Magnetic glass in shape memory alloy: Ni$_{45}$Co$_5$Mn$_{38}$Sn$_{12}$

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Abstract

The first order martensitic transition in the ferromagnetic shape memory alloy Ni$_{45}$Co$_5$Mn$_{38}$Sn$_{12}$ is also a magnetic transition and has a large field induced effect. While cooling in the presence of a field this first order magnetic martensite transition is kinetically arrested. Depending on the cooling field, a fraction of the arrested ferromagnetic austenite phase persists down to the lowest temperature as a magnetic glassy state, similar to the one observed in various intermetallic alloys and in half doped manganites. A detailed investigation of this first order ferromagnetic austenite (FM-A) to low magnetization martensite (LM-M) state transition as a function of temperature and field has been carried out by magnetization measurements. Extensive cooling and heating in unequal field (CHUF) measurements and a novel field cooled protocol for isothermal $MH$ measurements (FC–$MH$) are utilized to investigate the glass like arrested states and show a reverse martensite transition. Finally, we determine a field–temperature ($HT$) phase diagram of Ni$_{45}$Co$_5$Mn$_{38}$Sn$_{12}$ from various magnetization measurements which brings out the regions where thermodynamic and metastable states coexist in the $HT$ space, clearly depicting this system as a ‘magnetic glass’.

(Some figures may appear in colour only in the online journal)

1. Introduction

The kinetics of any first order phase transition (FOPT) is governed by the time required to extract the latent heat of the system and can be arrested by rapid enough cooling [1]. When a magnetic first order transition is kinetically arrested it gives rise to states which are magnetically ordered metastable states and are referred to as ‘magnetic glasses’ [2].

Kinetic arrest of the martensite transition (MT) in ferromagnetic shape memory alloys (FSMAs) has been reported extensively in recent years, especially in NiMnIn and NiMnSn based FSMAs [3–22]. Apart from the technological importance of FSMAs, they are also very good candidates for fundamental studies in the field–temperature ($HT$) phase space since the first order martensite transition is influenced by both field and temperature. The parent austenite phase shows a stronger ferromagnetism than that of the martensite phase and at the martensitic transition temperature ($T_m$) a sharp change in magnetization is observed in thermomagnetization measurements. $T_m$ decreases on increasing the magnetic field of measurement. The kinetics associated with magnetic FOPT gets hindered in many of these materials resulting in phase coexisting states at low temperature which comprise of an arrested (metastable) state and the transformed (stable) state. In spite of the wide ranging studies justifying the presence of kinetic arrest of martensite transformation in FSMAs, the magnetic glass concept has been discussed in only a few In and Sn based FSMAs [18–22].

In general, magnetic field may enhance or reduce the kinetic arrest, depending on whether the low temperature equilibrium state has a lower or higher value of magnetization [23, 24]. It has been noted that by cooling in a certain field ($H_c$) and then heating in a different field ($H_w$), this glass like arrested state (GLAS) can be de-arrested and on
further heating a reverse magnetic transition is observed. Hence by cooling and heating in unequal fields (CHUF), a re-entrant transition can be seen but only for the appropriate sign of \((H_c - H_w)\) [24]. In the case of FSMAs, since the high temperature phase is ferromagnetic austenite (FM-A) while the low temperature phase is low magnetization martensite (LM-M), this re-entrant transition will be seen only for a positive sign of \((H_c - H_w)\). Kainuma’s group has reported magnetization measurements on cooling in various fields and warming in lower fields \((0.05 \text{ T})\) on Ni_{50}Mn_{34}In_{16} alloy and observed the ‘unfreezing of the P + M coexisting state’ [14]. This ‘de-arrest’ stated as ‘unfreezing’ is analogous to the ‘devitrification’ observed in manganites with charge ordered ground states [25–27]. These results are consistent with what is expected in a magnetic glass; however, the well established CHUF protocol provides a crucial test for validating whether the kinetically arrested state corresponds to a magnetic glass. The CHUF protocol requires that the measurements are performed in fields both below and above \(H_c\), i.e. for both positive and negative values of \((H_c - H_w)\).

In this paper our studies on a Ni_{43}Co_{35}Mn_{38}Sn_{12} ribbon sample demonstrate all the features crucial for unambiguously proving the magnetic glass state by various thermomagnetization measurements, including CHUF measurements. Besides, we highlight an important aspect, namely that the transition between LM-M and FM-A phases cannot be achieved at low temperature by the conventional protocol of isothermal field variation of the ZFC state because of the limit of experimentally accessible field range. By following the novel protocol of field cooling and then isothermally reducing the field we are able to observe the transition between LM-M and FM-A phases within the accessible field range even at the lowest temperature. We justify this by constructing the qualitative phase diagram identifying bands of supercooling (SC), superheating (SH) and kinetic arrest (KA) from the variety of measurements spanning the HT space and check its self-consistency. As has been discussed in detail in [23] the SC limit \(T^{\ast}(H)\), the SH limit \(T^{\ast\ast}(H)\) and the kinetic arrest line \(T_k(H)\) are broadened into bands in systems with quenched disorder. Thus we show that the technologically useful FSMAs are magnetic glasses, as shown earlier in manganites.

2. Experimental details

The Ni_{43}Co_{35}Mn_{38}Sn_{12} (Sn12) sample used in this study was prepared by arc melting the high purity elements into buttons and preparing ribbons from these buttons by melt spinning in an inert atmosphere. The composition and crystal structure were determined by an energy dispersive x-ray spectrometer (EDXS) attached to a scanning electron microscope (SEM) and powder x-ray diffraction (XRD). Details of sample preparation and characterization can be seen in [20]. The DC magnetization was measured with a commercial 14 T PPMS-VSM (manufacturer Quantum Design).

3. Results and discussion

The Sn12 sample exhibits a clear first order phase transition with respect to field and temperature, displaying broad hysteresis. The high magnetization ferromagnetic austenite (FM-A) phase transforms to the low magnetization martensite (LM-M) phase and vice versa on cooling and heating, respectively [20, 21]. The first order nature of the martensite transition with respect to field in this sample was established in our earlier work [20] by performing zero field cooled magnetization versus field \((MH)\) measurements during cooling and heating [28]. The \(MH\) isotherms in both the cases (cooling and heating) were completely different for \((T^* \sim 75 \text{ K}) < T < (T^{\ast\ast} \sim 215 \text{ K})\). Similarly, in the plot of magnetization versus temperature i.e. \(M(T)\), a broad hysteresis is observed in the same temperature range during cooling and heating. These results signify that the fractions of coexisting metastable martensite and austenite phases not only vary with field and temperature but also depend on the path followed in the HT space. These metastable states at low temperature are governed by an interplay of supercooled and kinetically arrested states, because neither the supercooled nor the arrested state is the equilibrium state of the system [29].

Figure 1 shows the magnetization as a function of temperature in three different measurement protocols, namely zero field cooled warming (ZFC), field cooled warming (FCW) and field cooled warming (FCW) at 1, 3, 5 and 9 T. The FCC and FCW curves shown here were reported in [20, 21]. They are reproduced here in a replotted version along with the ZFC curve to explain the thermomagnetic irreversibility in these systems at low temperatures. As shown in figure 1(a), the cooling and heating curves at 1 T exhibit a clear first order phase transition from the FM-A phase to the LM-M phase, showing a broad hysteresis in the range of \((T^* \sim 75 \text{ K}) < T < (T^{\ast\ast} \sim 215 \text{ K})\). At higher fields the MT gets gradually inhibited and completely arrested at 9 T, as shown in figures 1(b)–(d). As a result, various coexisting metastable states are observed, depending on the magnitude of the magnetic field during cooling and heating. These ‘kinetically arrested states’ are due to a slowing down of the growth of the martensite phase from the supercooled austenite phase, similar to the viscous retardation in structural glasses. In structural glasses the liquid to crystal freezing is arrested, while in magnetic glasses the magnetic re-ordering is arrested.

In both cases the material is cooled through the first order transition temperature without extracting latent heat. While in a jamming process the translational kinetics is arrested, in the formation of both these glasses the specific heat is extracted, while the latent heat (which has a different coupling with the thermal conduction process) cannot be extracted [1]. In both cases the higher entropy phase persists down to the lowest temperature [30]. In this sense the magnetic glass and the structural glass address similar physics. However, in structural glasses the pressure is the variable parameter. The experimental advantage of magnetic field \((H)\) over pressure \((P)\) is that it can be easily varied reversibly without a control medium, and its variation does not interfere with temperature control.
As the first order MT gets arrested in the presence of a field, the low temperature states turn out to be non-ergodic. At 1 T, there is no significant difference between the ZFC and FCW curves as shown in figure 1(a). However, at fields above 1 T, the hysteresis width $\Delta T$ does not change significantly but the area enclosed by the hysteresis reduces and the difference between $M_{ZFC}$ and $M_{FCW}$ at low temperature increases. This difference is known as thermomagnetic irreversibility (TMI), which is defined as $TMI = M_{FCW} - M_{ZFC}$, at 5 K. The TMI at low temperatures could be due to spin glass behavior, pinning of variants, hindrance of domain motion or glass like arrest of dynamics. Nevertheless, for the first three cases, this irreversibility decreases with increasing field; hence the ZFC and FCW magnetization curves would merge, in contrast to the present case. Here, the TMI increases with increase in field as shown in the inset of figure 1(d). This rise in thermomagnetic irreversibility is due to the kinetic arrest of the martensite phase transition at low temperatures which gives rise to non-ergodic states having coexisting FM-A and LM-M phases. We have almost 100% LM-M phase fraction when cooled in zero field but on increasing the cooling field the ferromagnetic austenite fraction increases. We have estimated this phase fraction by assuming the field cooled magnetization value at 5 K and 9 T as 100% FM-A as there is no FM-A to LM-M transition on heating and cooling at this field [21]. The TMI at 5 K gives the amount of arrested FM-A mixed with the LM-M phase. This attribute classifies this system as a magnetic glass.

This system undergoes a complete transformation below 1 T ($H_1$), while above 8 T ($H_2$) the martensite transformation is almost completely hindered resulting in a completely austenite phase at low temperature. However at field values between $H_1$ and $H_2$, we achieve a fraction of transformed martensite phase along with an arrested austenite phase at low temperatures [21]. In the earlier studies of manganites and intermetallic alloys [25–28, 33], referred to as magnetic glasses, the arrest of kinetics across the first order phase transformation was established from the fact that the virgin $MH$ curve lies outside the envelope curve in the isothermal magnetization curves at the lowest temperatures. In these cases, the samples have undergone a first order transformation within the available field and temperature range and the observed low temperature anomaly is inferred as the arrested kinetics of the first order transition. In all these cases, the virgin states considered are the zero field cooled states from well above the superheating limits of the samples. They have also shown a non-monotonic behavior of the metamagnetic transition field with respect to temperature in the zero field cooled $MH$ isotherms, which is an important experimental method of studying the arrested kinetics in these samples.

We performed similar zero field cooled isothermal magnetization (ZFC–$MH$) experiments in the range of 5–150 K, as shown by the symbols (green) in figures 2(a)–(h). The sample is cooled from temperatures well above the MT temperature. After cooling in zero field, the low temperature state of the sample is in a low magnetization martensite

**Figure 1.** (a)–(d) $M$ versus $T$ plots for the Sn12 sample obtained in the ZFC, FCC and FCW modes in applied fields of $H = 1, 3, 5$ and 9 T respectively. The FCC and FCW data are taken from [20, 21].
Figure 2. (a)–(h) The isothermal magnetization (solid red lines) at various temperatures from 5 to 150 K after cooling the sample in 8 T from 350 K to the respective temperature after raising the field to 12 T. For comparison the zero field cooled $M$ versus $H$ curves (green symbols) are shown along with the field cooled data. The inset to panel (h) shows the difference in magnetization for FC–MH and ZFC–MH at 8 T for various temperatures obtained from (a)–(h). (i) Schematic diagram showing the path of the 8 T field cooled (green, X) and zero field cooled (blue, Y) MH isotherms.

(LM-M) phase, as seen by the dashed lines in figure 2(a). The initial field increasing cycle at 5, 15 and 25 K shows a sharp increase in magnetization up to $\sim$1 T and then almost saturates, while the return cycle follows the same path indicating the soft ferromagnetic behavior. However, we did not observe the reverse martensitic transition (RMT) at low temperatures up to 14 T. For temperatures above 75 K, the rise in magnetization in the forward field cycle followed by an opening of hysteresis indicates the onset of the martensite to austenite phase transformation, which confirms the RMT in the temperature range of 75–150 K. Therefore, to investigate the absence of such an RMT at low temperature, which we attribute to kinetic arrest in this system, we designed a novel protocol of field cooled $MH$ isotherms (FC–MH), as shown by the solid lines (red) in figures 2(a)–(h). For FC–MH measurements, we cooled the sample in 8 T from 350 K, which is well above the superheating limit ($\sim$210 K) of the sample, and considered the field cooled state as the initial state of the sample. On cooling in 8 T, the sample acquires a maximum fraction of FM-A phase as an arrested phase. In each case, the sample is cooled in 8 T to the respective temperature, and then the magnetization is measured while isothermally raising the field up to 12 T, subsequently cycling from $12 T \rightarrow 0 \rightarrow (–12 T) \rightarrow 0 T$.

We explain these measurements by the schematic band diagram specified for magnetic glasses shown in figure 2(i). Since the FOPT (here MT) induced by field and temperature is broadened in $HT$ space, as a result the sharp lines corresponding to the transition temperature (here $T_m$) as well as the spinodal lines of supercooling ($H^*, T^*$) and superheating ($H^{**}, T^{**}$) broaden into bands [29, 31, 32]. As discussed earlier [24, 25, 28, 33], if kinetic arrest occurs below the ($H_k, T_k$) line in a pure system, the disordered system would have an ($H_k, T_k$) band. The kinetic arrest band in the $HT$ space contains a set of lines below which the dynamics of the MT is hindered on the time scale of the experiments, as in structural glasses. These lines in the band would correspond to a local region of the sample and therefore a correlation between the positions of ($H_k, T_k$) and ($H^*, T^*$) bands can be established. An anticorrelation between the supercooled and kinetically arrested regions has been proposed and experimentally confirmed in many materials termed as magnetic glasses having first order magnetic transitions [23–28, 33, 34].

On reducing the field from 5 K, 12 T approached by following the path XA, to point D (5 K, 0 T) as shown in the $HT$ phase diagram in figure 2(i), we observe a step at 1.5 T in the $MH$ curve (figure 2(i)), which is expected to be due to the de-arrest of the arrested high temperature phase as it crosses the KA band [23]. At higher temperatures, e.g., 15 K, a signature of de-arrest of the FM-A phase at the field marked by a circle in figure 2(b) is observed by a clear slope increase at the de-arrest field $H_d$. This field $H_d$ marked by circles in figure 2 increases on increasing the temperature and the de-arrested fraction also increases up to 75 K. At temperatures greater than 75 K, $\delta M$ (estimated by the drop that occurs with this increased slope) decreases and we observe a smaller fraction of the de-arrested phase. The corresponding arrested FM-A phase is reduced due to the presence of a higher supercooled fraction at temperatures between $T = 75$ and 150 K. Hence, the width of the linear slope in the return field cycle increases from 5–75 K and then decreases up to
150 K. Considering \( T = 150 \) K at point C in the schematic diagram 2(i), if we traverse on the field axis, we observe a completely symmetrical and reversible \( MH \) curve as shown in figure 2(h). At this temperature whether we approach by zero field cooling (from point F) or cooling in a finite field (from point C), we would get the same behavior, since 150 K lies outside the \((H, T)\) band.

Since the ZFC–\( MH \) shows a fully martensite state and the FC–\( MH \) shows a fully arrested metastable state at 5 K, we can estimate the arrested FM-A fraction from the difference in magnetization \( \Delta M \) between FC–\( MH \) and ZFC–\( MH \) at 8 T for various temperatures as shown in the inset to figure 2(h). After cooling in an 8 T field, the arrested FM-A fraction is almost constant below 75 K, which is the \( T^* \) at 8 T for this sample. Thereafter, due to the presence of a supercooled austenite fraction, \( \Delta M \) gradually decreases and almost vanishes on heating above 150 K. At \( T \sim 150 \) K the kinetic arrest and supercooled bands tend to overlap. Hence, FC–\( MH \) measurements demonstrate the de-arrest of the glass like arrested metastable FM-A states below 150 K.

Cooling and heating in unequal field (CHUF) measurements confirm the kinetically arrested metastable states at low temperatures by observing the de-arrest of the arrested phase [34, 35]. They are also used to distinguish the equilibrium phase from glass like arrested coexisting phases [33, 35]. To verify the GLAS at low temperatures by observing the de-arrest on warming, we have carried out four sets of CHUF measurements. In the first two sets of measurements, magnetization is performed by cooling in a constant cooling field \( (H_c) \), in various different measuring fields \( (H_w) \) for \( H_c > H_w \). In the other two sets, the sample is cooled in various different fields and the measurement field is kept constant during heating. The CHUF set is said to be complete when the measurements are performed in fields below and above the cooling and/or warming field. To confirm the re-entrant transition mentioned above to demonstrate the glass like arrested state, a complete set of CHUF measurements is required.

In the first two sets of CHUF measurements, the constant cooling fields \( (H_c) \) of 6 and 3 T are chosen, for which one observes a finite fraction of both arrested and transformed phases at low temperature (figures 3(a) and (b)). In each case the sample is cooled from 350 down to 10 K. At 10 K, the field is reduced isothermally to the respective measuring field \( (H_w) \) and measurement is carried out while heating. For the other two set of measurements (figures 3(c) and (d)), the sample is cooled in various fields \( (H_c) \) ranging from 0 to 8 T down to 10 K and then the field is isothermally increased or decreased to the constant measuring field \( (H_w) \) of 4 T or 1 T; \( M(T) \) is measured while heating.

As shown by the \( M(T) \) in figure 3(a), when the system is cooled in 6 T and heated in 0.5, 1, 2, 3 or 4 T an initial drop is observed in the magnetization and this drop is more profound when the difference between \( H_c \) and \( H_w \) is large. A similar

![Figure 3. Magnetization as a function of temperature using the CHUF (cooling and heating in unequal field) protocol. (a), (b) The sample is cooled in a constant field of 6 or 3 T and measurements are carried out in various different fields. (c), (d) The sample is cooled under different magnetic fields whereas measurements during warming are carried out in 4 or 1 T respectively. The data shown in (c) were first reported by ourselves in [20].](image-url)
trend is observed in figure 3(b) when the system is cooled in 3 T and warmed in 0.5, 1 or 2 T, except that the initial arrested fraction is smaller. The fall in magnetization on increasing the temperature indicates the de-arrest of the FM-A to LM-M state, i.e., the arrested FM-A phase (untransformed phase during cooling in 6 T/3 T) transforms to the LM-M phase on heating in lower fields. On the other hand, if the warming field is kept constant while the sample is cooled in different fields, the clear drop in magnetization in figures 3(c) and (d) indicates the transition to the LM-M state when \( H_c > H_w \) i.e., for positive values of \( (H_c - H_w) \). Only one transition back to the FM-A state is observed at higher temperature while warming in 4 T after cooling in 0, 1, 2 or 3 T as well as during warming in 1 T after cooling in 0 T. This also shows that the LM-M is an equilibrium state and the FM-A state is a glass like arrested state (GLAS).

For the systems where \( T_m \) (and \( T^* \)) falls with increase in magnetic field, \( H_c > H_w \) is required to observe the de-arrest; \( T^* \) would be lower during cooling in \( H_c \) and higher during warming in \( H_w \). Such a transformation to the low temperature equilibrium phase caused by thermal energy is analogous to the phenomenon of devitrification of the glassy phase, which has been demonstrated in half doped manganites and in many intermetallic alloys classified as magnetic glasses. Devitrification of glasses has been used as evidence for metallic glasses [36]. Another transition of the LM-M phase back to the FM-A phase at higher temperatures is analogous to the melting of the devitrified crystalline equilibrium phase on increasing the temperature. Both these transitions are seen only when the difference between the cooling field and warming field is positive. Hence, the de-arrest of FM-A to LM-M is observed when \( H_c > H_w \), and when the difference between the cooling and the heating field \( (H_c - H_w) \) decreases, de-arrest starts at higher temperatures. Hence, regions which have higher \( T^* \) will have lower \( T_m \). This relation is universal for all magnetic glasses [23, 37].

Based on the results from all the magnetization measurements, we have developed a quantitative phase diagram, as shown in figure 4, similar to the one given for the magnetic glasses mentioned above. The filled squares represent the supercooling limit for the corresponding fields obtained from the peak points of the FCC magnetization data and the empty squares are taken from the points of slope change in the return envelope curve of the ZFC–MH measurements. The triangular symbols represent the superheating limit for various fields obtained from different measurements, as indicated in the legend of figure 4 and explained in the figure caption. The filled and empty circles correspond to the limit of kinetic arrest acquired from CHUF magnetization measurements performed for \( H_c = 6 \) T and 3 T respectively. The star symbols represent the upper limit of the kinetic arrest band obtained from the FC–MH isotherms. We observe that the temperature and fields corresponding to the superheating and supercooling limits obtained from diverse measurements like FCW–MT, ZFC–MH, ZFC–MT, 3/6 T cooled CHUF and FCC–MT fall uniformly in the respective bands. Similarly, the field and temperatures corresponding to the kinetic arrest edges obtained by the quite different sets of measurements mentioned above are consistent. Hence, the validity of our phenomenological model is reinforced by the results obtained from the various measurement protocols falling in the same bands. This phase diagram gives the quantitative information about the phase fraction kinetically arrested across the \( H_H T_K \) band on cooling and/or applying the field as well as about the de-arrested fraction on warming and/or reducing the field. This phase diagram also illustrates that, although the metamagnetic transition from the FM-A to the LM-M state at the lowest temperature takes place at \( \sim 30 \) T for this sample, which is far above the available field range of measurements, we can visualize these features at much lower fields in the field reducing cycle of FC–MH by observing the de-arrest of the FM-A state to the LM-M state.

The formation of magnetic glass in \( \text{Ni}_{50} \text{Mn}_{50-x} \text{Sn} \), FSMAs could be related to the mechanism of structurally modulated phases observed experimentally below the MT. The true structural ground state in these alloys is found to be experimentally ambiguous at low temperatures as various modulated states are observed which are metastable and vary with the concentration \( x \) [38, 39]. Therefore as the degeneracy in the structure of FSMAs is affected by small variations of Sn/Mn concentration, various metastable magnetic states can be formed by the kinetic arrest of the MT in varying magnetic fields, leading to the formation of magnetic glass.
4. Conclusion

We have performed a comprehensive study of the field induced first order FM-A to LM-M transition with respect to temperature and field by a variety of magnetization measurements on ferromagnetic shape memory alloy ribbons of Ni_{45}Co_{5}Mn_{38}Al_{12}. The kinetically arrested glass like metastable states are revealed by cooling and/or applying a field and their de-arrest is observed on warming and/or reducing the field by various thermomagnetic measurements. This phenomenon is validated by a CHUF protocol to study the arrested states. The coexisting states have been produced and higher arrested fraction at higher cooling fields has been confirmed by the novel protocol of field cooled isotherms where the 8 T field cooled state is considered as the initial state of the system. Based on these measurements we regard this ferromagnetic shape memory alloy system as a ‘magnetic glass’, similar to charge ordered ground state manganites.

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