



Two-way shape memory effect under multi-cycles in [001]-oriented $\text{Ni}_{49}\text{Fe}_{18}\text{Ga}_{27}\text{Co}_6$ single crystal

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ABSTRACT

The effects of prior thermo-mechanical treatment on two-way shape memory effect (TWSME) and its stability under thermal cycles in [001]-oriented $\text{Ni}_{49}\text{Fe}_{18}\text{Ga}_{27}\text{Co}_6$ single crystals were investigated. In this study, TWSME in quenched, stress-free and stress-assisted aged crystals was induced by 100 isothermal loading/unloading cycles ($\epsilon = 6\%$, $\sigma_{\max} = 140$ MPa). Maximum reversible TWSME strain up to 5.5% was observed in stress-assisted aged single crystals in which a full one-way transformation strain ϵ_{tr} is achieved under minimum stress levels and the favourably oriented internal stress fields are formed by the selection of certain precipitate variants during stress-assisted ageing. In quenched and stress-free aged crystals TWSME strains are less than 2.2%. The mechanisms of cycling degradation of TWSME (decrease of TWSME strain and change in martensitic start temperature M_s) have also been discussed. In quenched single crystals, 100 thermal cycles result in irreparable degradation of TWSME. In contrast, age hardening in stress-free and stress-assisted aged crystals results in unchanged M_s temperature during thermal cycles and the restoration of the TWSME strain due to additional training (10 isothermal loading/unloading cycles).

1. Introduction

Two-way shape memory effect (TWSME) enables the enhancement of the functionality of the working element from the shape memory alloy at the engineering of actuators [1–5]. The actuator working element with TWSME changes its size with temperature variation reversibly and without any external stresses, unlike the one-way shape-memory effect (SME). The opportunity to realize a multiple reversible strain in ferromagnetic shape memory alloys, not only due to temperature variation, but also to the magnetic field, makes significant attention necessary to further the investigation of TWSME.

Different thermomechanical treatments are used to obtain TWSME, which can be achieved by a plastic deformation in either austenite or martensite, thermal cycling through the martensitic transformation temperature (MT) interval under a constant load or stress cycling under a constant temperature, and a stress-assisted ageing in austenite [1–9]. In all cases, the oriented internal stress fields form in the material because of the generation of dislocation structure, residual martensite and the oriented growth of coherent non-equiaxial precipitates, extended along special crystallographic direction. These oriented internal stress fields provide the oriented martensite growth, and thus the reversible

macroscopic strain (TWSME strain) with cooling/heating under stress-free conditions.

The main problem that should be solved for wide application of TWSME in shape memory alloy actuators is the small reversible strain. Commonly, the TWSME reversible strain ϵ_{TWSME} does not exceed 10–60% of the maximum theoretical transformation strain ϵ_{tr} [6]. However, the maximum experimental value of reversible strain at one-way SME corresponds to the theoretical transformation strain $\epsilon_{SME} \approx \epsilon_{tr}$. In addition, it is necessary to enhance the stability of both MT temperatures and the TWSME strain with repeated thermal cycling through the interval of MT.

It was experimentally shown [6] that thermomechanical treatment, including the stress-assisted ageing at 673 K for 4 h and subsequent thermal cycling (cooling/heating) under compressive stresses of 80 MPa (isobaric training), is the effective way to induce TWSME in [001]-oriented ferromagnetic $\text{Ni}_{49}\text{Fe}_{18}\text{Ga}_{27}\text{Co}_6$ single crystals. This method yields high values of reversible TWSME strain, up to 4.5% (that is equal to $0.7\epsilon_{tr}$).

To date, the systematic studies of TWSME stability with thermal cycling under stress-free conditions have not been carried out on ferromagnetic $\text{NiFeGa}(\text{Co})$ single crystals. It is assumed that isothermal

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training in superelasticity temperature intervals (loading/unloading cycles at constant temperatures) generates an increase in the reversible TWSME strain and yields a high cyclic stability of TWSME in comparison to isobaric training in NiFeGa(Co) single crystals [6]. Firstly, only oriented martensite is formed in the whole sample volume during the stress-induced MT. It was shown [7] on TiNi alloys that training resulting in the formation of self-accommodating structure of martensite at cooling, followed by its reorientation under stress, is less effective for inducing TWSME as compared to the oriented growth of stress-induced martensite. Secondly, it is assumed that the stable crystal microstructure during isothermal training is due to an increase in the number of loading/unloading cycles, up to 100. Thirdly, the Ni₄₉Fe₁₈Ga₂₇Co₆ single crystals were chosen for the investigation due to the opportunity of controlling both the crystallographic structure of austenite (*B2* or *L2₁*) and the type of stress-induced MT (*L2₁-14M-L1₀* or *B2-L1₀*) [6,10,11]. The type of martensite (*14M* or *L1₀*) defines the critical stress level for martensite formation and the dissipative energy during stress-induced MT. Single crystals of NiFeGa(Co) alloys possess SME, superelasticity (SE) and magnetically induced strain with a large reversible strain ~ 10%, and they are one of the most perspective ferromagnetic materials with thermoelastic MT for application [12,13].

Based on the above, the aim of the present work is to study the efficiency of isothermal training to induce TWSME and to investigate TWSME cyclic stability depending on preliminary thermo-mechanical treatment in [001]-oriented Ni₄₉Fe₁₈Ga₂₇Co₆ (at%) single crystals.

The [001]-oriented crystals were chosen for study because they possess the maximum reversible strain $\epsilon_{tr} = 6.2\%$ in conditions of the absence of *L1₀*-martensite detwinning under applied compressive stress at reversible thermal- and stress-induced MT [6]. Consequently, the twinning structure of *L1₀*-martensite and the orientation of the habit plane are not changed with the increase of external compressive stress levels during the stress-induced MT in [001]-oriented NiFeGa(Co) crystals. This factor defines the high mobility of the interphase boundary and the minimum values of dissipative energy (minimum thermal and stress hysteresis) at MT.

2. Experimental procedure

Single crystals with a nominal composition of Ni₄₉Fe₁₈Ga₂₇Co₆ (at%) were grown by the Bridgman technique in an atmosphere of inert gas. Samples were electro-discharge machined with the orientation along the [001]-direction. Compression samples had dimensions of 3 mm × 3 mm × 6 mm. The orientation of the samples and their single-crystalline structure were verified with a DRON-3 M X-ray diffractometer. The study was carried out on Ni₄₉Fe₁₈Ga₂₇Co₆ single crystals after different thermo-mechanical treatments: annealing at 1373 K for 25 min (min) followed by a quenching in water (quenched *B2*-crystals), stress-free ageing of quenched *B2*-crystals at $T = 673$ K for 4 h (*L2₁*-crystals) and stress-assisted ageing under a stress level of $\sigma = 100$ MPa, applied along the $[\bar{3}12]$ -direction (*L2₁(σ)*-crystals). The details of stress-assisted ageing were shown in [6]. The isothermal loading/unloading cycles (100 cycles) in the condition of SE at temperatures $T = A_f + (16 \div 23)$ K were used to induce TWSME and the maximum strain was ~ 6.0% with maximum applied stresses of 140 MPa. All experiments were conducted on the electro-mechanical testing frames ElectroPuls E3000 and Instron VHS 5969 at the deformation rate of $d\epsilon/dt = 1 \times 10^{-3} \text{ s}^{-1}$. TWSME was investigated on a specially constructed frame at cooling/heating with the minimum applied stress of 0.7 MPa, which is necessary for sample clamping in order to measure its reversible strain. The characteristic temperatures of MT (M_s , M_f , A_s and A_f) were defined both by the dependence of electrical resistance on temperature and by differential scanning calorimetry. Optical metallography was obtained by using the microscopic complex Keyence VHX-2000. Sample strain was measured by calliper (hole-gauge) and controlled by the ASTM E83 extensometer. Measurement error is $\pm 0.2\%$. The microstructures were examined by transmission

electron microscopy (TEM) using a Philips CM 200 operated at a nominal accelerating voltage of 200 kV. The specimens for TEM investigations were electrical machined from the samples as 3 mm discs with a plane normal to a sample axis, then mechanically thinned to 100 μm in thickness and finally prepared by double jet electrochemical polishing using Tenupol-5 at 283 K 12.5 V.

3. Results

The microstructures of Ni₄₉Fe₁₈Ga₂₇Co₆ (at%) single crystals after different thermal treatment (annealing at 1373 K followed by quenching; stress-free and stress-assisted ageing at 673 K for 4 h) have already been studied and are presented in [6,14,15]. The high-temperature phase in quenched crystals consists of *B2*-austenite and large γ -phase particles (*B2*-crystals). The precipitates of γ -phase possess fcc crystal structure and they are incoherent with the austenite *B2*-matrix. The precipitation size is 5–10 μm and the volume fraction is $\sim 7 (\pm 1)\%$. One-stage MT from *B2*-austenite into *L1₀*-martensite is observed in *B2*-crystals [6,14–17].

The subsequent ageing of the quenched *B2*-crystals results in, first, a change in the austenite crystal structure (*L2₁*-type ordering occurs). Second, the change of the sequence of MT takes place, and *L2₁-14M-L1₀* stress-induced MT is observed. Third, the coherent γ -phase particles are precipitated (*L2₁*-structure) with the size of 10–30 nm and a volume fraction of up to $\sim 3 (\pm 1)\%$. Thus, there is a bimodal distribution of dispersed particles in the aged single crystals in the form of large γ -phase particles and the nano-sized γ' -phase particles. During ageing, the four crystallographic equivalent variants of γ' -phase particles extended along the $\langle 111 \rangle_A$ direction are precipitated (*L2₁*-crystals). If stress-assisted ageing along the $[\bar{3}12]$ -direction is carried out, the γ' -phase particles oriented along one $[111]$ -direction are grown (*L2₁(σ)*-crystals) and the long-range stress fields are formed due to the sum of local stress fields from the particles.

It was experimentally shown that after thermomechanical treatments, without any additional training, quenched *B2*-crystals and stress-free aged *L2₁*-crystals do not possess TWSME; however, stress-assisted aged *L2₁(σ)*-crystals exhibit TWSME with the strain of 0.5%. This has been proven by experimental data shown in [6]. It is necessary to apply an additional training to increase the reversible strain of TWSME in *L2₁(σ)*-crystals because of the low volume fraction of γ' -phase particles. It is known that the high fraction (8–20%) of oriented dispersed particles in CoNiAl and TiNi single crystals after stress-assisted ageing in austenite induces TWSME with a strain of 1.7–2.0% without any additional training [5,8].

In the present work, the isothermal training at $T = A_f + (16 \div 23)$ K (under the conditions of SE existence) with 100 cycles of loading/unloading were carried out to induce SME in quenched, stress-free and stress-assisted aged [001]_A-oriented Ni₄₉Fe₁₈Ga₂₇Co₆ single crystals (Fig. 1). The maximum values of applied strain and stress for all studied crystals were 6% and 140 MPa, respectively.

Fig. 1 shows that in loading/unloading cycles in all crystals the degradation of SE is observed. An irreversible strain ϵ_{irr} increases, and the critical stress level of martensite formation σ_{cr} decreases as well as the stress hysteresis $\Delta\sigma$. The maximum degradation of the SE loop in all crystals is noticed after the first cycle. The first loading/unloading cycle is characterized by an irreversible strain, which is associated with dislocation formation and crystals of residual martensite, which is known from [17] and will be proven below. These factors result in the easy generation of stress-induced martensite, leading to the decrease of critical stress level of martensite formation in subsequent cycles. The most significant accumulation of the irreversible strain, up to 1.2%, is observed in quenched *B2*-crystals. On the one hand, the large γ -phase particles are the barriers for *L1₀*-martensite growth and the size of martensite crystals is limited by inter-particle distance. On the other hand, the interphase boundaries “matrix-particle” are the sources for the crystal formation of martensite. Such interaction of the different

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