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ScienceDirect
Acta Materialia 57 (2009) 1015–1025
Acta MATERIALIA
www.elsevier.com/locate/actamat

Fracture mechanics and microstructure in NiTi shape memory alloys

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Received 9 September 2008; received in revised form 16 October 2008; accepted 16 October 2008
Available online 26 December 2008

Abstract

Crack extension under static loading in pseudoplastic and pseudoelastic binary NiTi shape memory alloy (SMA) compact tension (CT) specimens was examined. Two material compositions of 50.3 at.% Ni (martensitic/pseudoplastic) and 50.7 at.% Ni (austenitic/pseudoelastic) were investigated. The SMAs were characterized using differential scanning calorimetry to identify the phase transformation temperatures and tensile testing to characterize the stress–strain behavior. A miniature CT specimen was developed, which yields reliable critical fracture mechanics parameters. At 295 K, cracks propagate at similar stress intensities of 30 ± 5 MPa $\sqrt{m}$ into martensite and pseudoelastic austenite. Integrating the miniature CT specimen into a small test device which can be fitted into a scanning electron microscope shows that this is due to cracks propagating into regions of detwinned martensite in both materials. Investigating a pseudoelastic miniature CT specimen in a synchrotron beam proves that martensite forms in front of the crack in the center of the CT specimen, i.e. under plane strain conditions.

Keywords: Shape memory alloys (SMA); Mechanical properties testing; Stress-induced martensite; Tension test

1. Introduction

Microstructures and properties of NiTi shape memory alloys (SMAs) have been described in detail [1–6]. NiTi SMAs are used in a wide range of applications in medical technology and engineering [5–7], because they combine good functional properties with good mechanical strength [1–6,8]. Their mechanical properties have been investigated using tensile (e.g. [9–11]) and fatigue testing (e.g. [9,12–17]). The formation of microcracks during cyclic loading has been observed [15] and crack propagation under cyclic loading conditions has been monitored [16,18–25]. However, the behavior of cracks under static loading conditions in martensitic, pseudoplastic (50.3 at.% Ni) and austenitic, pseudoelastic (50.7 at.% Ni) NiTi microstructures is not yet clear. A systematic study on critical parameters, like K_{IC} values, has not yet been performed, and there is a need for identification of conditions under which cracks in martensitic and pseudoelastic NiTi SMAs become unstable. As of yet, only critical stress intensities of 31 [19,26] and 35 MPa $\sqrt{m}$ [24] for martensitic NiTi SMAs and 34 MPa $\sqrt{m}$ [27,28] for pseudoelastic NiTi SMAs have been reported; the latter value comes from fatigue-loaded very-thin-walled (~400 μm) compact tension (CT) specimens, which is most probably in a plane stress condition [27,28].

The elementary processes governing crack initiation and crack instability are not well understood. Thus, McKelvey and Ritchie [20,21] investigated fatigue crack propagation in pseudoelastic and pseudoplastic NiTi SMAs using disk-shaped compact tension specimens of 9 mm thickness. They found that fatigue crack growth resistance increases with decreasing temperature and that fatigue thresholds were higher and crack growth rates slower in martensite as compared to austenite. They did not observe the forma-

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doi:10.1016/j.actamat.2008.10.055

tion of martensite in front of fatigue cracks growing into pseudoelastic NiTi and relate this absence of stress-induced martensite to the triaxial stress state, which characterizes their plane strain condition. Dauskardt et al. [22] suggest that the formation of martensite in front of a crack tip is a beneficial process which renders the material more damage tolerant and that the absence of stress-induced martensite decreases the material's resistance to crack propagation. It is well known that small plastic zones can form in CT specimens of ductile engineering alloys even under plane strain conditions. Such specimens yield reasonable fracture mechanics parameters as long as the size of the plastic zone (r_p) is significantly smaller than the specimen dimensions (a, B and W), where a, B and W represent crack length, specimen thickness and specimen width, respectively [29,30]. When plastic zones can form by dislocation activity in front of cracks under plane strain conditions, there is no reason why the formation of stress-induced martensite in front of a pseudoelastic crack should be suppressed, because both dislocation slip and martensitic shear are driven by microscopic shear stresses.

The crack tip regions of NiTi SMA CT specimens have been studied using a variety of methods, including optical microscopy [19,26], synchrotron [16,24,26] and neutron [23] diffraction. It has been shown that the size of pseudoplastic zones (regions in front of cracks with favorably oriented martensitic variants) can be reasonably well estimated using fracture mechanics approaches [19,26]. Daymond et al. [24] showed that detwinning indeed occurs in front of cracks in pseudoplastic martensitic NiTi, though Vaidyanathan et al. [23] were unable to observe this due to the limited lateral resolution in their neutron experiments. Robertson et al. [16] studied a thin miniature pseudoelastic NiTi CT specimen subjected to cyclic loading using synchrotron X-ray microdiffraction. In contrast to the conclusions of McKelvey and Ritchie [20,21], they observed the formation of stress-induced martensite very close to the crack tip and were able to resolve the local microtexture [16]. Their thin specimen (thickness: 0.4 mm) was most probably in a plane stress condition and thus it remains to be proven whether stress-induced martensite also forms under plane strain conditions, i.e. in the center of a thicker CT specimen. In addition, a comprehensive comparison of the behavior of cracks in martensitic and austenitic NiTi has never been performed and would be very interesting.

The present work has four aspects. First, it provides a comprehensive treatment of the fracture mechanics characteristics of three material states in NiTi: martensitic, pseudoelastic (austenitic, but prone to the formation of stress-induced martensite) and austenitic (without the potential to form stress-induced martensite) NiTi. Secondly, we compare and discuss the response of miniature CT specimens to mechanical loading and provide critical data for crack extension under static loading for all three material states. We then study the evolution of microstructures in front of cracks, which grow into martensitic and pseudoelastic NiTi using in situ experiments in a scanning electron microscope. Finally, we perform in situ synchrotron experiments to clarify whether stress-induced martensite forms in front of the central part of a crack in a thick pseudoelastic CT specimen (i.e. under plane strain conditions).

2. Materials and experiments

Two NiTi SMAs with 50.3 at.% Ni (martensitic/pseudoplastic at room temperature) and 50.7 at.% Ni (austenitic/pseudoelastic at room temperature) were purchased from Memory Metalle, Weil am Rhein. Both alloys were subjected to thermomechanical treatments (forming and aging steps) including a final 6 min heat-treatment at 500 °C for the pseudoelastic alloy. Both materials were characterized using differential scanning calorimetry (DSC), uniaxial tensile testing, CT fracture testing, optical and scanning electron microscopy (SEM), and synchrotron diffraction.

DSC was performed on both SMAs using a TA Instruments' DSC 2920CE machine in a temperature range from 123 to 423 K at a heating/cooling rate of 10 K min^{-1}, whereby specimens were held for 5 min at the maximum und minimum temperatures. The details of DSC testing have been described elsewhere [31]. Fig. 1a shows the DSC chart of the martensitic material investigated in the present study. When forward and reverse transformations represent one-step events, the characteristic transformation temperatures are defined as M_s (start of the martensitic transformation), M_f (temperature where the forward transformation is completed), A_s (start of the austenitic transformation) and A_f (temperature where the reverse transformation is completed). For the martensitic alloy investigated in the present study, the characteristic temperatures were obtained as $M_s = 317$ K, $M_f = 289$ K, $A_s = 333$ K and $A_f = 357$ K (Fig. 1a). As shown in Fig. 1b, the austenitic pseudoelastic alloy shows a complex three-step transformation upon cooling from the high temperature phase, which is typically related to the presence of small- and large-scale microstructural heterogeneities [31–36]. We attribute the first peak upon cooling to the formation of R-phase ($R_s = 284$ K and $R_f = 267$ K), where R_s and R_f indicate the starting and finishing temperature of the R-phase formation. A small second peak indicates the start of the formation of B19′ ($M_s = 229$ K), which accounts for yet a third peak which ends at the martensite finish temperature ($M_f = 210$ K). While the DSC chart features on cooling appear complex, our pseudoelastic alloy exhibits a simple one-step reverse transformation ($A_s = 276$ K and $A_f = 293$ K). The DSC charts in Fig. 1a and b show that, at room temperature (295 K), the alloy with 50.3 at.% Ni is martensitic while the alloy with 50.7 at.% Ni is fully austenitic. No further effort was made to rationalize DSC chart features.

Uniaxial tensile tests, with dog-bone tensile specimens identical in dimensions to those reported previously [19], were performed using a Zwick/Roell Z100 electromechanical test rig with a temperature chamber. These flat tensile