

Crack propagation mechanisms of an aged nickel-titanium-hafnium shape memory alloy

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ABSTRACT

Mechanisms of crack propagation in a NiTiHf sample were studied from post-mortem microstructure characterizations about cracks. Transmission electron microscopy (TEM) analysis showed that a crack propagated within a martensite band along {011} type II twin planes. The crack was observed to switch between different twin planes to avoid precipitates. The crack tip terminated outside of the martensite band, within surrounding austenite. Amorphization of crack edges in both austenite and martensite was also detected, together with Ni enrichment. High-resolution TEM examination of crack tip inside the austenite phase also confirmed the formation of martensite nanocrystals embedded in the amorphous regions.

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Nickel-titanium-hafnium (NiTiHf) alloys have received considerable attention for their use as high-temperature shape memory alloys (SMAs). They can exhibit stable shape memory effect and superelastic behavior resulting from a solid-state phase transformation from high-temperature B2 austenite to low-temperature B19' martensite through temperature change or applied stress [1,2]. An advantage of NiTiHf alloys is that the predicted stresses for slip are greater than that for twinning in the solid solution compounds [3], and the slip stresses can be driven even higher with H-phase nanoprecipitate strengthening via aging treatments [2,4]. This is why martensitic phase transformations of NiTiHf alloys are highly reversible while they exhibit little or no plastic deformation [1,2,5–8], which has been key to their emerging commercialization.

Due to the increased focus of NiTiHf alloys in medical and aerospace applications, cyclic thermo-mechanical durability and fracture resistance is highly desired. As a result, fundamental understanding of crack propagation in NiTiHf alloys is essential, though most studies on these systems have not included a detailed understanding of failure processes. In binary NiTi alloys, efforts have been made to understand the fundamental mechanisms of phase transformations [9–11], defect accumulation on cycling [12–15], and fracture [16–19]. From these studies, it is known that transformation toughening occurs as a result of stress-induced martensitic transformation accompanying crack growth in initially austenitic materials. However, there is a limited understanding of how microstructure affects the propagation of cracks in the low

crystallographic symmetry martensite phases. Such understanding of fracture behavior in martensites is important to inform damage models for SMAs, which currently focus primarily on the role played by the dissipative mechanism of stress-induced phase transformation on crack growth [20–22], while ignoring phenomena related to the martensite phase itself. Through detailed TEM analysis, the present study documents crack propagation behavior within the martensite phase both near and away from strengthening nanoprecipitates, as well as observations of stress-induced amorphization around the crack tip inside both austenite and martensite phases in NiTiHf alloys.

A NiTiHf alloy with target composition of Ni_{50.3}Ti_{41.2}Hf_{8.5} (at.%) was initially solution-annealed at 1050 °C for 30 min, water quenched, and then aged at 550 °C for 3.5 h and air-cooled to grow strengthening H-phase nanoprecipitates [4]. The samples were encapsulated in a quartz tube under Ar during all heat treatments. Differential scanning calorimetry (DSC) was performed using a TA Instruments Q100 V9.9 with heating and cooling rates of 10 °C/min between –180 °C and 150 °C.

Conventional, high-resolution and high angle annular dark field scanning TEM (HAADF-STEM) were used for characterization of the aged NiTiHf sample using an FEI Talos TEM (FEG, 200 kV). Additionally, a Titan G2 80–200 with ChemiSTEM technology using four embedded Bruker SDD detectors was also used for high spatial resolution energy-dispersive X-ray spectroscopy (EDX) mapping. A Fischione automatic twin-jet electropolisher (model 120) at 13 V was used to make a thin TEM foils from 90 to 100 μm grinded foils. An electrolyte of 30% HNO₃ in methanol (by volume) at around –35 °C was used for electropolishing.

The DSC measurement was performed on a Ni_{50.3}Ti_{41.2}Hf_{8.5} sample after aging at 550 °C for 3.5 h. The transformation temperatures were:

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M_s (Martensite start temperature) = -8°C , M_f (Martensite finish temperature) = -75°C , A_s (Austenite start temperature) = -19°C and A_f (Austenite finish temperature) = 47°C . Therefore, after cooling from the final heat treatment, the sample would be expected to consist of an austenite matrix containing nano-sized H-phase precipitates. This was confirmed by bright field (BF) micrograph as shown in Fig. 1(a). The corresponding selected area electron diffraction (SAED) pattern is also shown in the inset of the figure. The main spots in the SAED pattern belong to the B2 cubic austenite structure, and the super reflections at $1/3$ positions along $\langle 110 \rangle_{B2}$, indicated by arrowheads, are reflections from uniquely oriented H-phase precipitates. The lenticular precipitates embedded in the austenite matrix were 105 ± 5 nm long and 24 ± 1 nm wide.

Further characterization showed the existence of martensite (Fig. 1(b)) in some regions of the same sample. Considering the DSC results, it is evident that partial martensite transformation occurred while electropolishing the TEM sample, which was carried out at -35°C , a temperature between M_s and M_f . Because $A_f = 47^\circ\text{C}$, which is warmer than the room temperature, some of the martensite was

thermodynamically stable and did not transform back to austenite when the sample was heated back to room temperature after electropolishing.

The formation of such large, preferentially-oriented martensite lath structures, as opposed to randomly oriented collections of twins of self-accommodated martensite that usually form during cooling without an external stress [23], is attributed to mechanical (stress relaxation) effects in the TEM foil during transformation. Stress relaxation effects in thin foil plan view TEM specimens have been studied using finite element analysis [24]. The stress can originate from lattice mismatch [24] and heterogeneity in stress distribution [25]. Abnormal stress-induced grain growth in nanocrystalline metals was also observed due to stress relaxation in TEM samples [25]. In the present NiTiHf sample, cracks were also observed, providing further evidence for local mechanical stresses in the sample, most likely due to lattice mismatch between the austenite and nano-H-phase precipitates [26] and internal stress heterogeneities inherent to nano-scale samples [27,28].

Fig. 2 shows a BF micrograph of one of the observed cracks. This condition provided opportunity to study crack propagation mechanisms. The crack initiated from the edge of the TEM specimen hole (upper right corner of Fig. 2(a)) and propagated through the martensite phase and finally entered and arrested in the austenite phase. Fig. 2

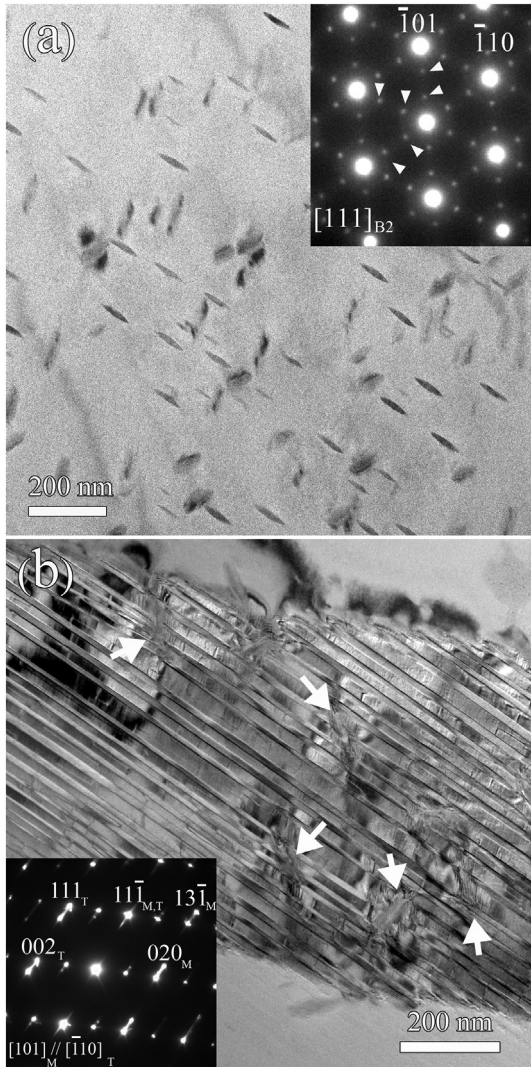


Fig. 1. (a) conventional BF micrograph of $\text{Ni}_{50.3}\text{Ti}_{41.2}\text{Hf}_{8.5}$ sample after aging at 550°C for 3.5 h. The corresponding SAED pattern along the $[111]_{B2}$ zone axis is presented in the inset, with H-phase super reflections indicated by arrowheads. (b) A BF-TEM micrograph and corresponding SAED from the of the same sample taken along $[101]_M // [\bar{1}10]_T$ showing the presence of B19' monoclinic martensite phase with (011) type II twin structures.

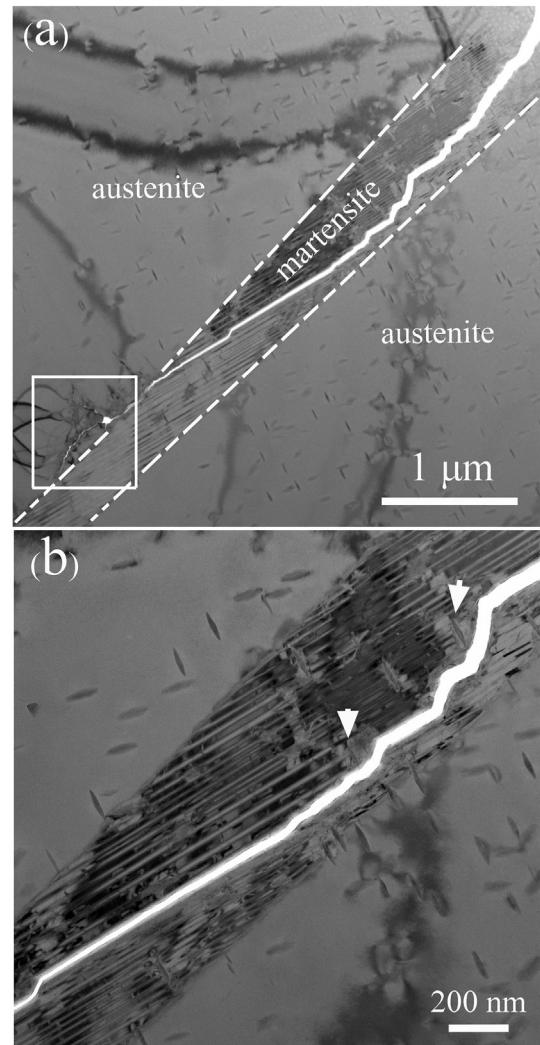


Fig. 2. (a) conventional BF micrograph showing a crack within the martensite phase of the NiTiHf alloy and (b) high magnification BF image showing the crack path along (011) type II twin planes. The crack is interrupted when it encounters H-phase precipitates (indicated by white arrows) and redirected to another twin boundary creating a torturous crack path.

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