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Stable elastocaloric effect under tensile stress of iron-palladium alloy and its *in situ* X-ray observation

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The elastocaloric effect under tensile stress along the [001] direction has been investigated in an Fe-31.2Pd (at.%) alloy, which exhibits a weak first-order martensitic transformation at 230 K from a face-centered cubic structure to a face-centered tetragonal ($c/a < 1$) structure. A stable temperature variation of 1.9 K was observed at room temperature (~288 K) up to 10000 cycles of sinusoidal tensile stress (200 MPa). The stress-strain curve shows a macroscopic strain of 1.2% under 200 MPa with a negligible stress hysteresis. The macroscopic strain coincides with the lattice strain obtained by *in situ* X-ray diffraction. A significant extension of the 200 reflection, perpendicular to the tensile direction, was observed. This peak extension could be the way the specimen realizes a high elastic strain, even under tensile stress.

1. Introduction

Due to their high efficiency and environmental friendliness, solid-state cooling systems hold great potential to replace conventional vapor-compression cooling systems [1]. The refrigerant of a solid-state cooling system uses one of the following caloric effects: magnetocaloric [2,3], electrocaloric [4,5], barocaloric [6,7], or elastocaloric [8,9], depending on the type of external field. Recently, elastocaloric effects in shape memory alloys (SMAs) have been attracting increasing attention due to the pronounced release and absorption of latent heat associated with stress-induced martensitic transformations (MTs) [10,11].

However, the use of first-order MTs in SMAs has several drawbacks for solid-state refrigeration. One is the hysteresis loss, which appears in the stress-strain curves [12–14]. The hysteresis loss directly deteriorates the coefficient of performance of the material (COP), which is defined as the ratio between cooling power and input power [15,16]. Another drawback is the accumulation of defects due to the repeating stress-induced MTs. The accumulation of defects could severely deteriorate the elastocaloric effect and result in fracture of the material. As an example, the adiabatic temperature variation caused by the elastocaloric effect of a $Ni_{50}Fe_{19}Ga_{27}Co_4$ (at.%) single crystal has been reported to decrease by approximately 30% after a fatigue test of up to 3000 cycles [17]. Hence, it is extremely important to reduce the hysteresis loss and accumulation of defects of refrigerant materials.

One solution to reduce both the hysteresis loss and accumulation of defects is to use materials with a weak first-order MT. Recently, a significant elastocaloric effect has been observed for an Fe-31.2Pd (at.%) alloy [18,19], which exhibits a weak first-order MT from a face-centered cubic (FCC) structure to a face-centered tetragonal (FCT) structure with tetragonality (c/a) smaller than one [20,21]. This alloy shows a significant softening of the elastic constant C' due to the band Jahn-Teller effect; therefore, the elastic strain along the [001] direction depends strongly on temperature, which causes significant elastocaloric effect. Since the elastocaloric effect of the Fe-31.2Pd (at.%) alloy under compressive stress is mainly caused by elastic strain, the hysteresis loss is negligible and

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the accumulation of defects is negligible as well, unless the stress exceeds the yield stress. Due to these characteristic features, an Fe–Pd alloy exhibiting FCC-FCT transformation could be a potential refrigerant material for solid-state cooling systems.

Recently, several devices using elastocaloric effects in SMAs have been proposed. Since a larger surface-to-volume ratio enables faster heat transfer, these devices use tensile stress instead of compressive stress to induce MTs [22–24]. Although Fe–Pd alloys exhibit excellent elastocaloric effects under compressive stress, the caloric effects under tensile stress have yet not been examined. Since the FCT phase is contracted along the [001] direction compared with the FCC phase [25], both the elastic behavior and elastocaloric effect under tensile stress could be significantly different from those under compressive stress.

The present study was carried out to reveal the elastocaloric effect and elastic deformation behavior of a single crystal of Fe-31.2Pd (at.%) alloy under tensile stress. We focused on the stability of the elastocaloric effect. For this purpose, up to 10000 cyclic tests were carried out. In addition, to understand the deformation behavior under tensile stress, *in situ* high-energy X-ray diffraction (HE-XRD) experiments were carried out under tensile stress.

2. Experiments

An ingot of an Fe-31.2Pd (at.%) alloy was prepared by arc melting using a Fe rod (99.998%) and a Pd sheet (99.99%) as starting materials. A single-crystalline boule was grown using a floating zone method at a growth rate of 3 mm h^{-1} and its orientation was determined by the back-reflection Laue method. After homogenization at 1375 K for 24 h, the boule was quenched in ice water to retain the disordered parent phase. From the boule, two single-crystalline sheets were cut by electrical discharge machining, with the dimensions of 0.2 mm × 4.2 mm × 25.2 mm (Specimen A) and 0.2 mm × 3.8 mm × 28.0 mm (Specimen B). All the edges were parallel to one of the ⟨001⟩ directions.

The stress-strain curves were measured using an Instron-5966 testing machine at a strain rate of 10^{-3} s^{-1} at room temperature (RT), and the strain was recorded using an advanced video extensometer.

The temperature variation during stress application and removal was measured using a stress-controlled dynamic testing machine (BOSE 3510) at RT in a stress range of 12–200 MPa and frequencies of 0.02, 0.05, 0.1, and 0.2 Hz. These frequencies correspond to the average strain rates of 8×10^{-4}, 3.2×10^{-4}, 1.6×10^{-4}, and 8×10^{-3} s^{-1}, respectively. One side of the specimen was coated with a thin black paint, and the temperature of this side was monitored using a multi-detector infrared (IR) camera FLIR SC7700M.

In situ HE-XRD experiments (photon energy = 105.7 keV) were performed at the beam line of 11-ID-C, Advanced Photon Source, Argonne National Laboratory. Uniaxial tensile stress was applied at RT along the [001] direction of the single crystal sheet, and the incident beam was close to the [010] direction. A PerkinElmer α-Si flat-panel large-area detector was used to collect two-dimensional (2D) diffraction patterns. At each stress state, the sample was oscillated ±5° around the [001] or [100] axes in order to obtain the Bragg reflections. The software package FIT2D was used for the diffraction data analysis.

3. Results

3.1. Elastic deformation and elastocaloric effect

Fig. 1 shows the stress-strain curve of the Fe-31.2Pd (at%) single crystalline sheet (Specimen A) at RT. In the measurement, a

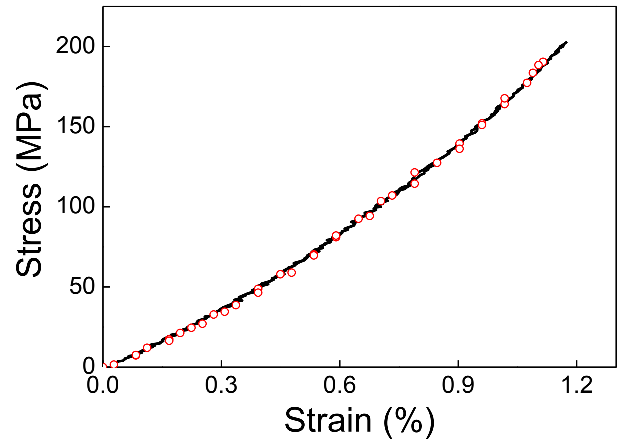

Fig. 1. Stress-strain curve of the Fe-31.2Pd (at%) single crystalline sheet under a tensile stress along the [001] direction up to 200 MPa at RT. The open red circles represent the strains calculated from *in situ* XRD. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

maximum tensile stress of 200 MPa was applied along the [001] direction. There is no detectable hysteresis between stress applying and removing processes. The elastic strain under 200 MPa reached 1.2%.

In order to detect the elastocaloric effect under tensile stress, the spatial-resolved temperature variation was measured. Fig. 2(a) shows a representative IR image obtained during the test. The dashed white rectangle in Fig. 2(a) represents the specimen. Fig. 2(b) shows a series of thermal images of the white rectangle area monitored during the unloading process from 200 MPa at a frequency of 0.02 Hz. The time interval between two adjacent images is ~4 s. As the stress varies at a rate of ~8 MPa s^{-1}, the stress difference between two adjacent panels is ~32 MPa. It is observed in Fig. 2(b) that the temperature of the specimen decreases with decreasing stress. We notice a slight difference in temperature

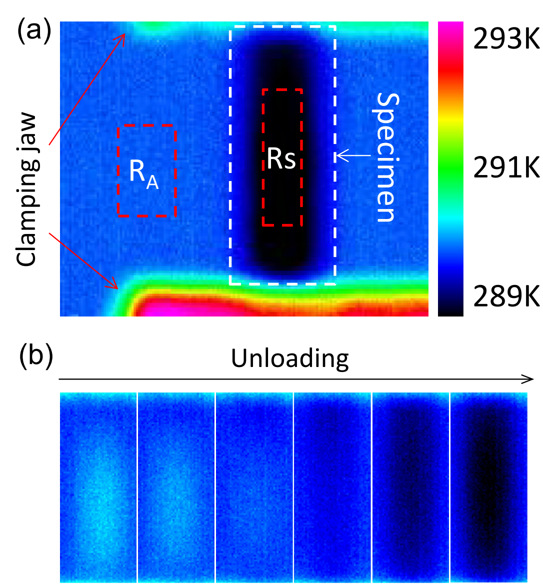

Fig. 2. (a) Typical temperature image of the Fe-31.2Pd (at.%) single crystalline sheet during the tensile test at RT. The specimen and ambient temperatures are averaged over the representative areas in the red rectangles R_S and R_A, respectively. (b) Temperature images of the specimen in the white rectangular area during the unloading process at a frequency of 0.02 Hz. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)