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Instability of cyclic superelastic deformation of NiTi investigated by synchrotron X-ray diffraction

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Abstract—Motivated by an assumption that the instability of the cyclic tensile superelastic behavior of NiTi polycrystal is linked to its fatigue performance (number of cycles till failure), the instability was investigated by high resolution in situ synchrotron X-ray diffraction method. NiTi wires were cyclically deformed in tension at room temperature while X-ray diffraction patterns were recorded in three preselected states along the superelastic stress–strain curve, analyzed and interpreted in terms of the gradual evolution of microstructural state during cycling. It is found that the cyclic instability is due to the gradual redistribution of internal stresses originating from the accumulation of incremental plastic strains accompanying the stress induced martensitic transformation in constrained polycrystalline environment. The degree of cyclic instability increases with the increasing involvement of slip in the hybrid slip/transformation process, which depends on initial microstructure (grain size, defects, precipitates), martensitic transformation (crystallographic incompatibility between transforming phases), temperature and parameters of the cyclic loading (strain rate, amplitude, stress state, type of loading etc.).

Keywords: Shape memory alloy; NiTi; Superelasticity; Cyclic deformation; In situ X-ray diffraction

1. Introduction

Functional thermomechanical responses of NiTi due to the martensitic transformations are frequently utilized in engineering applications [1] and stress–strain temperature constitutive behavior is simulated by SMA models [2]. In both cases, it is frequently implicitly assumed that the involved strains are completely recoverable and the responses are stable upon cycling. In reality, however, this is not the case. On the contrary, the cyclic thermomechanical behaviors of NiTi are inherently unstable, the concern is mainly how much. The drift of the stress–strain-tempera ture response during cycling is termed functional fatigue [3]. It is considered to be a problem because of two reasons: (i) since it adversely affects the performance of NiTi elements in engineering applications [1,3] (superelastic members, vibration damping members or actuators) and (ii) since it is presumably responsible for fatigue degradation and preliminary failure of NiTi [8]. Impact of the functional fatigue on practical applications (issue i) can be partially eliminated through stabilizing the cyclic responses by training [4] but the limited fatigue life remains to be a serious problem for the SMA technology. The link between the functional fatigue [3] and structural fatigue [8] can be possibly understood in terms of dissipation energy based criteria [9] for structural fatigue (the more energy is dissipated during the cyclic stress induced martensitic transformation, the shorter is the fatigue lifetime). Given the growing evidence on the role of environmental effects in superelastic fatigue [10], however, this cannot be safely considered as a general controlling mechanism of the Nitinol fatigue, particularly to the superelastic deformation in fluids.

It has been known from early days of the Nitinol research that the instability of cyclic superelasticity is due to plastic deformation by dislocation slip accompanying the stress induced martensitic transformation and accumulation of residual martensite [11,12]. Today's research along this direction concerns mainly fatigue, particularly, how the accumulation of dislocation defects upon cycling is exactly related to the functional and structural fatigue of NiTi [3,6,8,10,13]. What are the dislocations created by thermal [5] and mechanical [6] cycling? How the dislocation slip activity upon cycling is related to the microstructure of NiTi wires [7]? Does the incremental slip occur during the forward or reverse transformations [14]? Does the slip occur in the austenite or in the martensite phase [14,28]? Does the slip occur at the propagating austenite/martensite interface [15] or elsewhere in the polycrystal in parallel to the transformation [26]? What are the actual stress and

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strain conditions at the propagating shear band front [16], where all the deformation/transformation proceeds in highly coordinated localized manner? To answer these questions, more experimental evidence on the relation between the martensitic transformation, dislocation slip, deformation instability, microstructure evolution, damage accumulation and fatigue of superelastic Nitinol will be needed.

In this work, we have investigated the instability of cyclic tensile superelastic deformation of NiTi at room temperature by employing high resolution in situ synchrotron X-ray diffraction method. The key advantage of this method for this particular research is that it can be used to follow the evolution of the microstructure during the cyclic superelasticity nondestructively - without interfering with the deformation/transformation processes involved. In contrast to the TEM method used traditionally to characterize deformation induced microstructure of fatigued metals, the in situ X-ray (neutron) diffraction method enables to perform quantitative analysis of the lattice defects, internal stress, textures and fractions of austenite and martensite phases in oriented grains evolving continuously during the tensile cycling of NiTi.

Superelastic deformation of NiTi in tension was already a subject of many dedicated in situ X-ray (neutron) diffraction studies in the literature [17–23]. Majority of in situ diffraction experiments reported in the literature, however, focused on 1–2 cycles on bulk samples in compression. Only very few studies dealt with tensile loads on NiTi wires in tension [21] and none with cyclic tension till failure. The reason is the localized deformation of NiTi in tension [22] preventing meaningful in situ diffraction studies of NiTi during continuous tensile loading. Moreover, since the actual material strains tend to be either less than 1% outside the shear band or more than 6% within the shear band, evaluation of strain amplitude dependence of lifetime in fatigue tests on NiTi wire in tension is questionable.

High energy synchrotron X-rays are very suitable for in situ diffraction studies of thin NiTi wires due to the small gauge volume involved. The nanograined microstructure yields these NiTi wires high strength and unique properties [6,7] beneficially used in superelastic medical devices. As far as we are aware of, there are no literature reports dealing with in situ X-ray diffraction studies during cyclic superelastic deformation of thin medical grade NiTi wires in tension. There are reports dealing with in situ diffraction studies during 1 or 2 tensile cycles [21], during thermal cycling under tensile load [23] or diffraction studies focusing on structural fatigue of NiTi [8].

Recently, micromechanics models in which plastic deformation accompanies cyclic martensitic transformations [24–28] were introduced in the literature to simulate unstable thermomechanical behaviors of NiTi (large strains, high temperature, actuation cyclic deformation with ratcheting, etc.). For example, Song et al. [24] claim that martensite plasticity upon excessive loading above the plateau restrains the martensitic transformation and degrades the cyclic superelasticity. Saaleb et al. [25] introduced internal state variables related to the inelastic deformation to regulate the material's evolutionary response during cycling. Novak et al. [28] introduced plastic deformation as additional deformation mechanism into an earlier developed micromechanics crystallographic model of SMA polycrystals [27] to analyze the instability of cyclic thermomechanical behaviors of NiTi in tension at various temperatures. This highly simplified uniaxial model (NiTi polycrystal is represented by an aggregate of mutually interacting uniaxial domains of oriented lattice, in which multiple deformation/transformation processes proceed) allows for detailed consideration of anisotropies of multiple deformation mechanisms acting in superelasticity. Besides predicting the instability of the cyclic macroscopic stress–strain-tem perature responses of polycrystals, it predicts also how phase fractions, plastic strains, transformation strains and stresses averaged over families of equally oriented domains (polycrystal grains) evolve during the cyclic thermomechanical loading. Since this "diffraction like information" from the model can be directly confronted with the results of in situ diffraction experiments, we frequently refer below to the simulation results [28] achieved by this model. Richards at al [26] published recently a computational study of the cyclic superelasticity of NiTi polycrystal dealing with the interplay between transformation and plasticity incorporating a more realistic 2D network of constrained polycrystal grains.

It comes out from this short introduction that investigation of dislocation slip accompanying martensitic transformation and consequent instability of cyclic superelastic deformation behavior of thin NiTi wires by combination of TEM, in situ X-ray (neutron) diffraction and micromechanics modeling is a suitable approach to establish the urgently needed link between the microstructure evolution and functional fatigue of NiTi.

2. Experimental procedures

2.1. NiTi wires

The experiments were performed on Fort Wayne Metals superelastic NiTi wires #1 (Ti-50.8at%Ni) with diameter $d = 0.1$ mm and length 50 mm. All samples were prepared by heat treating cold worked wires by short electric current pulses [6] of controlled power $P = 125$ W/100 mm using annealing time 12, 14, 15, 16, 16, 5 and 18 ms in Peltier chamber with controlled temperature. Following these short time electropulse treatments, all wires have the same chemical composition but very different microstructures due to a different degree of recovery and recrystallization. Grain size of the microstructure increases [6] and its resistance to plastic deformation decreases [7] with the increasing pulse time (maximum temperature reached during the joule heating). Cyclic superelastic stress–strain responses of selected wires in 10 tensile cycles at room temperature and corresponding microstructures with and without dislocation defects created during superelastic cycling are shown in Fig. 1. Fig. 2 shows evolution of residual strain (not recovered upon unloading) during the tensile cycling for all investigated wires.

2.2. In situ X-ray diffraction experiment

In situ synchrotron X-ray diffraction experiments were performed at high resolution powder diffraction beamline ID31 (newly ID22) at ESRF Grenoble. Energy used was 31 keV ($\lambda = 0.4$ Å) and beam size 1×2 mm. A self-made thin wire tester dedicated for in situ X-ray diffraction studies was designed and built. Wire samples of initial length ~50 mm were carefully gripped using capillary. The rig was mounted on the X-ray diffractometer in vertical