Effect of B addition on the superelasticity in FeNiCoAlTa single crystals

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HIGHLIGHTS
• Fe28Ni17Co11.5Al2.5Ta0.05B single crystals oriented along [001] exhibit fully reversible superelastic strain up to 14.3%.
• B addition suppressed the formation of brittle β phase and reduced the average precipitate size of γ' to about 3–4 nm.
• Boron addition counters the initial effect of mechanical stabilization.
• A very different austenite/martensite interface as compared to thermally induced martensitic transformation was observed.

GRAPHICAL ABSTRACT

ABSTRACT
The study focuses on the superelastic effect in single-crystalline boron-doped Fe-based shape memory alloys. The homogenized and quenched single crystals were subjected to a heat treatment at 973 K for variable aging times. As a result, small and coherent nanometer-sized γ' (Ni3Al-type) precipitates were formed. It was established that Fe-28Ni-17Co-11.5Al-2.5Ta-0.05B single crystals oriented along [001] direction exhibit the fully reversible superelastic behavior up to 14.3% compression strain at 77 K reaching the maximum theoretical value. The boron addition suppressed completely the formation of the brittle β phase and reduced the average precipitate size of the γ' precipitates. Using high-energy synchrotron radiation and high-resolution transmission electron microscopy analysis the volume fraction and precipitate size of γ' were determined indicating that both factors are critical in obtaining the largest superelastic reversibility. Boron addition counters the initial effect of mechanical stabilization which was detected in single crystals without boron. Unlike the thermally induced martensitic transformation, applied stresses produce a different austenite/martensite interface composed of interchanging austenite and martensite variants. It is also demonstrated that upon loading/unloading cycles the moving transformation front divides the material into three district regions i.e. single variant of austenite, austenite intermixed with martensite and single variant of martensite.

1. Introduction

Shape memory alloys (SMAs) have been widely used in medical industries, smart technologies and applications because of their excellent shape recovery and superelastic properties [1–6]. Fe-based SMAs exhibit functional properties including high damping capacity, sensor
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and actuation properties [7–11]. They are also considered as potential cost effective substitution to NiTi SMAs especially when large sample masses are required. Nevertheless, the martensitic transformation is hard to observe in this system without nano-sized coherent γ precipitates [12–16]. The L12-type precipitates make the transformation reversible, strengthen the matrix and introduce short-range-order in the disordered matrix [17–22]. It appears that alongside the matrix strengthening, the principle function of the coherent γ precipitates is to generate stress/strain field in the matrix (if accommodated elastically). Small and coherent precipitates may serve as stress concentrators promoting the stress-induced martensitic transformation (SIMT) [22–26]. It is widely accepted that particles in the diameter range of 5 to 10 nm encourage SIMT in this system. However, the back SIMT is not always ensured. As shown in [21] a huge mechanical stabilization in Fe-28Ni-17Co-11.5Al-2.5Ta in at.% (NCAT) single crystals is established. A large mechanical stability is typically associated with a single martensite variant state and it is referred to as the shift of austenite start (As) temperate towards a higher temperature. On the micro-

structure level, the process involves formation of subtle interfacial twin elements (austenite/twinned martensite interface) that formation energies increase the nucleation barrier for austenite and thus increase As. In simple terms, the reverse SIMT requires twin nucleation and twin propagation which can be stimulated by precipitates. Therefore, a necessary prerequisite for achieving large superelastic strains in this system is a proper precipitation hardening which promotes forward and reverse SIMT [27–32]. Other requirements deal with proper texture and grain size of the material [33–39]. In our recent studies, the effect of one-step and two-step aging processes on the precipitation behavior in Fe-28Ni-17Co-11.5Al-2.5Ta in at.% (NCAT) single crystals was reported. The papers provided an overview of morphology, chemical composition, degree of coherence, and mechanical response of NCAT single crystals [21,40]. In this work the effect of boron addition in Fe-28Ni-17Co-11.5Al-2.5Ta-0.05B (NCATB) is studied. The addition of boron is known to suppress the formation of the brittle β phase at grain boundaries, therefore it is used in polycrystalline materials [41–50]. Nevertheless, the effect of boron on the superelastic strain and precipitation hardening in single crystalline materials is not fully understood. Another issue is a large mechanical stability [51–53]. This effect associated with the single variant state of martensite was shown in NCAT single crystals and other alloys [21,40]. By boron addition, it was possible to control this effect and to reduce the transformation hysteresis. The stabilization effect also allows to reveal the interface between austenite and martensite upon loading and unloading cycles. This provided more insight into the transformation mechanism and interface compatibility between both phases. Although the superelastic effect has been widely studied by many researches the exact transformation interface is still a subject of interest leaving some space for controversy. This becomes particularly important when the stress-strain asymmetry for compression and tension as well as the abnormal large reversible strains (higher than the theoretical transformation ones) are considered. Therefore, in this study quasi in-situ experiments were performed to reveal the transformation front and variant reorientation. As a result, a transition region composed of alternating austenite and martensite variants between single variant of austenite and single variant of martensite was unveiled.

2. Experimental procedure

The Fe-28Ni-17Co-11.5Al-2.5Ta-0.05B alloy was prepared by induction melting. Single crystals were grown by the Bridgman method [21,40]. The material was then solution-treated in a vacuum furnace at 1573 K for 48 h. Subsequently, the single crystal samples of [100] (001) orientation were cut into rectangular samples with dimensions of 3 × 3 × 10 mm3. In order to confirm the crystallographic orientation, the polished samples were investigated with a FEI Quanta 3D scanning electron microscope equipped with TSL EBSD system.

Then, the samples were sealed in quartz ampoules, annealed at 1573 K for 1 h followed by water quenching. The aging treatment was done at 973 K for variable times i.e. 0.5, 1, 5, 10, and 24 h. The same aging conditions were selected for NCAT single crystals. In order to define the volume fraction of individual phases X-ray diffraction analysis using high energy...
synchrotron radiation and single-crystalline samples were applied. The X-ray diffraction was done at Petra III DESY Hamburg synchrotron using beamline P07B (87.1 keV, \( \lambda = 0.0142342 \) nm) and 2D Mar345 Image Plate detector in the so-called continuous mode [21]. Transmission geometry significantly improved matrix/precipitate statistics (beam size 0.8 \( \times \) 0.8 mm\(^2\)). Furthermore, the samples were continuously rotated 180° around the \( \omega \)-axis (about 6° away from \( <100> \)) during the measurement, to get rid of the texture (orientation) effect [54,55]. Such a procedure enabled to obtain all diffraction reflections on one single image and it practically reduces the texture effect (even for single crystals). Aligning the \( \omega \)-axis parallel to the \( <100> \) direction would result in an overexposed intensity of (200) reflections. It is due to the fact that the Bragg diffraction condition for these planes would be satisfied over the total measuring time (18 s). Subsequently, 2D diffraction patterns were converted into 2Theta/Intensity profiles with Fit2D software.

The chemical and microstructure composition were also investigated by transmission electron microscopy (TEM) employing Tecnai G2 operating at 200 kV equipped with an energy dispersive X-ray

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Fig. 3. DF TEM microstructures and the corresponding selected area electron diffraction patterns, showing the size of \( \gamma' \) precipitates in the FeNiCoAlTaB single crystal aged at 973 K (a) for 0.5 h, (b) for 5 h and (c) 24 h. \( D_0 \) = average precipitate diameter [nm], SD – standard deviation of precipitate diameter [nm].
The growth rate of (NiAl-type, B2 structure) was detected \cite{21}. Similar to NCAT single crystals, the almost linear increase in the volume fraction, the precipitate size appears to reach a saturation value since the curve tends to become horizontal, Fig. 2a. This strongly indicates that the boron addition not only suppresses the formation of the brittle \( \gamma' \) phase at grain boundaries, but also slows down diffusion processes reducing the maximum size of the \( \gamma' \) precipitates. It seems that this behavior occurs due to interstitial positions of boron which limits the atomic diffusion. Using numerical diffraction pattern (fast Fourier transform (FFT)) by reconstruction from the high resolution (HRTEM) images, the evolution of diffraction patterns for single crystals subjected to different aging times is demonstrated in Fig. 4. Besides the diffraction pattern coming from the whole area (top row), this figure shows diffraction originating from two separated regions i.e. matrix and precipitate. An analogous analysis is shown in Fig. 5 for the initial state, 5 h, and 24 h of aging treatment.

From Fig. 4 can be also seen that with increasing aging time the intensity of (100) reflections, corresponding to the \( \gamma' \) phase, becomes more pronounced. It implies that apart from the increasing particle size also the chemical composition tends to a higher degree of order (compound crystal structure). The opposite relation is given by the tweed--like contrast in the \( \langle 110 \rangle \) direction manifested by diffusion streaks of (020) matrix reflections indicated by narrow on the reduced FFT for the initial case (Fig. 5). These diffuse streaks diminish with increasing aging time indicating that the precipitation process decreases supersaturation, releasing successively the solute atoms back into the matrix. Consequently, it leads to stress relaxation. These tweed patterns are also assigned with the premartensitic transformation phenomena due to local stress concentration and lattice instability \cite{56}. Therefore, on this level the precipitates can be treated as stress concentrators that promote martensitic transformation. Additionally, the HRTEM images and corresponding FFT and IFFT images in Fig. 5 show a good coherence between the matrix and \( \gamma' \) precipitates.

### 3. Results and discussion

Fig. 1 shows synchrotron X-ray diffraction patterns taken from the annealed NCATB samples of \( <001> \) orientation aged at 973 K for variable times. All samples exhibit only reflections coming from the austenitic matrix and the coherent \( \gamma' \) precipitates (Ni3Al-type). No \( \beta \) phase was observed even after 24 h of aging. This indicates a large change as compared to NCAT single crystals where a significant amount of \( \beta \) (NiAl-type, B2 structure) was detected \cite{21}. Similar to NCAT single crystals, the intensity of \( \gamma' \) reflections is found to increase with increasing annealing time. These changes can be clearly seen following the intensity of \( \langle 110 \rangle \gamma' \) and \( \langle 221 \rangle \gamma' \) reflections. However, as can be seen in Fig. 2 the growth rate of \( \gamma' \) in NCATB is significantly lower than for NCAT single crystals. To calculate the volume fraction of both phases the so-called continuum method and Rietveld refinement using the HighScore Plus software were applied \cite{54,55}. The obtained results are plotted in Fig. 2 where the amount of \( \gamma' \) increases almost linearly from about 2\% for the quenched sample to about 18\% after 24 h of aging treatment for NCATB single crystals. The growth rate of \( \gamma' \) precipitates in NCAT single crystals is significantly higher, however, falls as the \( \beta \) phase occurs (after 24 h).

The average diameter of the \( \gamma' \) precipitates in the single crystal aged at 973 K for 0.5 h was determined to be about 3 nm, Fig. 3. In principle, it is in the range of the critical size for nucleation which is within 2–3 nm. In this stage of precipitation, the precipitates shall stand out clearly against the final ones resembling more clusters of solute elements. Since in the initial (quenched) state the precipitate size is even smaller their determination has been technically impossible. Nevertheless, applying even very short aging times allows particles to grow to a size that can be resolved with TEM.

On further annealing, the precipitates continue to grow and reach a size of about 6.5 nm when the aging time increases to 24 h, Fig. 3. Unlike the almost linear increase in the volume fraction, the precipitate size appears to reach a saturation value since the curve tends to become horizontal, Fig. 2a. This strongly indicates that the boron addition not only suppresses the formation of the brittle \( \beta \) phase at grain boundaries, but also slows down diffusion processes reducing the maximum size of the \( \gamma' \) precipitates. It seems that this behavior occurs due to interstitial positions of boron which limits the atomic diffusion. Using numerical diffraction pattern (fast Fourier transform (FFT)) by reconstruction from the high resolution (HRTEM) images, the evolution of diffraction patterns for single crystals subjected to different aging times is demonstrated in Fig. 4. Besides the diffraction pattern coming from the whole area (top row), this figure shows diffraction originating from two separated regions i.e. matrix and precipitate. An analogous analysis is shown in Fig. 5 for the initial state, 5 h, and 24 h of aging treatment.

From Fig. 4 can be also seen that with increasing aging time the intensity of (100) reflections, corresponding to the \( \gamma' \) phase, becomes more pronounced. It implies that apart from the increasing particle size also the chemical composition tends to a higher degree of order (compound crystal structure). The opposite relation is given by the tweed--like contrast in the \( \langle 110 \rangle \) direction manifested by diffusion streaks of (020) matrix reflections indicated by narrow on the reduced FFT for the initial case (Fig. 5). These diffuse streaks diminish with increasing aging time indicating that the precipitation process decreases supersaturation, releasing successively the solute atoms back into the matrix. Consequently, it leads to stress relaxation. These tweed patterns are also assigned with the premartensitic transformation phenomena due to local stress concentration and lattice instability \cite{56}. Therefore, on this level the precipitates can be treated as stress concentrators that promote martensitic transformation. Additionally, the HRTEM images and corresponding FFT and IFFT images in Fig. 5 show a good coherence between the matrix and \( \gamma' \) precipitates.

| Time (h) | Initial | 0.5 h | 1 h | 5 h | 10 h | 24 h |
|---------|---------|-------|-----|-----|------|------|
|         | Reduced area FFT |         |     |     |      |      |
|         | Precipitate FFT |         |     |     |      |      |
|         | Matrix FFT |         |     |     |      |      |

**Fig. 4.** A series of FFT images for single crystals treated with variable aging time at 973 K.
Fig. 5. HRTEM image and corresponding FFT and IFFT images of the initial single crystal and aged at 973 K for 5 h and 24 h.
In the next step, the aged single crystals were subjected to compression tests. The tests were conducted using uniaxial compression at 77 K. Fig. 6 shows a set of stress-strain curves taken for the NCATB single crystals aged in the time range between 0.5 and 24 h. For comparison, they are presented along with those of NCAT single crystals. Generally, they yield a comparable superelastic strain of about 14.3% being in good agreement with the theoretical one [21]. However, the addition of a small amount of boron increases the stress for the onset of martensitic transformation from about 500 to 600 MPa for samples treated with short aging times (0.5 and 1 h). This can be correlated with the size and the amount of the γ’ precipitates that for NCAT single crystals treated with analogous aging times are considerably larger (3 nm and 2% for NCATB and 5.4 nm and 5.5% for NCAT aged for 0.5 h). On the other hand, the boron addition canceled out the mechanical stability yielding a fully reversible superelastic strain at 77 K. For NCAT single crystals aged for 0.5 and 1 h the strain was permanent at 77 K and went back if the samples were heated to about 123 K. The very abrupt back transformation for NCAT single crystals can be seen in [21], and in supplementary video files where the sample passively heated up leaps high into the air.

In principle, the stabilization effect refers to the twinning/detwinning process. To accommodate the transformation strain a complete stress-induced martensitic transformation produces a single martensite variant. This in turn, removes completely the initial austenite/twinned-martensite interfaces hindering austenite nucleation. This effect can be clearly seen in NCAT aged for 0.5 and 1 h, however with regards to NCATB is not observed. Thus, a proper particle size allows to control not only the forward but also the reverse SIMS. By inference, it seems that small precipitates (up to 4 nm) stabilize the austenite or facilitates twinning in a single variant martensite state while the larger ones promote forward transformation (stabilize martensite).

Another issue is the transformation hysteresis which significantly decreases as the size of γ’ precipitates decreases. The narrowest one can be observed for the NCATB single crystal aged for 0.5 h. This sample shows also the highest stress for the onset of martensitic transformation (618 MPa). Increasing the size and amount of γ’ decreases the onset stress to 585 MPa and 462 MPa for samples annealed for 1 h and 5 h, respectively. Thus, reducing the γ’ size sets perfect conditions for fully reversible superelastic strain, however at the same time it increases the stress for the onset of martensitic transformation.

Longer annealing times (10 h and 24 h) lead to a elasto-plastic response of the crystals. Nevertheless, the origin of this phenomenon is of a two-sided nature. For the NCAT single crystals, the permanent strain is derived from the plastic deformation of austenite. In this respect, the austenite deforms plastically without any phase transformation. On the other hand, the NCATB single crystals aged for longer times (10–24 h) transform to martensite. However as the stress is released, they do not go through a complete reverse transformation. It proves once again that larger precipitates promote the forward SIMT whereas the smaller ones the reverse transformation. Therefore, to obtain optimum superelastic properties a narrow process window for the heat treatment parameters has to be defined.

Fig. 6. Superelastic response of the [001] oriented NCAT/NCATB single crystal aged at 973 K for 0.5 h (a) for 1 h (b) for 5 h (c) for 24 h (d) under compression at 77 K.

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Fig. 7. BSE/SEM images showing the transformation front composed of alternate austenite and martensite variants in the NCAT single crystal aged for 0.5 h.

Fig. 8. Pole figures (a) demonstrating the orientation of particular variants with respect to Fig. 7 and to EBSD map (b). X-ray synchrotron diffraction pattern (d) taken from 3 different areas as indicated in (c).
The mechanical stabilization has also enabled the austenite/martensite interface to be visualized. Using the NCAT single crystal aged for 0.5 h the sample was first fully compressed at 77 K and then placed in a channel die to constrain the austenite back transformation. Subsequently, the sample was heated up to room temperature triggering the reverse transformation. The width of the channel die was selected in such a way that about half of the sample could transform back to austenite. For illustration, the sample arrangement is schematically shown in the inset of Fig. 7. In principle Fig. 7 shows a transformation front and a transition region composed of alternate martensite and austenite variants. This microstructure differs substantially from that typically observed at the austenite-twinned martensite interface where a clear interface between the single variant of austenite and multi-variant of martensite can be distinguished [53]. In the NCAT case, a very symmetric needle microstructure composed of austenite/martensite variants at the interface is created. Depending on the direction of applied force the interface moves towards the single variant of austenite or martensite. The crystallographic orientation of all variants is plotted in pole figures in Fig. 8a and corresponds to EBSS measurement, Fig. 8b.

The single martensite variant region has an orientation with the c-axis, the shortest lattice parameter expressed in the so-called parent-based coordinate system [56–61], parallel to the compression direction. This is also the only variant when the transformation is complete (upon compression). In the transition region, the austenite needles appear alternately with martensitic variants. However, in this region, the martensitic lamellae are always composed of two variants separated by a twin boundary with orientation plotted in Fig. 8a. Another observation is that areas close to the interface (marked with white dashed line in Fig. 7) are dominated by martensitic variants while moving away for the interface the austenite prevails successively. This can be clearly seen in Fig. 7 where the martensitic variant exhibits light contrast whereas the austenite is dark. The phase structure was also confirmed by X-ray synchrotron radiation taken in three different regions i.e. single variant of austenite, austenite intermixed with martensite, and single variant of martensite as illustrated in Fig. 8 c and d. These diffraction patterns validate the BSE/EBSD measurements but also show huge elastic stresses at the austenite/martensite interface related to peak broadening.

In principle, the illustration given in Fig. 7 shows how a single variant of martensite transforms to a single austenite variant. As already mentioned this transition significantly differs from that observed conventionally. It appears that the reason for it is the compressive nature of the test and a high twinning stress in this system. In this context, a different interface is expected upon tensile loading. Revealing the austenite/martensite interface upon tensile would shed more light on a district tension-compression asymmetry in the superelastic stress-strain response for these alloys [62–65].

4. Conclusions

In summary, it can be concluded that a small addition of boron completely suppressed the formation of the brittle $\beta$ phase in NCATB single crystals. It also reduced the average precipitate size of $\gamma$ precipitates to about 3–4 nm being beneficial for fully reversible superelastic behavior. Larger precipitates stabilize martensite structure giving rise to a huge mechanical stabilization. Diffraction of high energy synchrotron radiation and high-resolution TEM analysis show that besides the precipitate size also their volume fraction is critical for the complete and reversible transformation. Boron addition counters the initial effect of mechanical stabilization which was detected in single crystals without boron. As compared to thermally induced transformation, a very different austenite/martensite interface composed of interchanging austenite and martensite variants was detected. This observation reveals the transformation mechanism from a single variant of martensite to a single austenite variant. It was also presented that upon loading/unloading cycles the moving transformation front divides the single crystals into three district regions i.e. single variant of austenite, austenite intermixed with martensite and single variant of martensite. Supplementary data to this article can be found online at https://doi.org/10.1016/j.matdes.2020.109225.

Credit author statement

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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