

## Direct Measurement of Reversible Magnetic-Field-Induced Strain in Ni-Mn-Co-In Metamagnetic Shape Memory Alloys

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### Abstract

Direct measurements of reversible magnetic-field-induced strain (MFIS) on a single crystalline Ni<sub>45</sub>Mn<sub>36.5</sub>Co<sub>5</sub>In<sub>13.5</sub> metamagnetic shape memory alloy were attained, for the first time, in the course of magnetic field-induced martensitic phase transformation using a custom designed micro magneto-thermo-mechanical testing system. MFIS levels were reported as a function of temperature, magnetic field and external bias stress. To be able to detect a notable MFIS, it was necessary to apply an external bias stress in these materials since magnetic field cannot favor a certain martensite variant. Fully recoverable transformation strains up to 3% were detected under repeated field applications in the presence of different compressive stress levels up to 125 MPa.

### 1. Introduction

Magnetic Shape Memory Alloys (MSMAs) are known to demonstrate some peculiar magneto-mechanical and magneto-thermal behaviors due to their coupling of magnetism and microstructure. Among these are the magnetic shape memory effect [1] and the giant magnetocaloric effect [2] such as in the Ni-Mn-Ga system. Although Ni-Mn-Ga can display a magnetic

field induced strain (MFIS) around 10% via martensite variant reorientation mechanism [3], its actuation stress remains at best around 10 MPa even with the contribution of size effect [4]. Ni-Mn-Ga system also demonstrates a magnetic field-induced martensitic phase transformation (FIPT) under stress levels on the order of 20 MPa accompanied by a cyclic MFIS of 0.5% [5].

Since its actuation stress is limited due to the limitation with the available magnetic energy to be converted into mechanical work, new alternatives have been sought for to replace the Ni-Mn-Ga system. Ni-Mn-X (X = In, Sn, Sb, Al) [6, 7] alloy families were introduced as promising alternatives where the replacement of Ga with In, Sb, Al or Sn in appropriate amounts results in the overlap of the first order magnetic and martensitic phase transformations and leads to weakly magnetic martensitic phases. In these new alloys, FIPT is observed as the main mechanism for the shape change between a ferromagnetic austenite and a paramagnetic/antiferromagnetic martensite for which the Zeeman energy serves as the driving magnetic energy source. This kind of magnetic FIPT is called as “*metamagnetic phase transition*” and was previously reported in other alloys systems such as Mn-As, Fe-Si-La, etc. [8] besides in

NiMnCoIn [9]. In the Ni-Mn-Ga system, on contrary to metamagnetic SMAs, the magnetocrystalline anisotropy energy (MAE) is responsible for MFIS. While the MAE is sensitive to crystal orientation and is limited with the saturation magnetization ( $M^S$ ) of martensite; the Zeeman energy is not as orientation sensitive and can continuously increase with the increasing applied field since it depends on the magnetization difference between austenite and martensite phases [10].

Addition of Co is found to increase the Curie temperature and thus, saturation magnetization of parent austenite and available Zeeman energy in Ni-Mn-In [9], Ni-Mn-Sn [13], and Ni-Mn-Al [14]. Among these Ni-Mn-Co-X alloys, NiMnCoIn is the easiest one to grow in single crystalline form resulting in a maximum transformation strain of about 6% along the [100] orientation under compression. In addition, it shows a moderate magnetic hysteresis (an average of 3 Tesla for the range of 150 to 250K) during the fully reversible FIPT. NiMnCoSn single crystals are difficult to grow and thus, NiMnCoSn only operates in polycrystalline form with a comparatively limited transformation performance. Since martensite in most of the metamagnetic SMAs has a tetragonal structure, intergranular brittleness is a major concern in polycrystals. Despite their large single crystalline forms, NiMnCoAl displays a large transformation hysteresis, hence is not desirable for applications.

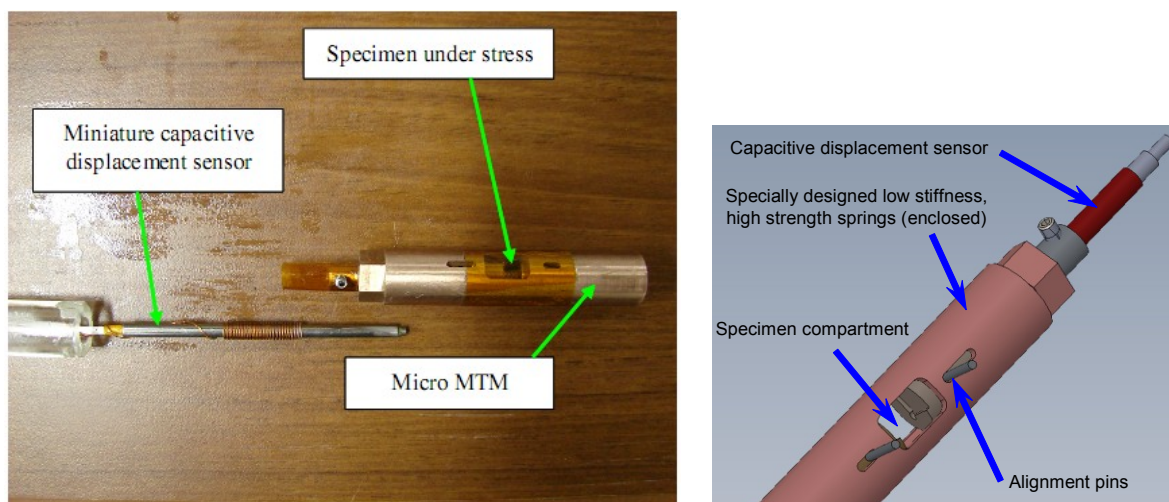
In Ni-Mn-Co-In, Kainuma *et al.* [9] have shown that 4 Tesla field is sufficient to recover 3% of pre-applied strain in martensite at room temperature. Their work stimulated several recent studies on metamagnetic SMAs [14-16]. Up to date, however, besides indirect evidences through magnetization vs. magnetic field [18], magnetization vs. temperature [18], electrical resistivity vs. magnetic field [19, 20] and DSC measurements [17, 18], there exists no report in

literature on reversible MFIS as a direct evidence of fully recoverable phase transformation in metamagnetic SMAs. In the present work, we report up to 3% fully recoverable MFIS under different stress levels up to 125 MPa bias stress through a direct measurement of shape change during the metamagnetic phase transition at different temperatures and magnetic field levels up to 18 Tesla.

Similar to temperature, magnetic field should not bias any specific martensite variant, and thus, result in large external strain, during the field-induced phase transformation in existing metamagnetic SMAs under magnetic field cycling. This is because of the fact that the MAE of martensite is negligible in the known metamagnetic SMAs including the present NiMnCoIn alloy and the martensite is weakly magnetic. Therefore, to be able to obtain reversible field-induced phase transformation accompanied with large MFIS, one needs to either built up internal local stress through thermo-mechanical or magneto-mechanical training, or a simultaneous stress, high enough to bias a variant, should be applied. From this point of view, a magnetic field in the present case is analogous to temperature, as both do not have a significant effect on the microstructure formation of martensite, but martensite transforms to austenite when they are increased, and austenite transforms back to martensite when they are reduced. As a result, it is of utmost importance to understand the effect of bias stress levels on the field-induced phase transformation during field cycling which is the main goal of the present work. Such a loading condition also constitutes what is used for many practical actuator applications.

## 2. Experimental

$Ni_{45}Mn_{36.5}Co_5In_{13.5}$  ingots were synthesized using vacuum induction melting. Single crystals were grown using the Bridgman technique in a He atmosphere. The composition of the single crystals



**Figure 1.** Custom built micro magneto-mechanical-testing system and the integrated 3mm diameter miniature-capacitive-displacement-sensor to fit in the 18 Tesla extraction type magnetometer are shown.

was determined as  $\text{Ni}_{45.7}\text{Mn}_{35.6}\text{Co}_{4.8}\text{In}_{13.8}$  using wavelength-dispersive spectroscopy [21]. The difference between the nominal and actual compositions is thought to be due to the Mn evaporation during single crystal growth [22]. The single crystal samples were then cut into rectangular prisms with dimensions of 2 mm x 2 mm x 4 mm using electro-discharge machining to assure that both magnetic field and stress can be applied along known crystallographic directions. In NiMnCoIn single crystals, the austenite orientations are used to describe the directions of the single crystal samples even if the sample might be in martensitic phase. [100] indicates the long axis of the rectangular prisms. After homogenization at 900 °C for 24 h in vacuum and water quenching, an additional heat treatment step of 500 °C for 1 h in vacuum was also employed to achieve martensitic transformation temperatures below room temperature and desired ordering in the samples.

The micro-magneto-thermo-mechanical testing system (micro MTM), integrated with a Capacitec miniature capacitive displacement sensor (both shown in Figure 1), was exclusively designed and manufactured for direct measurements of MFIS in the course of reversible phase transformation. This gadget consists of precipitation hardened



**Figure 2.** 18 Tesla extraction type magnetometer in the High Field Laboratory for Superconducting Materials, Institute for Materials Research, Tohoku University, Japan.

nonmagnetic Cu-Be body and inner components. It can apply compressive stress on specimens utilizing SS-302 type Belleville springs which are driven by a simple screw mechanism. The level of applied stress remains nearly constant with small variations during phase transformation. The outer diameter of

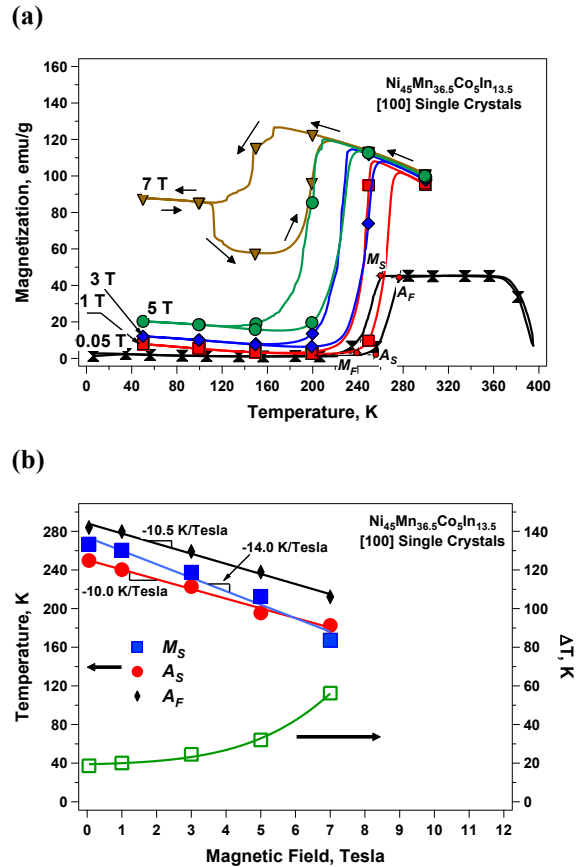
the micro MTM is 10 mm with a total length of 50 mm.

The micro MTM includes a miniature (OD 3mm) capacitive sensor for the measurement of transformation strains during magnetic FIPT. This sensor is capable of measuring the displacement with an accuracy of  $\pm 0.0001$  mm at temperatures as low as 4.2 K and under magnetic fields as high as 18 Tesla.

The integrated testing system was utilized in a custom design extraction type superconducting magnet with a magnetometer (shown in Figure 2) to obtain magnetization and MFIS behavior of NiMnCoIn specimens under a cycled magnetic field between 0 to 18 Tesla at test temperatures ranging from 4.2 to 250 K.

### 3. Results and Discussion

Figure 3.a shows the change in magnetization as a function of temperature under different magnetic field levels obtained from the magnetization vs. temperature (M-T) curves measured using a SQUID magnetometer. Under 0.05 T, the austenite to martensite transformation started at 260 K ( $M_S$ : martensite start temperature) and finished at 238 K ( $M_F$ : martensite finish temperature) upon cooling. The reverse transformation started at 254 K ( $A_S$ : austenite start) and finished at around 277 K ( $A_F$ : austenite finish) upon heating. The transformation is reversible with a small thermal hysteresis ( $\sim 20$  K). As the applied magnetic field increased, the transformation temperatures were reduced; e.g.,  $M_S$  decreased from 260 to 168 K as the field increased from 0.05 to 7 T. This is due to the fact that the applied magnetic field favors the phase with the higher saturation magnetization (austenite in this case). Additional undercooling is needed to supply the required chemical energy to overcome the magnetic energy opposing the forward phase transformation. Figure 3.b shows the change in  $A_S$ ,  $A_F$ , and  $M_S$  as a function of magnetic field, extracted from the experiments shown in Figure 3.a. The



**Figure 3.** (a) Magnetization vs. temperature response of  $\text{Ni}_{45}\text{Mn}_{36.5}\text{Co}_5\text{In}_{13.5}$  single crystals under different constant applied magnetic field levels and (b) the transformation temperatures and thermal hysteresis as a function of these field levels extracted from (a).

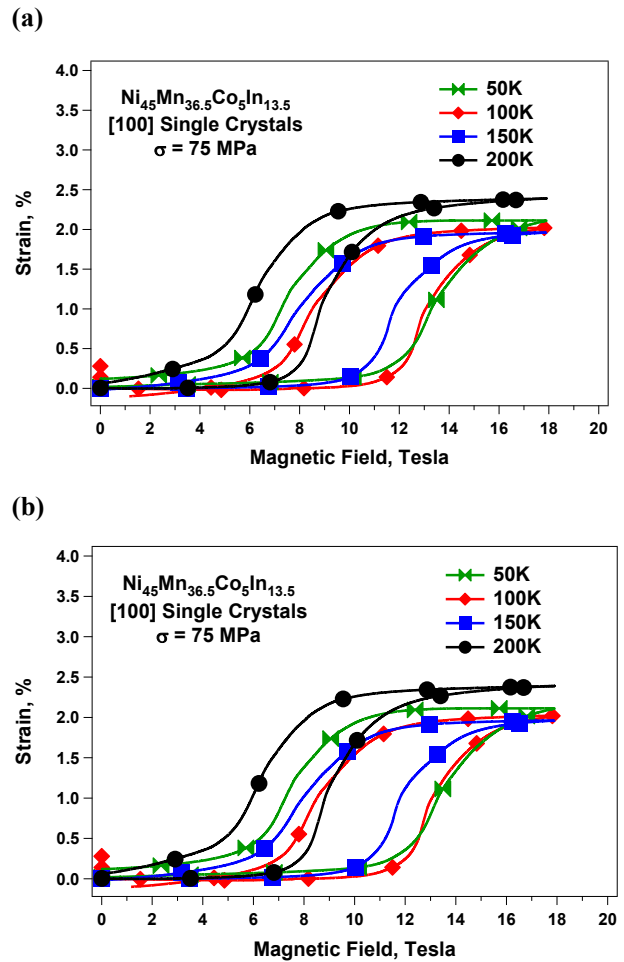
level of change in  $A_S$  as a function of bias field is approximately 10.0 K/Tesla. Other transformation temperatures show similar trend as  $A_S$ .  $M_S$  and  $A_F$  decrease with magnetic field with the rates of 14.0 K/Tesla and -10.5 K/Tesla, respectively. Suppression of the transformation temperatures and the separation of the M-T curves under different field levels is an indication of the possibility of reversible FIPT in the metamagnetic SMAs [6].

The reason for the shift in transformation temperatures is the requirement of additional undercooling which in turn supplies the required chemical energy to overcome the magnetic energy opposing the forward phase transformation since the applied magnetic field favors austenite. The

variation in saturation magnetization of martensite for each curve under different levels of applied field is also noteworthy in Figure 3.a. The reason for this variation is the fact that upon cooling under relatively high magnetic fields, the forward transformation is not completed even though the temperature reaches much below the “apparent”  $M_f$ . We have confirmed the existence of austenite at very low temperatures using neutron diffraction measurements when cooled under high magnetic fields (not shown here). This phenomenon is known as kinetic arrest of martensitic transformation, reason of which is not clear at this point [23]. Along with the certain fraction of kinetically arrested austenite, the transformation temperature hysteresis also increases with increasing applied field as shown in Figure 3.b. However, it is not obvious whether this increase in hysteresis is due to kinetic arrest of austenite or low temperature levels increasing the lattice friction, or even due to high magnetic field levels changing the compatibility between austenite and martensite thru magnetostriction and thus, transformation hysteresis.

Figures 4.a and 4.b demonstrate MFIS response of the NiMnCoIn single crystalline specimen as a function of magnetic field at various test temperatures and under two different compressive bias stress levels, i.e. 75 and 125 MPa, respectively. These results provide the direct evidence of reversible MFIS utilizing FIPT mechanism, for the first time in literature. It should be noted that under zero stress level, there was no notable MFIS levels observed supporting our initial argument on the inability of magnetic field biasing a particular martensite variant. In Figure 4, as the test temperature decreases, all the critical magnetic field values required to start and finish reverse

$(H^{A_s}, H^{A_f})$  and forward  $(H^{M_s}, H^{M_f})$



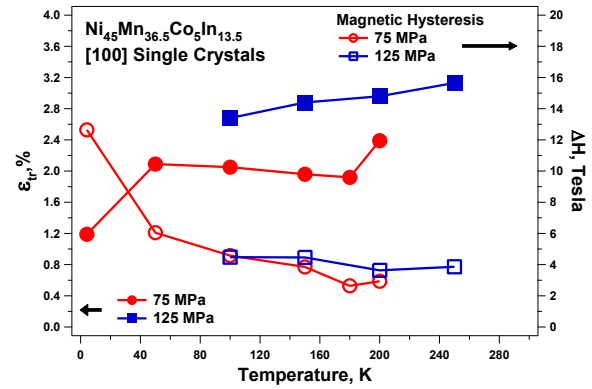
**Figure 4.** Magnetic field induced strain vs. applied magnetic field response of Ni<sub>45</sub>Mn<sub>36.5</sub>Co<sub>5</sub>In<sub>13.5</sub> single crystals oriented along the [100] direction under (a) 75 MPa and (b) 125 MPa compressive bias stress at different test temperatures in the course of metamagnetic phase transition.

transformations are shifted towards higher field values since martensite is more stable at low temperatures and more magnetic energy is needed to reach the energy level of austenite. The sense of this shift is similar to the increase in transformation temperatures in conventional SMAs under constant stress, i.e. the higher the stress levels are applied, the more the transformation temperatures and critical magnetic fields shifts to higher levels.



The maximum MFIS achieved in this study was 3.13% at 250 K under 125 MPa with an irrecoverable strain of 0.26%. The reason for the irrecoverable strain is the incomplete forward transformation because 250 K is higher than the  $M_f$  temperature under zero field. Fully recoverable MFIS values were attained as 2.92% and 2.39% at 200K under 125 and 75 MPa, respectively. Figure 5 was constructed using the results in Figure 4 and it depicts the trend in transformation strain and magnetic transformation hysteresis as a function of bias stress. The strain levels observed here is smaller than what we expect from this material theoretically, i.e. approximately 6.5% [9], if the austenite has  $L2_1$  and the martensite has six layered monoclinic structure. However, in the present crystal, the austenite could be either B2 or  $L2_1$  and the martensite can be a mixture of  $L1_0$  and six-layered monoclinic structure according to the magnetization results in Fig. 3.a and following the recent study by Ito *et al.* [24] on the different structure of the transforming phases depending on the order heat treatments. Therefore, one of the reasons for the lower transformation strains than expected from our previous work [9] might be due to the different heat treatment in the present crystal. To validate this argument, the structures of the transforming phases will be determined in the near future.

In our previous work on the isobaric thermal cycling experiments of the same material, we reported the transformation strain to increase with bias stress and saturate above a certain stress level before it starts decreasing under high stress levels due to simultaneous plasticity [21]. Such changes in transformation strain are a consequence of the evolution of martensite variants as a function of applied stress. During thermal or magnetic cycling under low stresses, the measured lower transformation strain levels imply that the applied stress may not be sufficient to bias a single variant martensitic structure; therefore a



**Figure 5.** Magnetic field-induced strain levels and magnetic transformation hysteresis in  $Ni_{45}Mn_{36.5}Co_5In_{13.5}$  single crystals oriented along the [100] direction as a function of temperature under 75 MPa and 125 MPa compressive bias stresses.

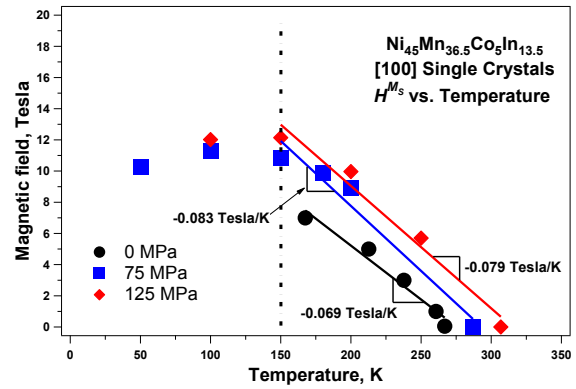
self-accommodating martensite structure may partially form. Difficulty of biasing a single variant martensite at low stress levels depends on the presence of second-phase particles and defect generation during phase transformation which is a direct consequence of lattice incompatibility between austenite and martensite phases. From the significant increase in the transformation strain (Figure 5) when the stress increased from 75 to 125 MPa, it is reasonable to assume that maximum transformation strain and single variant martensite morphology will be reached above much higher stress levels. These stress levels are significantly larger than 6 MPa required for reaching maximum transformation strain in  $Ni_2MnGa$  [21]. Such a large difference in the saturation stress can originate from the second phase particles in the  $NiMnCoIn$  samples [22] and the higher lattice friction in  $NiMnCoIn$  alloys due to solid solution hardening and off-stoichiometry which in turn yields more defect generation during transformation. For the alloy used in this study, the composition of the matrix and the second phase were determined as  $Ni_{45.7}Mn_{35.6}Co_{4.8}In_{13.8}$  and  $Ni_{42.0}Mn_{40.3}Co_{16.0}In_{1.6}$ , respectively using WDS [22].

The small variations in the MFIS levels under

each bias stress at different test temperatures (Figure 4) can be attributed to several different reasons: (1) a small change in the spring constant of the spring used in the micro MTM as a function of temperature, which in turn affects the level of stress, (2) the effect of temperature on the lattice parameters of austenite and martensite, thus on the transformation strain, (3) the effect of temperature dependent magnetostriction on lattice parameters of austenite and martensite, and (4) incomplete reverse transformation even under 18 T at low temperatures and under 125 MPa.

As shown in Figure 5, the magnetic transformation hysteresis remains almost constant around 4 - 4.5 Tesla in the 125 MPa experiments for a temperature range of 100 K to 250 K, whereas it slowly declines under 75 MPa from 4.5 Tesla to 2.5 Tesla for the same temperature range. In the entire temperature range investigated, magnetic transformation hysteresis increases with decreasing temperature under 75 MPa. Applied stress also increases the field hysteresis at a given temperature (not shown here). This trend is opposite of what we observed during isobaric thermal cycling experiments where the transformation thermal hysteresis diminishes with increasing bias stress [22]. Since transformation hysteresis is a measure of compatibility between the transforming phases and energy dissipation during the transformation, this discrepancy in the hysteresis trends is attributed to temperature dependent lattice friction and change in compatibility. It is well-known that Peierls-Nabarro stress is very high in BCC like structures, and at low temperatures this stress governs the lattice friction. Therefore, since magnetic field suppresses transformation temperatures, the phase transformation occurs at low temperatures, and the phase front motion has to move in the presence of high lattice friction causing large dissipation.

From thermal cycling and pseudoelastic (PE) experiments on similar NiMnCoIn crystals, the rate

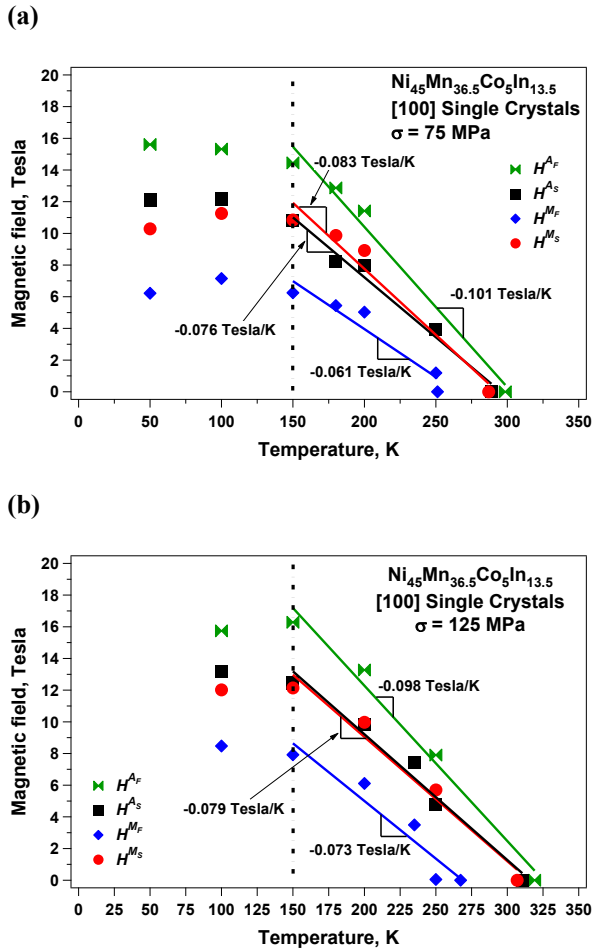


**Figure 6.** Magnetic field vs. temperature phase diagram of  $\text{Ni}_{45}\text{Mn}_{36.5}\text{Co}_5\text{In}_{13.5}$  single crystals oriented along the [100] direction under three stress levels, 0, 75, and 125 MPa.

of change in the critical stress for the onset of martensitic transformation (i.e.  $\sigma^M$ ) was determined to be  $\frac{d\sigma}{dT} = 2.1 \text{ MPa} / \text{K}$  under zero magnetic field [21]. From the magnetization response of the same material, the rate of change in  $M_S$  as a function of applied magnetic field is determined to be  $\frac{dT}{dH} = -14.0 \text{ K} / \text{Tesla}$  (Figure

3.b). Multiplying these two values gives us the shift in critical magnetic field required to start forward transformation as a function of applied bias stress as  $\frac{dH}{d\sigma} = -0.034 \text{ Tesla} / \text{MPa}$ . Figure 6 shows the

change in critical magnetic field levels as a function of temperature under the influence of bias stress. In order to verify the increase in the critical field level for the onset of forward transformation as a function of stress, we can compare the predicted value,  $-0.034 \text{ Tesla} / \text{MPa}$ , with the experimental results presented in Figure 6. According to this prediction, there should be 2.55 Tesla difference between the 0 and 75 MPa curves. Indeed, the measured differences are 2.6 Tesla at 200 K and 2.8 Tesla at 175 K. Since these two values are close to the prediction, we can conclude that the results from the magnetization and conventional isobaric thermal cycling or pseudoelastic experiments can



**Figure 7.** Magnetic field vs. temperature phase diagram of  $\text{Ni}_{45}\text{Mn}_{36.5}\text{Co}_5\text{In}_{13.5}$  single crystals oriented along the [100] direction under (a) 75 and (b) 125 MPa compressive bias stress.

be used to roughly predict the change in required magnetic field values for the onset of transformation under different bias stress levels. If we use this simple approach, then it is possible to estimate the approximate value for the real stress level on the sample under 125 MPa at room temperature, which should be around 115 MPa at 200 K due mainly to the change in the spring constant with temperature and due to the transformation strain relaxing the spring force.

Figures 7.a and 7.b show the magnetic field vs. temperature phase diagram under 75 and 125 MPa bias stress levels, respectively. It is clearly observed that for both stress levels, the change in critical

field values with temperature follow a linear trend down to 150 K. However, at temperatures lower than 150 K, this linear trend ceases and the field levels changes only slightly with reduction in temperature. The equilibrium magnetic field,  $H_0$ , where Gibbs free energies of austenite and martensite phases are equal, can be defined as the arithmetic mean of  $H^{M_s}$  and  $H^{A_F}$ .  $H_0$  gradually increases with decreasing temperature and saturates at temperatures lower than 150 K, while magnetic hysteresis continuously increases below 150 K. If a bias stress is applied, both  $H_0$  and  $\Delta H$  curves shift upwards to higher magnetic field values, indicating that stress makes it harder for reverse transformation to take place. Umetsu *et al.* [25] explained the aforementioned trends in  $H_0$  and  $\Delta H$  curves by the diminishing transformation entropy during forward transformation which in turn yielding kinetic arrest of austenite making it thermodynamically more stable than martensite during cooling under magnetic field. The reason for the kinetic arrest is not clearly known at this point.

#### 4. Summary

Direct measurement of magnetic field induced strains during metamagnetic phase transition was attained in a metamagnetic SMA, for the first time in literature. Magnetization vs. temperature behavior of  $\text{Ni}_{45}\text{Mn}_{36.5}\text{Co}_5\text{In}_{13.5}$  single crystals oriented along the [100] direction were reported with and without bias stress at various temperatures. The effect of bias stress on the critical magnetic field levels for the forward and reverse phase transformations were determined. Up to 3% fully recoverable MFIS was confirmed at 125 MPa.

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