# L10 Ordering in MnAl and FeNi Influenced by Magnetic Field and Strain

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# $L1_0$ Ordering in MnAl and FeNi Influenced by Magnetic Field and Strain

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Due to various materials supply chain challenges, magnets free of constrained elements are attracting increasing interest [1]. Magnetic materials such as rare-earth free FeNi and MnAl have been receiving considerable attention due to the high magneto-crystalline anisotropy and other associated magnetic properties derived from their unique chemically ordered tetragonal crystal structure, denoted as the $L1_0$ structure [2-4]. However, synthesis of $L1_0$ FeNi has had limited success due to the extremely low atomic mobilities of Fe and Ni [5]. In this work, isostructural MnAl was first studied as proxy to understand the $L1_0$ ordering process. Furthermore, evidence of $L1_0$ ordering in FeNi derived from TEM studies is presented, where ordering was facilitated by the application of strain and magnetic field provided during thermal treatment of a severely plastically deformed FeNi alloy.

The MnAl samples were synthesized by melt-spinning (MS) in an argon atmosphere to produce thin ribbons containing the non-equilibrium ϵ-phase (hexagonal close-packed structure), which is the precursor to the $L1_0$ phase. To induce chemical order, a sample was isothermally annealed at 390 °C for 15 minutes with the influence of a static magnetic field ($\mu_0 H = 600$ Oe) derived from two SmCo magnets that formed a closed flux path. Two FeNi ribbon specimens of 1 cm x 7 cm x 0.3 mm each were extracted from a cold-rolled (CR) sample with 93% strain. One of these samples, labeled as FSA ("field-strain annealed"), was further annealed in $N_2$ at 285 °C for 48 days [2]. During the annealing process, a tensile stress of 10 MPa and a magnetic field of 0.7 T were applied parallel to the rolling direction. A Carl Zeiss Auriga® 60 CrossBeam® focused ion beam tool and a scanning electron microscope (FIB/SEM) were used for imaging and for the preparation of TEM cross-sectional specimens. TEM imaging and energy-dispersive spectroscopy (EDS) were performed using a JEM-2010F (200 kV) system with an EDAX detector.

The TEM image in Figure 1a presents a cross-section view of FIB-milled, field-annealed MnAl ribbon. Selected-area electron diffraction (SAED) analysis reveals that two phases of MnAl coexist in this sample; these are the residual ϵ-phase and the ordered τ-phase ($L1_0$ structure). Figure 1b is a diffraction pattern obtained from the circled region in the image which shows a dark-contrast grain that contains τ-phase superlattice reflections. Although relatively large τ-phase grains with irregular grain boundaries are detected, Figure 1a, most τ-phase grains appear to be lamellar in shape and are alternatively distributed with the ϵ-phase lamellae, as shown in the area labeled as τ + ϵ in Figure 1c. This morphology is typical of a martensitic phase transformation, consistent with other observations in literature [4]. It is observed that this lamellar structure generally starts from an original ϵ-phase grain boundary or from an interface. Additionally, a higher density of τ-phase grains appears to be distributed in the region close to the initial quenched region of the ribbon, suggesting that residual strain arising from the melt spinning process may promote the heterogeneous nucleation of τ phase and the martensitic phase transformation.

TEM images of FIB-prepared cross-sections of the cold-rolled (CR) and field-strain annealed (FSA) FeNi specimens are shown in Figure 2a and 2b. A cold rolling texture {112}<111> consisting of elongated grains with approximate dimensions 2 μm x 200 nm, as shown in Figure 1a, was detected in the CR specimen. Recrystallization occurred in the FSA specimen (Figure 1b) under the synergistic effect of thermal energy (at a temperature well below the FeNi normal recrystallization temperature of 500-700 °C), *in-situ* strain and magnetic field. The new structure after dynamic recrystallization appears to retain the overall original grain texture donated by cold rolling but with a greatly reduced grain size of about 600 nm x 75 nm. The grains are still approximately parallel to each other. In addition, SAED analysis confirms the existence of the $L1_0$ phase as manifested by the superlattice reflections, indicated by arrows in Figure 2c. These results are consistent with recrystallization and with the fcc-to-$L1_0$ phase transformation promoted by the simultaneous application of uniaxial strain and magnetic field during annealing of a deformed bulk Fe-Ni-based alloy.

In conclusion, it is observed that the application of a magnetic field during heat treatment produces a large percentage of $L1_0$-structure grains in MnAl and promotes the $L1_0$ phase in FeNi. It is hypothesized that residual strain, perhaps originating from the melt-spinning synthesis process, enhances the phase transformation that forms the ordered τ-phase within the original ϵ-phase matrix during annealing. The cold-rolled FeNi sample possesses a crystallographic texture which appears to be beneficial to the phase transformation of fcc to $L1_0$. The $L1_0$ phase was detected in the partially recrystallized, textured microstructure. This recrystallized texture is largely preserved from the original cold-rolled crystal orientation and was produced by low temperature annealing with simultaneous application of longitudinal strain and magnetic field. The application of strain (unintentionally for MnAl) initiates the phase transformation to $L1_0$ which is further assisted by applying magnetic field in the parallel direction. Ongoing investigation includes *in-situ* TEM experiments to detect $L1_0$ nucleation and growth characteristics of MnAl and FeNi [6].

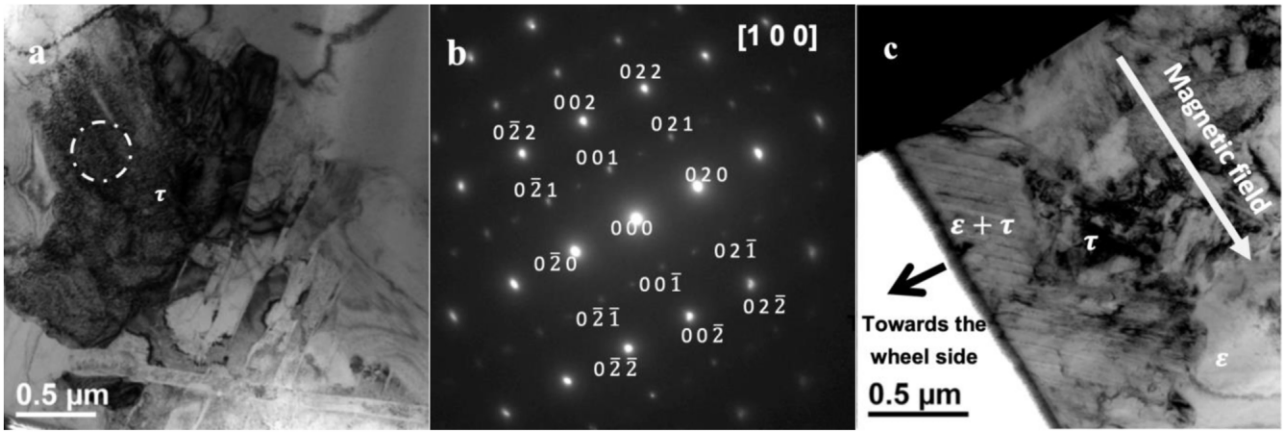


**Fig. 1.** a) TEM image of the cross-section of MnAl ribbon annealed with magnetic field. b) Selected area electron diffraction of the circled region in a, showing the super-lattice reflections from $L1_0$ phase. c) TEM image indicating different phases, their distribution and morphology.

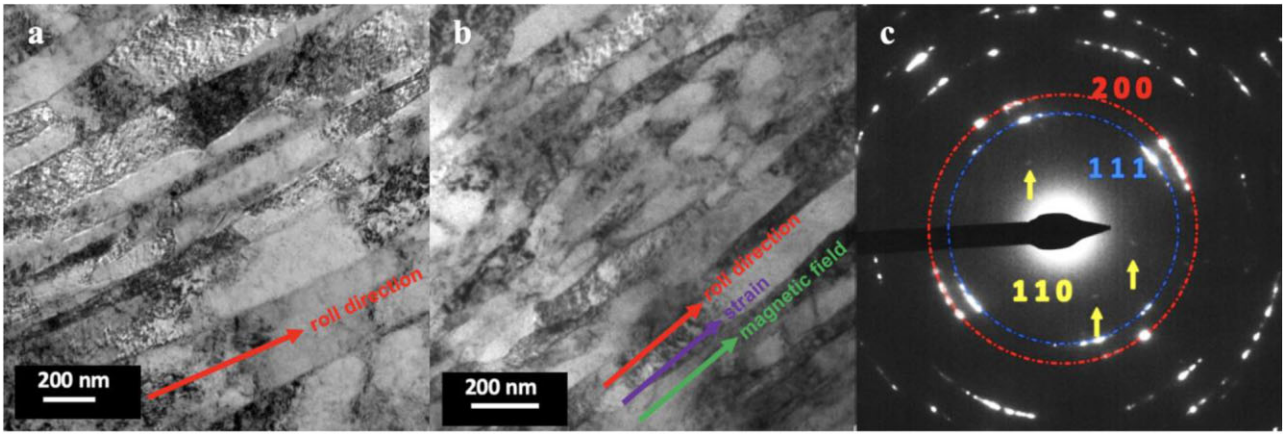


**Fig. 2.** TEM images of FIB-milled cross-sections of a) the CR ribbon, red arrow indicating rolling direction, b) the FSA ribbon, arrows indicating rolling direction, strain and magnetic field directions during thermal treatment. c) SAED in an area of FSA cross-section sample.

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