (IR) thermography during in situ loading and unloading. IR thermographic measurements allow for the observation of heat effects associated with the stress-induced transformation of martensite from B2 to B19′ during loading and the reverse transformation during unloading. The results are compared with optical images and discussed in terms of the crack growth mechanisms in pseudoelastic NiTi SMAs. Direct experimental evidence is presented which shows that crack growth occurs into a stress-induced martensitic microstructure, which immediately retransforms to austenite in the wake of the crack.

Keywords: Shape memory alloys (SMA); Martensitic phase transformation; Infrared thermography; Fracture; Tension test

1. Introduction

NiTi shape memory alloys (SMAs) can be found in a broad range of applications in medical technology and engineering [1–4], due to their unique functional and mechanical properties. Shape memory components can fail, and therefore understanding crack initiation and growth is of utmost importance. Although NiTi SMAs have been studied using tensile [5–7] and fatigue [6,8–12] testing, non-destructive techniques, such as infrared (IR) thermography [13] and synchrotron X-ray diffraction [14–20], can provide insight into crack initiation and growth and aid in the prevention of catastrophic failure. Using IR thermography, it is possible to observe thermal changes on the material's surface during mechanical loading which can indicate heat flows associated with martensitic forward and reverse transformations. IR thermography can therefore be used for the surface observations of deformation and failure mechanisms [21–24]. Real-time IR thermography can moreover provide valuable non-destructive information about fatigue-life, making it ideal for continuous monitoring of fatigue for failure prevention [25–28]. Although historically limited by the image acquisition time and detector resolution, current thermographic devices provide real-time image acquisition up to 50 Hz with high thermal resolutions up to 0.02 K. Recently, real-time IR thermography in combination with digital photography was used to examine pseudoelastic polycrystalline NiTi thin tubes during tensile deformation, and it was concluded that IR thermography is valuable for its ability to map the stress-induced martensitic transformations [13].

In the present paper, we use thermography to monitor cracks in pseudoelastic NiTi compact tension (CT) specimens. We assess the potential of thermography to study

* Corresponding author. Tel.: +49 234 32 25910; fax: +49 234 32 14235.
E-mail address: marcus.young@rub.de (M.L. Young).

1359-6454/$36.00
doi:10.1016/j.actamat.2009.08.015

local stress-induced martensitic transformation events in the vicinity of a crack. In order to assess this potential, we compare optical microscopy and synchrotron X-ray diffraction results with our thermographic results. We also show that thermography can provide new insight into the nature of crack propagation in pseudoelastic NiTi.

2. Materials and methods

As described in detail by Gollerthan et al. [29–31], miniature CT specimens were prepared and pre-cracked, according to ASTM standard E399-90 [32], from a commercially available NiTi SMA (SE508) with 50.7 at.% Ni (austenitic, pseudoelastic). The material and its heat treatment are described in detail elsewhere [29,31].

Thermographic measurements were performed on a pseudoelastic NiTi CT specimen with $a/W = 0.525$, where a is the crack length and W is the specimen width, using a VarioTHERM (InfraTec GmbH) thermal camera. Its spectral range related to the lateral resolution of the method extends from 3.4 to 5 μm and the instrument can be used over a wide temperature range. Close to room temperature, temperature differences of the order of 0.1 °C can be easily resolved. Mechanical loading of the CT specimen during thermography was performed using a Zwick/Roell Z100 electromechanical test rig with a temperature chamber. Specimens were coated with graphite ($\varepsilon = 0.98$) to optimize the emissivity (ε). Transmission effects from the back of the sample can be excluded due to the thickness of the specimen (8 mm). The ambient laboratory conditions (21 °C, air pressure of 1013 mbar, and 85% relative air humidity) appear almost completely transparent in the operating spectral range, and thus no further signal corrections are required. Thermographic measurements allow for the detection of heat effects associated with the exothermic stress-induced formation of martensite (B19′) from austenite (B2). Thus, the transformation zones can be observed by monitoring surface temperatures. As compared to the displacement rate of standard CT fracture tests (0.05 μm s^{-1}), higher displacement rates of 20 and 25 μm s^{-1}, where thermal equilibrium is not so rapidly attained, were chosen, thus making latent heats associated with stress-induced martensitic transformations easier to detect. Two sets of experiments were performed, in which thermographic measurements were taken continuously every 5 Hz during uninterrupted loading and unloading. In the first set of experiments, thermographic measurements under displacement control (rate: 20 μm s^{-1}) were taken during loading and unloading below the critical stress intensity. At maximum load, the experiment was held constant to allow for thermal equilibrium before beginning the unloading cycle. The overall experiment lasted less than 3 min. Although measurements were taken every 0.2 s, only eight selected images will be shown here upon loading and unloading. In the second set of experiments, thermographic measurements were collected in displacement control (rate: 25 μm s^{-1}) upon loading above the critical stress intensity.

3. Results and discussion

Fig. 1a and b show optical micrographs of the crack tip region for a fully loaded CT specimen (load: 2860 N, Fig. 1a) and after subsequent unloading (load: 0 N, Fig. 1b). The optical micrograph in Fig. 1a shows features of an affected zone in front of the crack tip which vanish after unloading (Fig. 1b). The optical micrograph in Fig. 1a, however, does not reveal the full size of the transformation zone in front of the crack. This was recently measured using synchrotron X-ray diffraction [29,31] and the white dashed line shows the limit of the affected zone where the presence of stress-induced martensite was detected [29,31]. The affected zone is not a plastic zone in the traditional sense because its size is determined by the extent of the zone where stress-induced martensite forms. The well-known procedures from fracture mechanics [32–34] can be used to obtain an estimate for the size of the plastic zone. Assuming a Poisson's ratio of 0.3, a critical stress for the formation of stress-induced martensite in pseudoelastic NiTi of 330 MPa [29,31], and a critical parameter for crack instability of 34 MPa $m^{1/2}$ [29,31], we obtain affected zone sizes of 1.7 and 0.3 mm for plane stress and plane strain conditions, respectively. These results are in very good agreement with the synchrotron data. Tensile data published in Refs. [29,31] suggest that the plastic deformation of detwinned stress-induced martensite starts at 600 MPa. Using this value to calculate the size of a conventional plastic zone yields a value of 0.5 and 0.1 mm for plane stress and plane strain conditions, respectively, which is much smaller than the size of the zone which we observe experimentally. Instead of detecting a plastic zone resulting from crack tip plasticity, we detect a zone which is associated with crack tip phase transformation. We therefore do not refer to the affected zone as a plastic zone. The optical micrograph of Fig. 1a clearly shows that crack propagation would occur in the affected zone. Fig. 1b suggests that after unloading no traces of this scenario can be detected, because the back-transformation obscures the characteristic features which govern crack growth.

The final objective of the thermographic experiments performed in the present study was to confirm that a crack in pseudoelastic NiTi first grows into a martensitic microstructure (this was proven in Refs. [29,31]) which then vanishes during unloading (this remains to be documented). Therefore, we studied the crack tip region during loading and unloading below the critical value of crack extension as reported in Refs. [29,31]. Thermographic images were collected during loading and unloading of a pseudoelastic NiTi SMA CT specimen as shown in Fig. 2a and b, where temperatures are represented by a range of colors (blue: 26.2 °C, dark green: 26.4 °C, yellow: 26.6 °C, red: 26.8 °C and violet/white: 27.0 °C and above).

In Fig. 2a, thermographic data obtained at four different loading stages are shown (500, 2600, 2670 and 2700 N at 26, 46, 50, and 51 s, respectively, of the experiment). Two zones of local elevated temperature were detected upon