RSC Advances

ROYAL SOCIETY OF CHEMISTRY

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ARTICLE TYPE www. rsc.org/xxxxxx | XXXXXXXX

Magnetic and magnetocaloric properties of Dy_5Pd_2: Role of magnetic irreversibility

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Received Xth XXXXXXXXXX 20XX, Accepted Xth XXXXXXXXX 20XX
First published on the web Xth XXXXXXXXXX 200X
DOI: 10.1039/b000000x

In this report magnetic behavior and magnetocaloric response of intermetallic compound Dy_5Pd_2 has been investigated from detail magnetization measurements. Magnetic cluster glass behavior has been observed in the crystalline Dy_5Pd_2 compound below 38 K. Magnetocaloric entropy changes evaluated using Maxwell's relation (MXR) from zero-field-cooled magnetization isotherms, which is used mostly in literature, shows giant inverse magnetocaloric effect (MCE) below 20 K and large conventional MCE around 60 K in this compound. The observed value of inverse MCE is ∼29 J/kg-K for 50 kOe external magnetic field change at T=3.5 K. In this context, origin of the giant inverse MCE and its temperature dependence has been studied. Applicability of MXR and procedure to obtain reversible MCE using MXR which is important for practical application has been discussed. Dy_5Pd_2 exhibits reversible MCE with a peak value of magnetic entropy change ($\Delta S_M{}^{max}$) 8.3 $Jkg^{-1}K^{-1}$ at 60 K under a magnetic field of 50 kOe and irreversible inverse MCE below 20 K.

1 Introduction

On application of the external magnetic field the magnetic moments of the paramagnetic (PM) or ferromagnetic (FM) material has the tendency to align along the direction of the magnetic field. As a result in the presence of external magnetic field under adiabatic conditions an increase of temperature occurs to compensate the decrease of magnetic entropy, which is known as magnetocaloric effect[1] (MCE). MCE is quantified[1] by the adiabatic temperature change ΔT_{ad}, or as the isothermal magnetic entropy change -ΔS_M. In contrast to the PM or FM materials in some magnetic materials on application of external magnetic field the configurational entropy of spin structure increases. As a result, application of magnetic field adiabatically generates cooling (-ΔS_M negative) of the material due to the decrease of lattice entropy, which is known as inverse magnetocaloric effect[2] (IMCE). The irreversibility due to metastable states also generates positive entropy[3].

In most of the literature, authors use Maxwell's relation (MXR) to evaluate magnetocaloric (MC) entropy change using zero-field-cooled (ZFC) isothermal magnetization data. The use of Maxwell's relation is only valid if the system is in thermodynamic equilibrium[4]. On the other hand, zero-field-cooled states do not yield information about the equilibrium states. The positive entropy due to irreversible process[5–8] evaluated using MXR from ZFC magnetization may be confused with reversible IMCE. These raises some important questions (i) can one calculate reversible MC entropy changes accurately from magnetization data of the materials having metastable states and irreversibilities, (ii) can one calculate the amount of irreversibility and the dependences involved in these irreversible processes. The origin of the giant irreversible IMCE and the procedure to discriminate the irreversible part of MCE from the reversible one has not yet been studied in detail and therefore detailed methodology needs to be worked out.

Intermetallic R_5Pd_2 (R=rare-earth) compounds have drawn much attention because the R^{3+} ions are arranged into triangular configuration having geometrical frustration[9–12]. Dy_5Pd_2 is probable to show glassy magnetic behavior associated with the frustration due to the atomic disorder and layered crystal structure, which are the inherent features of R_5Pd_2-type compounds[12,13]. We have investigated the magnetic behavior of Dy_5Pd_2 at different magnetic field strengths under zero field cooled warming (ZFC), field cooled cooling (FCC), and field cooled warming (FCW) modes. Our dc and ac magnetic measurements reveal that Dy_5Pd_2 exhibit a nonequilibrium cluster glass (CG) magnetic state consistent with the triangular lattice configuration favoring spin frustration[14] and glass dynamics. The magnetic entropy change (-ΔS_M) evaluated from the measured M(H) curves under ZFC protocol shows a large irreversible negative to giant reversible positive values with the increase of temperature. Magnetic entropy change due to the application of external magnetic field has also been calculated from isothermal M(H) curves extracted from M(T) curves measured under different applied magnetic field value in ZFC, FCC and FCW protocols. Contrastively, the gian

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IMCE has not been observed in the MC entropy change calculated from FCC and FCW magnetization data. In this aspect, the validity of MXR and applicability of the procedure to calculate MCE by using magnetization method has been described. An estimation of internal entropy production and its temperature dependence has been presented.

2 Experimental details

The polycrystalline Dy_5Pd_2 compound was prepared by arc melting of constituent elements in argon atmosphere. Dy of purity 99.9% were bought from Alfa Aesar (A Johnson Matthey Company) and Pd of purity better than 99.99% was bought from Arora-Matthey Limited. The sample was remelted several times to ensure homogeneity. The x-ray diffraction (XRD) study was carried out around room temperature by using a Rigaku diffractometer using Cu-Kα source having wavelength 1.54Å. The dc magnetization measurements were carried out in the temperature range from 2 K to 300 K and for applied magnetic field up to 7 T using a superconducting quantum interference device (SQUID VSM, Quantum Design) magnetometer.In ZFC mode at first the sample was cooled in the absence of external magnetic field from the temperature which is well above the transition temperature to lowest possible temperature and required magnetic field was applied, followed by the measurement during warming cycle. After ZFC measurement without switching off the applied magnetic field magnetization was measured during cooling(FCC mode) and also during warming (FCW mode). AC susceptibility measurements were carried out in the temperature range of 4-90 K using a commercial susceptometer (CryoBIND Model) at frequencies in the range 37-1737 Hz. Specific heat study were carried out using the semi-adiabatic heat pulse method.

3 Results and discussion

The room temperature XRD spectrum of the prepared sample has been shown in Fig.1. The lattice structure of the bulk crystalline sample has been analyzed by standard profile fitting method using FULLPROF 2009 program. The XRD pattern confirms the single-phase nature of the compound, which crystallizes in cubic Dy_5Pd_2 type (space group Fd3m) structure. The evaluated value of lattice parameter of Dy_5Pd_2 from XRD pattern using Rietveld profile fit is found out to be 13.517(1) Å. The Bragg R-factor and RF-factor are 2.31% and 3.26% respectively. The lattice parameter of the sample is in agreement with the previously reported value [15].

The temperature dependence of ZFC susceptibility (χ) data for applied magnetic field of 50 Oe has been shown in Fig.2(a). χ(T) exhibits a cusp at about T_f = 38 K. The high temperature region of the inverse susceptibility curve has been fitted using the Curie-Weiss law. With the decrease of temperature χ^{-1}(T) deviate from the Curie-Weiss straight line with cooling below 80 K. This deviation from the Curie-Weiss law indicates the beginning of the development of the short-range magnetic correlations. The estimated value of effective magnetic moment (μ_{eff}) and the paramagnetic Curie temperature (θ_P) using the slope of the temperature dependence of $1/\chi$ in the PM region comes out to be 11.43 μ_B/Dy-atom and +50 K respectively. These values are in agreement with previously reported values [16]. The estimated value of frustration parameter [9] (f= θ_P/T_f) is 1.3. The temperature dependence of ZFC, FCC and FCW dc susceptibility (χ) under different values of the externally applied magnetic fields are shown in Fig.2(b). From the figure it is evident that the magnetic susceptibility of Dy_5Pd_2 exhibit irreversible behavior while measuring in ZFC and FC protocols. The extent of irreversibility and irreversibility temperature T_{irr}(H) decreases with the increase of applied magnetic field. This is generally observed in glass-like systems [17]. Though the value of frustration parameter is not too high, the bifurcation in M_{ZFC} and M_{FC} has been observed for maximum value of externally applied magnetic field as high as 50 kOe.

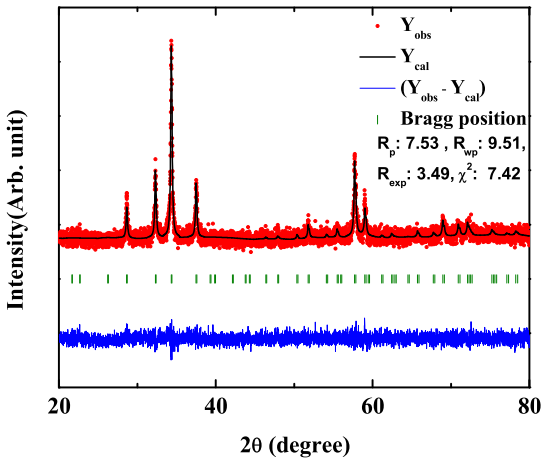

Fig. 1 Powder x-ray diffraction pattern and profile fitting of XRD spectrum of Dy_5Pd_2.

To investigate the nature of the underlying magnetic state ac susceptibility measurements has been performed. The ac susceptibility data at ac field H_{ac} = 1.5 Oe with frequency (ω) 37-1737 Hz has been shown in Fig.3(a) and (b). Both the real and the imaginary part of ac susceptibility data for 37 Hz frequency show a maximum at T_f = 41 K. The susceptibility maximum at T_f shifts with ω (Fig.4). A criterion that is often used to compare the frequency dependence of T_f in differen

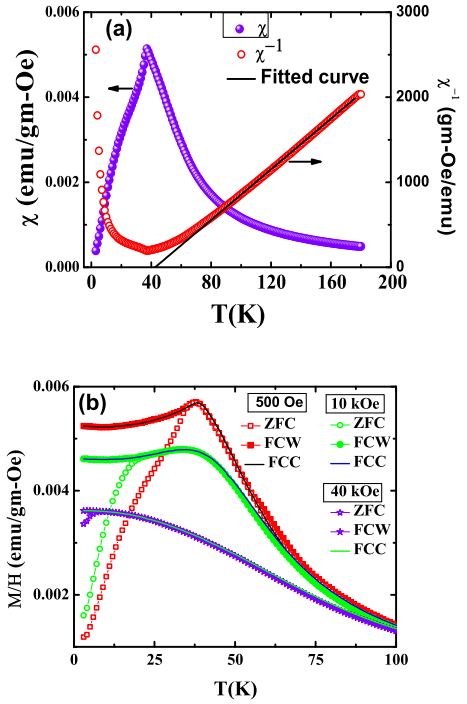

Fig. 2 (a)The temperature dependence of susceptibility and inverse susceptibility measured in the presence of 50 Oe external magnetic field. The solid line is a linear fit to the data.(b)Zero-field-cooled warming, field-cooled warming and field-cooled cooling branches of magnetization measured as a function of temperature in different applied magnetic fields for Dy_5Pd_2 indicating large irreversibility in low temperature region.

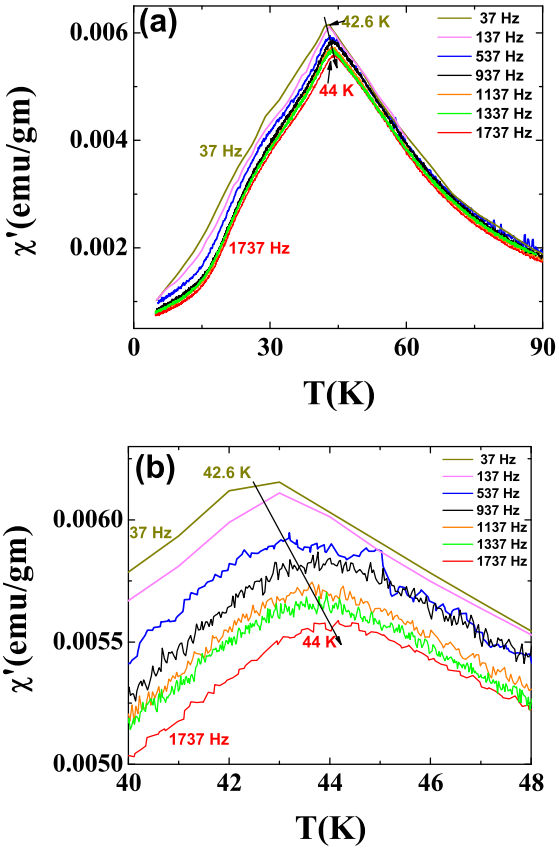

Fig. 3 (a)The real part of ac susceptibility [$\chi'(T,\omega)$] data. (b)$\chi'(T,\omega)$ in the temperature region ranging from 40 to 48 K.

glassy systems is to compare the relative shift in freezing temperature per decade of frequency: $\delta T_f = \Delta T_f/[T_f\ \Delta(\log\ \omega)]$. The obtained value of δT_f is 0.017, which is intermediate between the canonical spin glass (SG) systems (e.g. δT_f for CuMn $\sim$ 0.005) and noninteracting ideal superparamagnetic systems[18] (e.g. δT_f for a-[$Ho_2O_3(B_2O_3)$] $\sim$ 0.28). The value of δT_f is better consistent with those of CG[19].

The frequency dependence of freezing temperature T_f [Fig. 4] obtained from the real part of the ac susceptibility (χ') was fitted with empirical Vogel-Fulcher (VF) law: $\tau = \tau_0 \exp [E_a /k_B(T_f - T_0)]$. Where τ is relaxation time, E_a is activation energy, and T_0 is VF temperature. The best fitting yields for E_a/k_B= 193.5 K, τ_0= 10^{-12} sec and T_0= 35.205 K. The VF temperature T_0 is slightly smaller than T_f obtained from ac measurements. The nonzero value of T_0, which is the measure of intercluster strength[20], indicates the presence of finite interaction between the clusters[21]. Altogether, the frequency dependence of the freezing temperature T_f provides a clear evidence for the formation of a CG state in Dy_5Pd_2.

Zero field heat capacity [$C_0(T)$] data of the Dy_5Pd_2 has been shown in Fig.5. Previously R_5Pd_2 compounds were suggested to exhibit long range AFM ordering[10] with possibility of temperature driven spin-reorientation transition at lower temperature. However no distinct feature has been observed in our measured $C_0(T)$ data around T_f which confirms absence of long range magnetic ordering[22,23]. The cusp observed in $M_{ZFC}(T)$ curve and the upturn of the $M_{FC}(T)$ curve below T_f are rather the characteristic of cluster glasses systems with high concentration of magnetic atoms[13,24].

The isothermal magnetization curves as a function of applied magnetic field are displayed in Fig.6. Before carrying out isothermal ZFC M(H) measurement at different temperatures, the temperature of the sample was raised to well above T_f each time and then cooled down slowly (1 K/ min) to the measuring temperature in the absence of magnetic field. Isothermal magnetization curves as a function of applied magnetic field has been extracted by transposing the isofield FCC and FCW temperature dependence of magnetization data mea

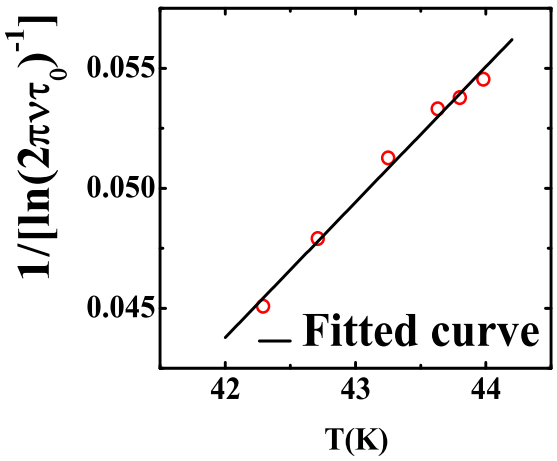

Fig. 4 The T_f dependence and fit of the experimental data of $\chi'(T)$ with the Vogel-Fulcher law.

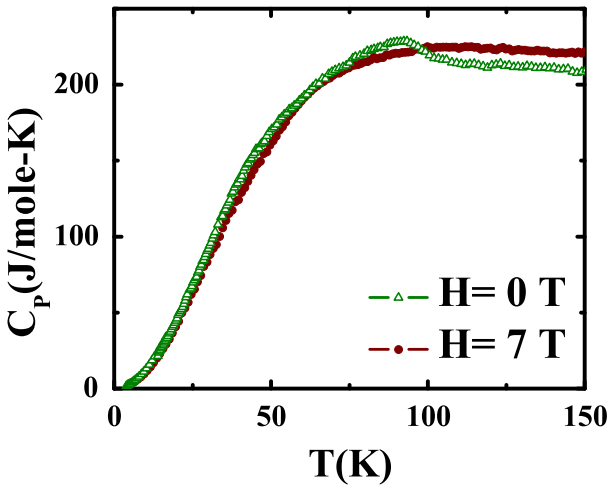

Fig. 5 The heat capacity C_p as a function of temperature for the cluster glass Dy_5Pd_2 both in presence and in absence of external magnetic field.

sured at different magnetic field values. With the decrease of temperature the ZFC isotherm steadily drops for T<20 K giving S-shaped M(H) curves and magnetization is far from saturation in magnetic field as high as 70 kOe. Magnetization isotherms obtained in FCC and FCW protocol steadily increases upto lowest measured temperature and are very similar to each other but different from those measured in ZFC protocol in low temperature region (T< 20 K).

The change in magnetic entropy of a system is usually derived by integrating $M_{ZFC}(H)$ data using MXR [1,25,26]:

$$\Delta S_M(T) = \int_{H_{min}}^{H_{max}} \left(\frac{\partial M(H)}{\partial T}\right)_H dH \qquad (1)$$

Here H_{min} and H_{max} represent the initial and final values of magnetic field. Eq.1 is integrated numerically in the desired range of magnetic fields on the basis of the set of $M_{ZFC}(H)$ curves at different temperatures. We have calculated the area between magnetic isotherms at neighboring temperatures, and then evaluated the entropy change by dividing the temperature difference. The temperature dependence of $-\Delta S_M$ for 70 kOe magnetic field change (ΔH) has been shown in Fig.7. $-\Delta S_M$ changes continuously from giant negative values (IMCE) at lowest temperature to giant positive values (MCE) as temperature increases and after showing a broad maximum around 60 K it tends to zero. The maximum observed value of $-\Delta S_M$ for 70 kOe magnetic field change is ($-\Delta S_M{}^{max}$=) 8.3 J/kg-K at T=57.5 K [Relative cooling power (RCP) = $\Delta S_M{}^{max} \times \Delta T_{FWHM}$ =5.51 J/cc], which is comparable to other giant MC materials [27–29]. The maxima in the temperature dependence of magnetic entropy change have been observed at higher temperature than the glass transition temperature, which is consistent with the earlier reports [30] on the magnetocaloric effect in glassy materials. However the giant IMCE observed in low temperature region is -30.5 J/kg-K for 7 T magnetic field change at 3.5 K, which is surprisingly large.

The temperature dependence of the entropy [25] (S) curve can be obtained from $C_H(T)$ data at the corresponding magnetic field using the relation [31] $S=\int_0{}^T [C_H(T)/T]dT$. The plot of $-\Delta S_M(T)$shown in Fig.7 follows a positive caret-like shape with maxima around 60 K. The values of $-\Delta S_M(T)$ and temperature dependence for different ΔH evaluated from both ZFC magnetization and specific heat data are similar to each other around 60 K and above. Below 60 K gradual deviation from each other has been observed. While the magnetic entropy change ($-\Delta S_M$) obtained from the MXR changes continuously from giant negative values to giant positive values with increasing temperature. In contrast to that, the $-\Delta S_M$ determined from the heat capacity data is always greater than zero.

The SG state is a nonequilibrium one in the thermodynamical sense and the ZFC magnetization does not yield information of the thermal equilibrium behavior [4]. According to mean-field theory there exist a large number of degenerate thermodynamic states with the same macroscopic properties but with different microscopic configurations separated by free-energy barriers in phase space exhibiting many valleys [32]. Multiple metastable states due to random magnetic anisotropy (RMA) give rise to irreversibility. Varying the external field can make the system jump to the nearest local minima [33]. The results of measurement then depend on the kinetics of the phase transition and on the experimental procedure.

Here entropy change in Dy_5Pd_2 has been calculated fol

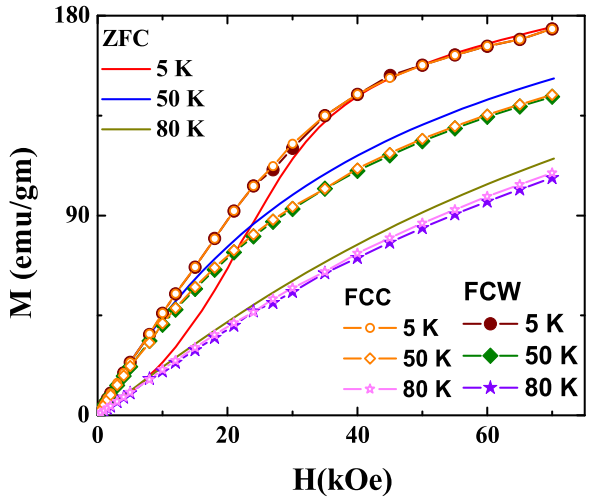

Fig. 6 The isothermal magnetization curves with increasing field at three different temperatures measured in ZFC protocol and extracted from the M(T) data in FCC and FCW protocols.

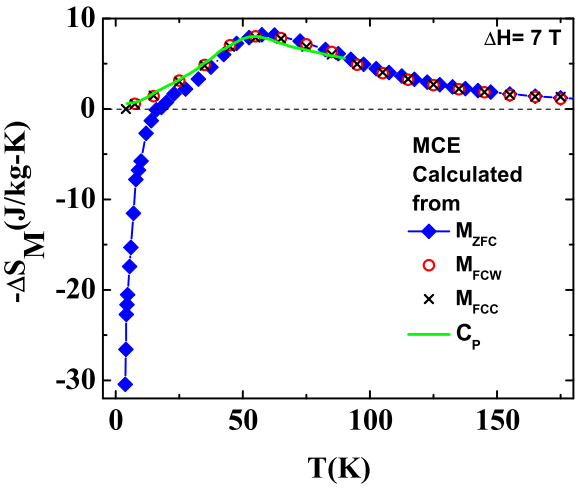

Fig. 7 Magnetic entropy changes under the magnetic field change of 7 T obtained from the heat capacity and magnetization (in ZFCW, FCW and FCC protocols) measurements.

lowing the method adopted in the reversible region, i.e., from isothermal ZFC magnetization data. As a result, there is an additional contribution introducing in the estimation of $-\Delta S_M$ from ZFC magnetization data due to the metastable nature of the measured state which is not taken into consideration. The irreversible positive internal entropy production due to RMA should correlate with the irreversibility in ZFC and field cooled (FC) magnetization which was also observed in rare-earth based bulk metallic glasses[33]. On the other hand the FC magnetization is reversible[34]. In FC measurement the system goes to the near equilibrium state without further "minima hop"[4]. We have calculated magnetic entropy change due to the application of external magnetic field in Dy_5Pd_2 by using MXR also from the magnetization isotherms obtained in FCC, FCW protocol. The temperature dependence of $-\Delta S_M$, shown in Fig.7, around and above the freezing temperature are very similar to those observed in ZFC protocol. However, the temperature dependence in lower temperature regime is distinctly different. The large IMCE observed in low temperature is absent in the $-\Delta S_M$ measured in FCC and FCW protocols. The magnetic entropy change ($-\Delta S_M$) determined from the FCC and FCW magnetization data using MXR is always larger than zero.

The above results indicates that the irreversible entropy in lower temperature region may be related to the irreversibility observed in ZFC and FC magnetization data. The ZFC susceptibility is the FC susceptibility modified by the random anisotropy field[35]. The temperature at which a maximum is observed in the ZFC susceptibility and the irreversibility starts is related to the magnitude and the temperature variation of the coercivity (H_C), which is a measure of the magnetic anisotropy.

$$\frac{M_{FC}}{H+H_c} = \frac{M_{ZFC}}{H} \qquad (2)$$

The temperature dependence of the coercive field H_c in Dy_5Pd_2 has been shown in Fig.8(a). It can be very well described by the exponential function[20,36] $H_C=H_{C0}\exp(-\alpha T)$. As a result Eq.2 can be expressed as,

$$\frac{M_{FC}-M_{ZFC}}{M_{ZFC}} = \frac{H_{c0}\exp(-\alpha T)}{H} \qquad (3)$$

The temperature dependence of $(M_{FC}\text{-}M_{ZFC})/M_{ZFC}$ (= H_C/H) below the freezing temperature for 10 kOe applied magnetic field in Dy_5Pd_2 has been shown in Fig.8(b) which follows $\exp(-\alpha T)$ dependence with the value of fitting parameter (α =) 0.153(1) K^{-1}.

To investigate the origin of the giant irreversible IMCE measured in ZFC protocol using MXR we have analyzed the difference in magnetic entropy change measured in ZFC and FC protocol. The irreversible magnetic entropy change[37,38] (δS_{irr}) from difference (irreversible) magnetization [(M_{FCW} - M_{ZFC}) and (M_{FCC} - M_{ZFC})] by using MXR has been calculated and shown in Fig.8(b). In Fig.8(b) it has been shown that , the temperature dependence of δS_{irr} in the low temperature region can be fitted well by an exponential dependenc [exp(-αT)/T] with α- value 0.156(4). The value of the fitting parameter α obtained from the irreversible magnetization and irreversible entropy changes agree well. The differenc

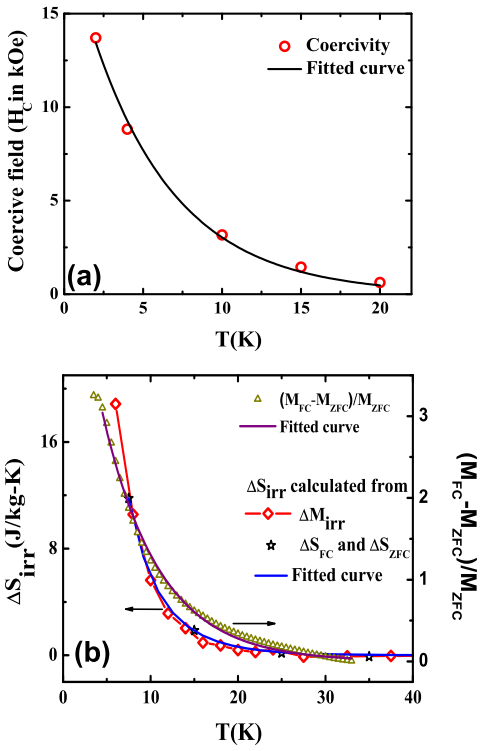

Fig. 8 (a)Temperature dependence of coercive field.(b)Temperature dependence of irreversible magnetization normalized with respect to ZFC magnetization and irreversible entropy changes calculated both from irreversible magnetization and taking difference between entropy FC and ZFC entropy changes. The irreversible entropy calculated in both ways give similar results. The solid lines are the fitted curves.

of magnetic entropy changes between ZFC and FC protocols for 5 T magnetic field change also has been shown in Fig.8(b), which follows similar dependance. These results shows that the additional contribution of positive entropy production on estimation of MCE using MXR from ZFC magnetization in systems having hysteresis related to nonequillibrium behavior. The metastable states increases with decrease of temperature and entropy also increases exponentially.

4 Conclusions

In the intermetallic compound Dy_5Pd_2, the mostly used procedure of calculating MCE from ZFC magnetization measurement using MXR results giant IMCE as well as large MCE. Further detailed analysis using FC magnetization and heat capacity data confirms that the giant IMCE observed in Dy_5Pd_2 is due to magnetic irreversibility and the MC entropy change measured by using MXR from ZFC magnetization data does not give correct estimation of reversible magnetic entropy change. The difference between the entropy changes measured in ZFC and FC protocols reflects the irreversibility in the nonequillibrium magnetic behavior. The present paper highlights the cluster glass magnetic behavior in Dy_5Pd_2 and the methodology for calculating the reversible magnetic entropy change which is the usable part for practical application using MXR from the FCW or FCC magnetization isotherms extracted from isofield magnetization curves.

5 acknowledgments

Tapas Paramanik would like to acknowledge DAE-India for the fellowship.

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