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Size effect on the mechanical behavior of single crystalline Fe-31.2Pd (at.%) micropillars

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ABSTRACT

The size effect on the mechanical behaviors of single crystalline Fe-31.2Pd (at.%) micropillars with four different pillar diameters (approximately 2 μm, 1 μm, 500 nm and 400 nm) were studied by compressing in the [001] direction. Both the Young's modulus and yield stress increase with the decrease of pillar diameter. The main defects in the plastically deformed pillars are the {111} deformation twins, which could be covered by high density of dislocations after further plastic deformation in a 400 nm pillar. A repeatable elastic-like strain ~4% was observed for more than 40,000 cycles.

Shape memory alloys (SMAs) are widely employed as sensors and actuators due to their two well-known properties: shape memory effect (SME) and superelasticity (SE) [1–3]. These characteristics are caused by thermoelastic martensitic transformations (MTs) which are diffusionless reversible structural transformations between the high symmetrical parent phase and the low symmetrical martensite phase. In some SMAs, the MT is accompanied by an anomalous softening in the elastic constant C' $[=(C_{11}-C_{12})/2]$ [4–10]. This softening occurs as the temperature approaches the transformation temperature and also as external uniaxial stress increases. However, the research on the softening associated with MTs was mainly conducted on bulk SMAs, and the size effects on the softening have yet to be studied, as far as the authors are aware. Considering the size effects on the MT temperature reported to date [11–13] and the relation between the softening and MTs, we may expect the appearance of size effects on the softening of elastic constants in SMAs.

Recently, owing to the development of micro/nano machining techniques, we can prepare small scale SMAs and measure their mechanical behavior. Micropillars of SMAs are suitable three-dimensional materials for the investigation of mechanical properties at a small scale [14–18]. Previous studies revealed that several important characteristics of the SMAs could be influenced by the pillar size. For instance, the critical stress for inducing an MT and stress hysteresis both increase with decreasing pillar diameter [16,17]. However, the size effect on the elastic constants is barely paid attention, because most SMA pillars are made from materials that exhibit typical first-order MTs, such as Ti-Ni-based SMAs, Cu-Al-based SMAs [14–18]. In these alloys, the elastic constant C' is relatively large (~10 GPa or larger), even in the vicinity of the transformation temperature [4,5]; therefore, the size effect on the elastic constant could be too weak to be detected.

To reveal the size effects on the elastic constants in SMAs, it is preferable to use an SMA that exhibits extreme low value of C' because a small change in elastic properties could be amplified. Recently, an elastic-like strain of more than 4% was observed in a disordered Fe-31.2Pd (at.%) bulk single crystalline specimen [19,20], which exhibits a weak first-order MT from a face-centered cubic (FCC) structure to a face-centered tetragonal (FCT) structure with a tetragonality (c/a) smaller than one [21,22]. This alloy showed a significant softening in the elastic constant C' due to the band Jahn-Teller effect, and C' decreases to the order of 1 GPa on approaching the transformation temperature [20,23].

In addition, the size effect on the yield stress of SMAs is also unclear [24]. The interactions of dislocations with the second phase or the modulated structure could significantly influence the yield stress of SMAs. Due to the simple structure and the elastic-like deformation for the

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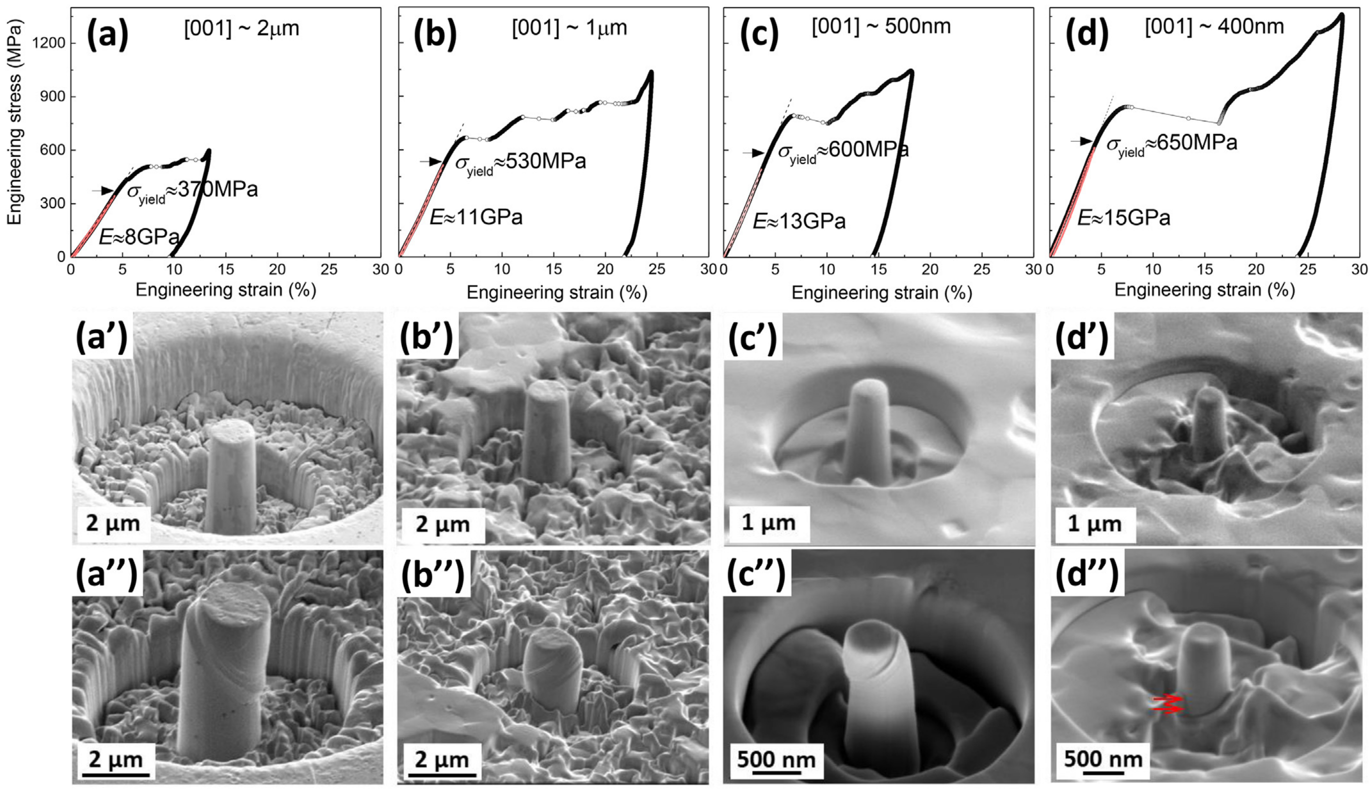

Fig. 1. The engineering stress-strain curves for Fe-31.2Pd (at.%) single crystalline pillars with initial diameters of approximately 2 μm (a), 1 μm (b), 500 nm (c) and 400 nm (d). The red curves indicate the elastic deformations, and the black curves are the plastic deformations. The dashes lines are fitted to the linear region between 1% and 4% of engineering strain. The morphologies of the pillars before (a′–d′) and after (a″–d″) plastic deformation. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fe-Pd single crystal, it is also a good candidate for investigating the size effect on the yield stress.

Therefore, in this work, we examine the mechanical behaviors of micropillars of a single crystalline Fe-31.2Pd (at.%) alloy exhibiting significant lattice softening. The size effect on the Young's modulus and yield stress were studied. The defects in the plastically deformed pillars were also investigated. The stabilities of the large elastic-like strain of the pillars were confirmed.

A bulk single crystal of an Fe-31.2Pd (at.%) alloy was grown by a floating zone method. Its crystallographic orientations were determined by a Laue camera. After homogenization at 1375 K for 24 h, the crystal was quenched into ice water to retain the disordered parent phase. Micropillars with compressive orientations of [001] were fabricated from the single crystal using a focused ion beam (FIB). The diameters of the pillars were approximately 2 μm, 1 μm, 500 nm and 400 nm with a typical height to diameter ratio of 3:1. The compression tests were conducted at room temperature (RT, ~298 K), which is higher than the MT temperature of the Fe-31.2Pd alloy (~240 K), using a TriboIndenter (Hysitron Ti-950) system equipped with a 10 μm flat-ended diamond tip. The loading/unloading stress rate was 20 μN/s. In the long-term cycling experiment, the compressive tests were conducted at a stress frequency of 0.5 Hz. The stress-strain curves were evaluated by using the mean diameter of the pillar.

For the plastically deformed pillars, an FIB lift-out technique was used to prepare the thin foils, and then the microstructures were characterized at RT using a JOEL 2100F transmission electron microscope (TEM) at an accelerating voltage of 200 keV.

Fig. 1 displays typical stress-strain curves for the [001] Fe-31.2Pd single crystalline pillars with initial diameters of approximately 2 μm (a), 1 μm (b), 500 nm (c) and 400 nm (d). (The corresponding load-depth curves are shown in Fig. s1(a–d), and those curves for similar pillars are also presented in (e–l).) The red curves in the figure correspond to the elastic deformation ranges where the hysteresis between the

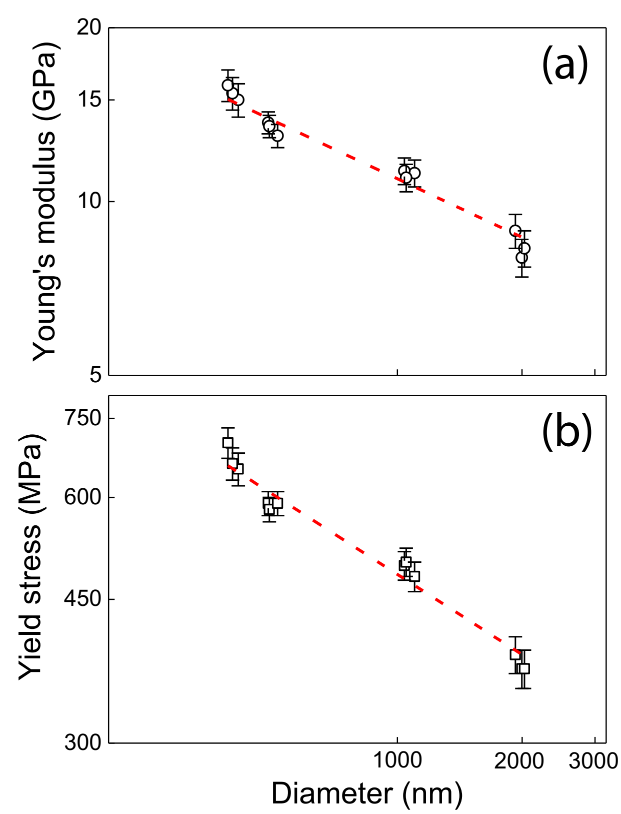

Fig. 2. Logarithmic plot of the Young's modulus (a) and the yield stress (b) as a function of the diameter size of the Fe-31.2Pd (at.%) single crystalline pillars. The error on each mark comes from the inaccuracy of evaluating the diameter of the pillar from SEM images. The dash lines indicate the power law fitting process as described in the manuscript in details.