Glassy ferromagnetism in Ni_3Sn-type $Mn_{3.1}Sn_{0.9}$

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Magnetic measurements indicate that the glassy ferromagnetism exists in the ordered intermetallic $Mn_{3.1}Sn_{0.9}$ compound with Ni_3Sn-type structure. This glassy ferromagnetism exhibits both weak canonical behaviors, including a susceptibility sharp peak at T_f=31.8 K and the frequency-dependent susceptibility below T_f, and long-range ferromagnetic ones, including a large coercivity and a magnetization increase after field-cooled below T_f. The glassy component is ascribed to both a small amount of disorder, arising from the random occupation of 2*c* sites in the space group $P6_3/mmc$ by excess Mn atoms, and the common frustration. A power law for the critical slowing down gives the flipping time $\tau_0=3.9\times10^{-4}$ s, the collective freezing temperature T_g=31.5 K, and the dynamical exponent $z\nu$=3.38.

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I. INTRODUCTION

There has been much recent interest on ordered crystal structures that exhibit spin-glass (SG) behaviors or coexistences of SG and long-range magnetic order.[1–3] Commonly, SG behavior is identified most prominently with dilute metallic alloys, in which the long-range Ruderman-Kittel-Kasaya-Yosida (RKKY) interaction dominates, leading to canonical SG effects such as a strong frequency dependence to the magnetic behavior. Recently, SG behavior has been found in a lot of ordered systems, for instance $FeAl_2$,[4] URh_2Ge_2,[5] and $Y_2Mo_2O_7$,[6] etc., which was ascribed to site disorder, crystallographic disorder, or geometrical frustration, respectively. Moreover, the coexistence of ferromagnetic ordering and glassy behavior (or dubbed "glassy ferromagnetism"[7]) has been observed in materials $La_{1-x}Sr_xCoO_3$,[7] $Y_{1-x}Ca_xMnO_3$,[8,9] $La_{0.7-x}Y_xCa_{0.3}MnO_3$,[10] $Sr_3FeCoO_{7-\delta}$,[11] $La_{0.7}Sr_{0.3}Co_{1-x}Ga_xO_3$ (Ref. 12) and $(LaSr)Co_{1-x}Fe_xO_4$.[13] Most of these glassy behaviors are interpreted in terms of the competition between ferromagnetic (FM) double exchange and antiferromagnetic (AFM) superexchange, both of which originate from the clusters as a result of phase separation.[7–13]

In Mn-Sn system, Mn_3Sn has a structure of hexagonal Ni_3Sn-type (DO_{19}) (space group $P6_3/mmc$),[14,15] with lattice parameters of a=0.5665 nm and c=0.4531 nm at room temperature. A mainly antiferromagnetism, along with weak ferromagnetism (WFM), appears below 420 K, the Néel temperature of Mn_3Sn.[14] Nagamiya[16] predicted two modes of spin ordering, i.e., a helix of FM basal net plane, and of triangular basal net plane, for Mn_3Sn. Tomiyoshi *et al.*[14] reported that the spin structure of Mn_3Sn at room temperature is a triangular configuration of inverse geometry in the *c* plane. According to their analysis, the WFM moment above 50 K appears when each spin in the inverse triangle tilts slightly toward its easy axis. Furthermore, Tomiyoshi *et al.*[17] also found that a very large FM component occurs along the *c* axis below 50 K. They argued that the FM arises from a little tilting of each spin, of the inverse triangular configuration in the *c* plane, toward the *c* axis. This assumption was confirmed by Brown *et al.*[18] In spite of the occurrence of the large FM, neutron diffraction measurements suggested that there is no drastic change of spin structure below 50 K.[17,19] On the other hand, the experimental results for $(Mn_{1-x}Fe_x)_3Sn_{1-\delta}$ (x=0.1, δ=0.1) (Ref. 20) and single-crystal $Mn_{3.2}Sn$ (Ref. 18) both reveal the presence of a cusp at low temperature. Unfortunately, little attention has been paid to investigation of the type of cusps.

In this paper, we investigate the low-temperature magnetic properties of Ni_3Sn-type $Mn_{3.1}Sn_{0.9}$ compound in detail. Combining the results of dc magnetization measurements after field-cooled (FC) and zero-field-cooled (ZFC), hysteresis loops, ac susceptibility measurements as a function of measuring frequency, we confirm unambiguously the presence of glassy ferromagnetism at low temperatures. We give a comprehensive exploration of the mechanism of the glassy ferromagnetism in this compound.

II. EXPERIMENTAL DETAILS

A $Mn_{3.1}Sn_{0.9}$ sample was prepared via melting appropriate metals, with purity higher than 99.9%, in a magnetocontrolled arc furnace under a high purity argon atmosphere. An excess (10 wt. %) of Mn over the stoichiometric amount was added to compensate for the mass loss during melting. The ingots were annealed at 1073 K for 50 h and gradually cooled to room temperature. Structural characterization was performed by x-ray diffraction (XRD, Cu K_α, λ=1.5406 Å), scanning electron microscopy (SEM; XL30, PHILIPS), and energy dispersive analysis of x ray (EDX). The XRD pattern of the annealed $Mn_{3.1}Sn_{0.9}$ sample is shown in Fig. 1, confirming that, at least within the sensitivity of XRD, the sample is a single-phase Mn_3Sn with a hexagonal structure of Ni_3Sn-type (a=0.5682 nm and c=0.4534 nm). According to Tomiyoshi's results,[14] Mn_3Sn is generally stable only in the presence of excess Mn, which replaces Sn atom on 2*c* sites of the space group $P6_3/mmc$. Moreover, our SEM observations (not shown here) show that, in $Mn_{3.1}Sn_{0.9}$ compound, there are two types of small zones (bright and dark-gray dots), which differ from the light-gray matrix. However,

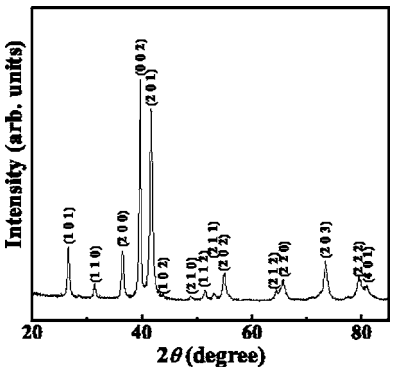

FIG. 1. X-ray-diffraction pattern of $Mn_{3.1}Sn_{0.9}$ compound.

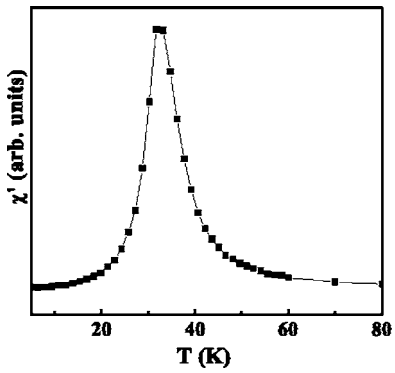

FIG. 2. Temperature dependences of the in-phase component of ac magnetic susceptibilities $\chi'(T)$ at 80 Hz for $Mn_{3.1}Sn_{0.9}$.

the results of EDX (not shown here) reveal that these bright and dark-gray dots have almost the same composition ratio as that of the matrix. Furthermore, the volume fraction of the bright and the dark-gray dots, estimated from the SEM pictures, is less than 1% and about 1%, respectively. The XRD and SEM+EDX results above confirm that the annealed $Mn_{3.1}Sn_{0.9}$ sample is single phase.

The magnetic properties were investigated by a superconducting quantum-interference device (SQUID, Quantum Design) magnetometer. Both ZFC and FC magnetizations (defined as M_{ZFC} and M_{FC}, respectively), as a function of temperature, were measured in a temperature range from 5 K to a certain temperature. For ZFC, the sample was cooled from 295 to 5 K in a zero field and applied a dc magnetic field at 5 K. Then we heated the sample while measuring the M_{ZFC} at the constant field. For FC, the sample was cooled in a dc magnetic field from a certain temperature to 5 K while measuring the M_{FC}. For the ac susceptibility measurements, the sample was cooled from 295 to 30 K at the absence of the magnetic field. Then the sample was heated while the susceptibilities were being measured for different frequencies simultaneously at each temperature.

III. RESULTS AND DISCUSSION

The in-phase component of ac magnetic susceptibility $\chi'(T)$ as a function of temperature for $Mn_{3.1}Sn_{0.9}$ was obtained by applying ac magnetic field with an amplitude of 5 Oe and a frequency of 80 Hz. As shown in Fig. 2, $\chi'(T)$ exhibits a sharp peak at 31.8 K (T_p). An earlier measurement suggested that $Mn_{3.67}Sn$ is in a coexistence of AFM and FM states below the Néel temperature.[21] Tomiyoshi and Yamaguchi[14,15] and Brown *et al.*[18] investigated the spin structures of the single crystals $Mn_{3+\delta}Sn$ ($0.2<\delta<0.7$) at different temperatures. Tomiyoshi *et al.*[17] also reported the occurrence of a very large FM component below 50 K. Some experimental results (see Fig. 2 in Ref. 18 and Fig. 1 in Ref. 20) revealed the occurrence of the cusp at low temperature for single-crystal $Mn_{3.2}Sn$ (Ref. 18) and $(Mn_{1-x}Fe_x)_3Sn_{1-\delta}$ ($x=0.1$, $\delta=0.1$),[20] respectively. To our knowledge, there has been no report on the interpretation of the sharp peak at 31.8 K, and thus a systematic investigation is indispensable.

To elucidate the apparently magnetic transition at 31.8 K, we measured M_{ZFC} with a dc magnetic field of 100, 500, 8000 and 50 000 Oe, and M_{FC}, 100 and 500 Oe, which are represented in Fig. 3 and its inset. The characteristics of the curves include: (1) For the magnetizations with a field of 100 and 500 Oe, M_{ZFC} is not consistent with M_{FC} above T_p; (2) a strong thermal irreversibility occurs below T_p; and (3) T_p shifts to a lower temperature while the transition temperature of the other side T_t (defined as the temperature corresponding to the minimum on the curves of the dM_{ZFC}/dT-T above 31.8 K) to a higher one with enhancing the magnetic field. Representative hysteresis loops, measured at different temperatures after ZFC, are shown in Figs. 4(a)–4(c), respectively. The loops above 50 K show similarly parasitic ferromagnetism (PFM), as proposed by Néel.[22] As mentioned above, the spin structure of Mn_3Sn is an inverse triangular configuration in the c plane at room temperature, and the WFM arises from the slight tilting of each spin in the inverse triangular toward its easy axis. These results can be well employed to interpret our observations above 50 K [see Figs. 3 and 4(c)]. Below 50 K, an abrupt increase of magnetizations (Fig. 3) suggests that a magnetic transition occurs. Correspondingly, the hysteresis loop in Fig. 4(b) shows a small coercive field, while its shape is changed significantly, compared with Figs. 4(c). These behaviors indicate that the FM component is enhanced drastically, which coincides with the findings of others.[14,15,17] Furthermore, T_t shifts to a higher temperature with increasing the magnetic field, as shown in

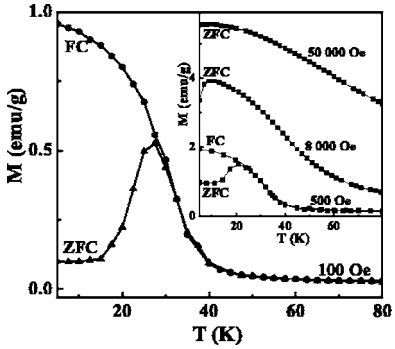

FIG. 3. Temperature dependences of the zero-field-cooled magnetization M_{ZFC} and field-cooled magnetization M_{FC} with a dc magnetic field of 100 Oe for $Mn_{3.1}Sn_{0.9}$. The inset: M_{ZFC} with a dc magnetic field of 500, 8000, and 50 000 Oe and M_{FC}, 500 Oe for $Mn_{3.1}Sn_{0.9}$.

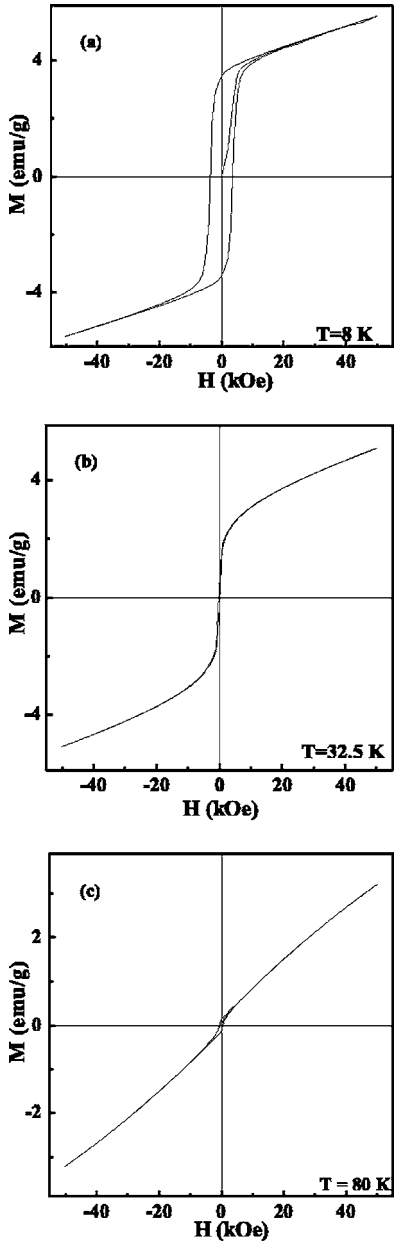

FIG. 4. Hysteresis loops of $Mn_{3.1}Sn_{0.9}$ measured at (a) 8 K, (b) 32.5 K, and (c) 80 K after zero-field-cooled.

the inset of Figs. 3. Far below T_p, the coercive field becomes as large as 8 kOe at 8 K [see Fig. 4(a)]. Besides, the magnetization of all the loops does not saturate up to 50 kOe, and this magnetization can be expressed by $M_F+\chi_{AF}H$ at high field, where M_F is the FM component and χ_{AF} is the slope of M versus H at high fields.

To further clarify the characteristics (2) and (3), the in-phase component of ac susceptibility $\chi'(T)$ as a function of temperature for $Mn_{3.1}Sn_{0.9}$, in an ac magnetic field of 5 Oe at several fixed frequencies in the range of $8<\omega<1000$ Hz, are plotted in Fig. 5(a). Obviously, $\chi'(T)$ is frequency dependent. As the frequency increases, the positions of these sharp peaks shift to a higher temperature, while the magnitude of the peaks decreases. Figure 5(b) represents the same dependences as in Fig. 5(a), with ac magnetic field amplitude of

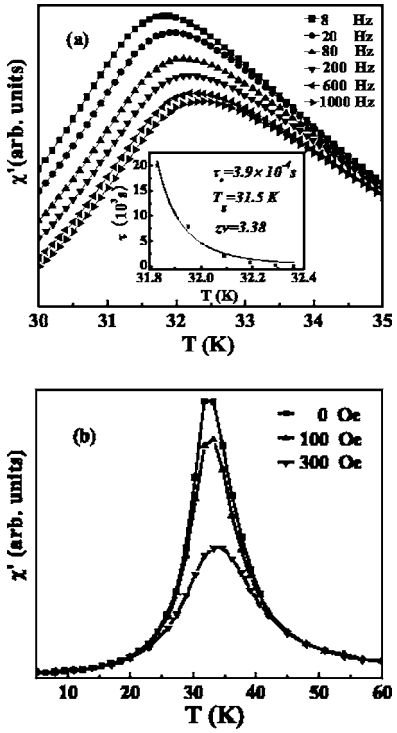

FIG. 5. Temperature dependences of the in-phase component of ac magnetic susceptibilities $\chi'(T)$ for $Mn_{3.1}Sn_{0.9}$ (a) at 8, 20, 80, 200, 600, and 1000 Hz; (b) at 80 Hz with a dc bias field of 0, 100, and 300 Oe. The inset is the best fit to Eq. (1).

5 Oe and a fixed frequency of 80 Hz, under a bias dc magnetic field of 0, 100, and 300 Oe. It is evident that the magnitude of the peaks decreases remarkably and the sharpness becomes rounded with increasing dc field.

In the case of a canonical SG state, undergoing from a paramagnetism to a SG transition, the in-phase component of ac magnetic susceptibility χ' as a function of decreasing temperature, exhibits a cusp at the spin-freezing temperature T_f.[2] The χ'-T curves display a strongly frequency-dependent cusp at a certain temperature. In other words, as measuring frequency decreases, the position of the cusp shifts to a lower temperature, while the magnitude of the cusp increases. The frequency-dependent ac susceptibility was often observed in experiments of canonical SG states.[23,24] Besides, a small bias dc magnetic field can round the cusp of the in-phase component of ac magnetic susceptibility.[25] Moreover, below T_f, a thermal irreversibility, constant M_{FC} can be observed, while M_{ZFC} drops toward zero as temperature decreases.[26] The difference between M_{ZFC} and M_{FC} curves at low temperature could be due to the competition between magnetic couplings.[11]

From our observations above, $Mn_{3.1}Sn_{0.9}$ exhibits canonical SG-like behaviors including a strongly frequency-dependent peak around T_f. However, this SG-like behavior obviously differs from canonical SG one due to: (1) M_{FC} exhibits increasing value as it occurs between T_f and 50 K, which indicates that a long-range FM order is present and dominant. Furthermore, only a small part of magnetic moments seems to be frozen and (2) a relatively small coercivity can be often observed for a canonical SG state,[27] whereas a

large coercivity of 8 kOe at 8 K occurs for $Mn_{3.1}Sn_{0.9}$, which also suggests the presence of long-range magnetic ordering. As a consequence, $Mn_{3.1}Sn_{0.9}$ exhibits signatures of FM ordering [i.e., sharp increase in $M(T)$ below T_t, constantly increased M_{FC}, and $M(H)$ that show finite remanence and coercivity], as well as a strongly frequency-dependent peak in the ac susceptibility around T_f. In other words, the glassy ferromagnetism occurs at low temperatures for $Mn_{3.1}Sn_{0.9}$.

Because $Mn_{3.1}Sn_{0.9}$ is neither a diluted magnetic alloy nor an amorphous sample, the mechanism of the glassy ferromagnetism deserves exploration. The realization of a SG state needs: (1) There must be a competition between FM and AFM interactions so that that no single configuration of the spins is uniquely favored by all the interactions, which commonly called frustration and (2) these interactions must be at least partially random.[28] The ordered intermetallic SG behavior in $FeAl_2$ can be due to both frustration on the complex lattice structure, and disorder due to occupation of the mixed sites.[4] But for U_2PdSi_3,[5] the statistical distribution of Pd and Si atoms on one site of the crystal lattice seems to introduce randomly frustrated U-U exchange interaction necessary for the occurrence of the SG state. Recently, the existence of the glassy ferromagnetism in doped cobaltites,[29–31] such as $La_{1-x}Sr_xCoO_3$, has attracted a great deal of attention. For $La_{1-x}Sr_xCoO_3$,[7,32] and $LaCo_{1-x}Ni_xO_3$,[33] the system evolves from a spin-glass or cluster-glass phase at low doping, to a state that has characteristics associated with ferromagnetic ordering at higher doping. The boundary between the two, or the transitional composition zone, manifests the occurrence of the glassy ferromagnetism. According to the phase separation theory, the glassy component of this glassy ferromagnetism was assumed to produce from the competition between FM double exchange (Co^{3+}-Co^{4+}) and AFM superexchange (Co^{3+}-Co^{3+} and Co^{4+}-Co^{4+}), both of which originate from the clusters as a result of phase separation.[29–33]

Our measurements illustrate that, at about 31.8 K, $Mn_{3.1}Sn_{0.9}$ compound experiences a transition, i.e., from the coexistence of AFM and enhanced FM states to such a mixed state that includes both a long-range FM component and a weakly disordered SG behavior, namely, glassy FM state. It is well known that the Mn-Mn exchange interaction depends on the interatomic distance, the exchange integral oscillating in sign with the interatomic distance. According to the ideal atom configuration of Mn_3Sn,[14,15,18,19] the first and second shortest distances between Mn atoms (i.e., the distance between the two neighboring Mn atoms at $z=1/4$ and $3/4$, and that in the same layer) are 0.3988 and 0.5682 nm, respectively. This reveals that the exchange interaction is different, in magnitude at least, along different directions, as verified by the measurements on the single-crystal $Mn_{3+\delta}Sn$ ($0.2 < \delta < 0.7$).[14,15,18] Brown *et al.*[18] suggested that the FM moment along [001], and a combination of FM and AFM component along [101] and [100], exist for the single-crystal $Mn_{3.2}Sn_{0.8}$. All those indicate that the FM component might compete with the AFM one in interaction and raises the common frustration. On the other hand, for $Mn_{3.1}Sn_{0.9}$, the occurrence of the randomness might arise from the mechanism that the excess Mn atoms can replace Sn atoms at $2c$ sites of the space group of $P6_3/mmc$ randomly, which means that only a small amount of randomness exists in this compound. It was worth noting that a long-range magnetic order may appear in randomly frustrated systems if the amount of randomness is small enough.[28] Therefore, the glassy behavior of the $Mn_{3.1}Sn_{0.9}$ compound can be ascribed to both the common frustration, from the different interaction coupling, and a little randomness. The latter is possibly from the occupation of the $2c$ sites in space group $P6_3/mmc$ at random by Mn or Sn atoms, which resembles much more closely what was found in U_2PdSi_3 and $FeAl_2$.[4]

A sample that exhibits SG behavior will show the critical slowing down, and therefore, the characteristic relaxation time τ diverges at the transition temperature according to the power law:

$$\tau = \tau_*(T/T_g - 1)^{-z\nu}, \quad T > T_g, \tag{1}$$

where T_g is the transition temperature, τ_* is the characteristic flipping time of the magnetic moments, and $z\nu$ is a dynamical critical exponent.[34] We extracted the freezing temperature T (T_f) from the in-phase component of the ac susceptibilities, associated with a relaxation time $\tau = 1/(2\pi \times \omega)$. According to formula (1), the analysis of the data results in a good fitting for the parameter set: $T_g = 31.5$ K, $z\nu = 3.38$, and $\tau_* = 3.9 \times 10^{-4}$ s [see the inset of Fig. 5(a)].

IV. SUMMARY

We have demonstrated that $Mn_{3.1}Sn_{0.9}$ exhibits the glassy ferromagnetism at low temperatures. With a freezing temperature of $T_f = 31.8$ K, this compound behaves as both a frequency-dependent susceptibility and long-range ferromagnetism below T_f. The glassy component of the glassy magnetism is ascribed to both a small amount of disorder, arising from the random occupation of $2c$ sites in the space group $P6_3/mmc$ by excess Mn atoms, and the common frustration. The analysis of our data for the critical slowing down gives the results: $T_g = 31.5$ K, $z\nu = 3.38$, and $\tau_* = 3.9 \times 10^{-4}$ s.

ACKNOWLEDGMENTS

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