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# Magnetic and magnetocaloric properties of $Gd_2In_{0.8}X_{0.2}$ compounds (X=Al, Ga, Sn, Pb)

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## ABSTRACT

We show that it is possible to replace in  $Gd_2In$  some amount of In by X=Al, Ga, Sn and Pb to obtain  $Gd_2In_{1-x}X_x$  samples after melting. The magnetic and magnetocaloric properties of the  $Gd_2In_{0.8}X_{0.2}$  intermetallic compounds have been investigated through *dc* magnetization measurements. We evidence that the substitution of Al and Ga for In barely changes the Curie temperature  $T_C$  but decreases the second magnetic transition temperature  $T'$  which corresponds to the transition from a ferromagnetic to an antiferromagnetic state. On the other hand, the substitution of Sn and Pb for In strongly increases  $T_C$  and changes the nature or even suppresses the transition at lower temperature. This magnetic behavior gives rise to an interesting way to tune the Curie temperature near room temperature without diluting the Gd network and thus to modify the magnetocaloric effect in  $Gd_2In_{1-x}X_x$  compounds.

### Keywords:

Intermetallic synthesis  
Metamagnetism  
Ferromagnetism  
Magnetocaloric properties

## 1. Introduction

Gd based intermetallics have attracted much attention these last years for their magnetic and magnetocaloric properties. These studies were strongly motivated by the high magnetocaloric effect (MCE) of Gd or by the discovery of the giant MCE in  $Gd_5Ge_2Si_2$  [1]. Among these intermetallics,  $Gd_2In$  is subjected to particular interest because of its unusual magnetic properties. It crystallizes in the hexagonal type structure  $Ni_2In$  (space group  $P6_3/mmc$ ) corresponding to a filled NiAs structure with the lattice parameters  $a=5.413$  and  $c=6.756$  Å [2]. The Gd atoms occupy two sites in  $2a$  (0 0 0) and  $2d$  ( $1/3$   $2/3$   $3/4$ ) and the In atoms one site  $2c$  ( $1/3$   $2/3$   $1/4$ ). As most of the  $R_2In$  ( $R$ =rare earth) binaries,  $Gd_2In$  exhibits a ferromagnetic (FM) ordering below room temperature, and in its case at  $T_C=190$  K [3 7]. Besides,  $Gd_2In$  undergoes an anti ferromagnetic (AFM) transition at  $T'=100$  K and presents a metamagnetic behavior at low temperature. Indeed, the Néel temperature is progressively suppressed with increasing applied field. The nature of the antiferromagnetic and the ferromagnetic structures remains unclear due to the high absorption of neutrons by gadolinium which does not enable a precise determination of the magnetic structure by means of neutron diffraction. Notably, the magnetization value at low temperature is far from zero suggesting the existence of a possible ferromagnetic component (canted or helical AFM structure). Likewise, the isothermal magnetization

curves between  $T_C$  and  $T'$  exhibit a linear behavior at low fields, different from that of a simple ferromagnet [6]. Another interesting point concerning the magnetic properties of  $Gd_2In$  is its behavior in the paramagnetic state, evidencing an effective moment significantly higher than that of the free  $Gd^{3+}$  ion ( $7.94 \mu_B$ ), i.e. between  $8.7$  and  $9.6 \mu_B$  according to different studies [7,3,8]. It reveals the participation of the Gd  $5d$  electrons to the total moment *via* the polarization of these itinerant electrons and thus the key role of the  $4f$   $5d$  exchange in the magnetic interactions and orders. This high effective magnetic moment, also evidenced by  $M(H)$  measurements, makes  $Gd_2In$  an interesting compound for the MCE.

The unusual magnetic behavior of  $Gd_2In$  may be attributed to the competition between anisotropic exchange interactions. The study of the dilution of the Gd network with non magnetic Y or La atoms enabled to better understand this competition. The dilution of the rare earth network leads to a decrease of  $T_C$  as expected, but also to the progressive vanishing of  $T'$  until its total suppression at a critical composition around  $x=0.5$  for  $Gd_2-x(La, Y)_xIn$  [8 10]. It exists two ranges of correlation lengths  $R_c$  between Gd atoms, namely the range of  $3.3 \text{ \AA} < R_c < 3.6 \text{ \AA}$  between Gd1 Gd1 and Gd1 Gd2, and that of  $R_c > 4 \text{ \AA}$  between Gd2 Gd2, with  $R_c = K_F |\mathbf{R}_i - \mathbf{R}_j|$ ,  $K_F$  being the Fermi vector and  $|\mathbf{R}_i - \mathbf{R}_j|$  the distance between the atoms  $i$  and  $j$ . This implies the occurrence of two exchange interactions of different sign and strength. The difference between these two types of exchange coupling could be the main cause of the competition between the FM and the AFM state.

In this work we present the results of the substitution of the non magnetic In specie by isoelectronic p elements as Al and Ga

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and element with one more p electron as Sn and Pb. The objective was to modify the magnetic and magnetocaloric properties of  $Gd_2In$  without diluting the Gd network, in particular to increase the Curie temperature. Composition and homogeneity are checked by X ray diffraction (XRD) and microprobe analysis. Magnetization measurements are presented to evidence the influence of the substitution of In by another p element. All results are discussed in comparison to those of  $Gd_2In$  and diluted compounds  $Gd_{2-x}(La, Y)_xIn$ .

## 2. Experiments

Samples with the following nominal compositions  $Gd_2In_{0.8}X_{0.2}$  for  $X=Al, Ga, Sn$  and  $Pb$  were prepared by melting precisely weighted amounts of high purity elements Gd, In and X (99.9%) in an arc melting furnace. Melting was performed three times to ensure a good homogeneity under a purified argon gas atmosphere. The weight losses during the overall melting process were less than 0.8 wt% and finally the as cast samples were quenched by switching off the power supply.

The composition and the homogeneity of the samples were checked by microprobe analysis using a Cameca SX 100 instrument. The analysis was performed on the basis of intensity measurements of Gd  $L\alpha_1$ , In  $L\alpha_1$ , Al  $K\alpha_1$ , Ga  $L\alpha_1$ , Sn  $L\alpha_1$  and Pb  $M\alpha_1$  X rays emission lines.

Routine X ray powder diffraction was performed with the use of a Philips 1050 diffractometer (Cu  $K\alpha$  radiation) for the characterization of the structural type and for the phase identification. As the samples are sensitive to air, they were ground to produce powder in a glove box and X ray diffraction measurements were performed under a protected atmosphere.

Magnetization measurements were performed using a superconducting quantum interference device (SQUID) magnetometer (Quantum Design MPMS XL) in the temperature range 5–360 K and in fields up to 4.6 T.

## 3. Microprobe analysis and X-ray diffraction

The energy dispersive X ray (EDX) profiles are shown on Fig. 1.

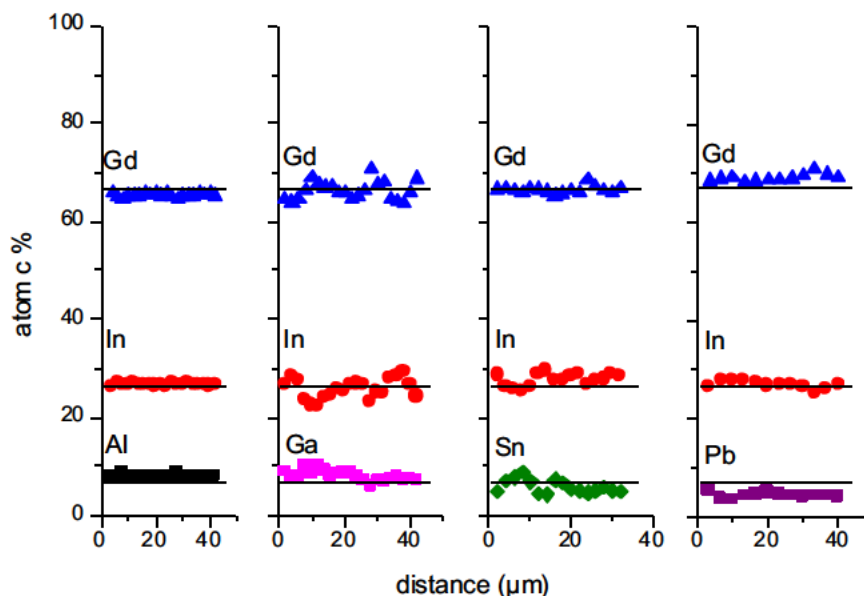


Fig. 1. Chemical composition profiles of nominal  $Gd_2In_{0.8}X_{0.2}$  compounds ( $X = Al, Ga, Sn, Pb$ ) as obtained by microprobe analysis. The solid lines indicate the nominal composition.

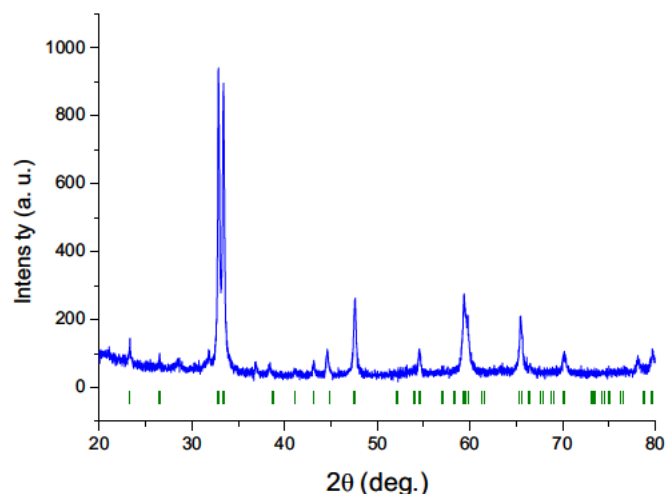


Fig. 2. X-ray diffraction pattern of  $Gd_2In_{0.8}Al_{0.2}$ . The ticks correspond to the peak positions of the phase with the space group  $P6_3/mmc$ .

The EDX analysis reveals a relatively good homogeneity of composition along the samples and confirms that the p metal X is found indeed inside the pseudo binary phases  $Gd_2In_{0.8}X_{0.2}$  with an amount of X close to the expected value of 6.6% as atomic composition percentage. The content of Pb is slightly smaller than the nominal one either because of the much larger size of Pb with respect to In and less solubility or most likely due to Pb losses during melting.

All X ray diffractograms present the same reflections as those of  $Gd_2In$  [2,4,5], confirming that the samples crystallize in the  $Ni_2In$  type structure with the  $P6_3/mmc$  space group (Fig. 2). The small extra peaks come from oxide impurities, the samples being very sensitive to oxygen, and sample holder in Al. The lattice parameters of  $Gd_2In_{0.8}X_{0.2}$  compounds are gathered in Table 1. For  $X=Al$  and  $Ga$  the lattice parameters are close to  $a=5.39$  and  $c=6.74$  Å, i.e. significantly smaller than those of  $Gd_2In$  ( $a=5.413$  and  $c=6.756$  Å [4]) in agreement with the smaller metallic radius of Al ( $r=1.43$  Å) and Ga ( $r=1.41$  Å) with respect with In ( $r=1.66$  Å) [11]. In the case of  $X=Sn$  and  $Pb$ ,  $a$  is higher and  $c$  slightly smaller than for the pure binary  $Gd_2In$ , resulting eventually in a higher

**Table 1**  
Lattice parameters of  $Gd_2In_{1-x}X_x$  compounds determined from X-ray diffraction.

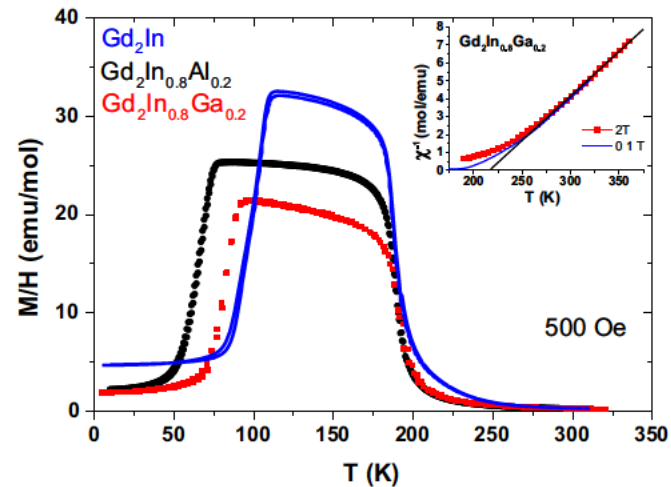
Compound	$Gd_2In$	$Gd_2In_{0.8}Al_{0.2}$	$Gd_2In_{0.8}Ga_{0.2}$	$Gd_2In_{0.8}Sn_{0.2}$	$Gd_2In_{0.8}Pb_{0.2}$
$a$ (Å)	5.413	5.391(1)	5.393(1)	5.430(1)	5.435(1)
$c$ (Å)	6.756	6.742(1)	6.748(1)	6.744(1)	6.751(1)
$V$ (Å <sup>3</sup> )	171.4	169.7	170.0	172.2	172.7
$T_C$ (K)	188	190	190	255	243
$T'$ (K)	104	68	82	-	40

volume cell. This is in agreement for example with the evolution of the lattice parameter  $a$  in the cubic solid solution  $Gd(In_{1-x}Sn_x)_3$  since  $a$  increases linearly from 4.611 Å for  $GdIn_3$  to 4.681 Å for  $GdSn_3$  [12]. Likewise, it is coherent with  $GdPb_3$  which has a higher cell parameter ( $a=4.816$  Å) than its homologous  $GdIn_3$  [13]. More generally, by substituting X for In, we observe that the  $a$  parameter is more affected than the  $c$  one. This can be understood regarding the smallest Gd-In distance (3.124 Å) which is proportional to  $a$  only, whereas the next smallest Gd-In distance (3.375 Å) is proportional to  $c$ .

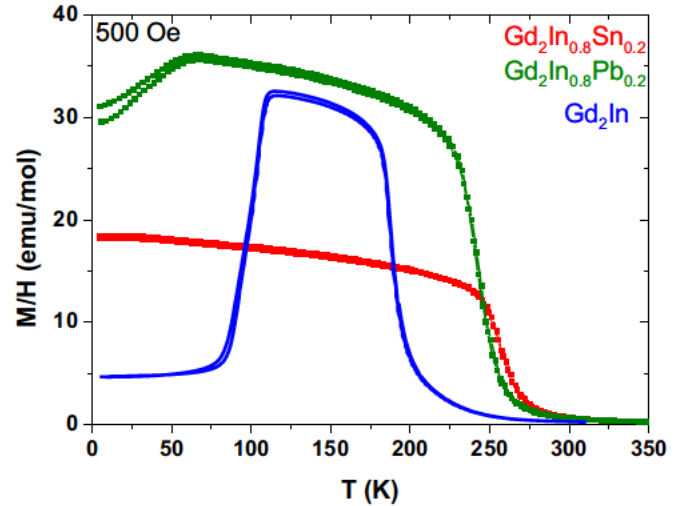
#### 4. Magnetic properties

The temperature dependence of the magnetization of the  $Gd_2In_{0.8}X_{0.2}$  samples is displayed in Figs. 3 and 4. As evidenced on Fig. 3 the features of the magnetization curve of  $Gd_2In$  are kept through In substitution by Al or Ga. In particular, upon decreasing temperature, we still observe a strong increase of the magnetization around  $T_C=190$  K and a sharp drop at a lower temperature  $T'$ . These two anomalies correspond to a FM transition and an AFM one, respectively. It is noteworthy that for an isoelectronic element of In,  $T_C$  barely changes whereas  $T'$  decreases significantly since  $T'=82$  K for  $X=Ga$  and  $T'=68$  K for  $X=Al$ . Based on resistivity and magnetic measurements, it was postulated that the magnetic ground state of  $Gd_2In$  was most likely not a conventional antiferromagnetic one and could correspond to a spiral AFM structure along the  $c$  axis with a possible tilt of the moments toward the  $c$  axis [4]. As the magnetization value at low temperatures is significantly weaker for  $X=Ga, Al$  than for In, it may indicate that the ferromagnetic component is smaller for these pseudo binaries.

The variation of the transition temperatures of  $Gd_2In$  with applied pressure  $P$  was studied by Liu et al. [14]. In particular, they showed that  $T'$  decreases with increasing pressure in the same



**Fig. 3.** Temperature dependence of the magnetization of  $Gd_2In_{0.8}X_{0.2}$  for  $X=In, Al, Ga$  after zero-field cooling and field cooling. The inset shows the inverse of the susceptibility of  $Gd_2In_{0.8}Ga_{0.2}$  measured at 2 and 0.1 T.



**Fig. 4.** Temperature dependence of the magnetization of  $Gd_2In_{0.8}X_{0.2}$  for  $X=In, Sn, Pb$  after zero-field cooling and field cooling.

way as does the Curie temperature of weak itinerant 3d ferro magnetic materials, suggesting the important role of conduction electrons in the magnetic ordering [15]. Interestingly, in the sequence In-Ga-Al we observe the same order of magnitude of diminution of  $T'$  with decreasing unit cell volume (Table 1), considering that a decrease of 1% of the volume corresponds usually to an applied pressure of typically 1 GPa. Therefore, with the substitution of Ga and Al for In, the chemical pressure has a similar effect as a physical pressure applied on  $Gd_2In$ . The variation of the Curie temperature with chemical pressure is less pronounced than with applied pressure because, for these isoelectronic compounds, it depends mainly on the smallest Gd-Gd distance equal to  $c/2$  which is barely affected by the p-metal substitution. The physical pressure  $P$  has a more isotropic effect on the lattice parameters and interatomic distances and thus  $T_C$  increases more significantly with increasing  $P$ .

The magnetic properties were also measured at high temperatures. As shown for example for  $X=Ga$ , the inverse of the susceptibility in the paramagnetic state follows a Curie-Weiss behavior in the investigated temperature range (inset of Fig. 3). The paramagnetic Curie-Weiss temperature is equal to 217 K which is comparable to the values found by Gamari-Seale et al. ( $\theta_p=212$  K [3]) and Ilyn et al. ( $\theta_p=230$  K [7]) for  $Gd_2In$ . The effective moment for  $Gd_2In_{0.8}Ga_{0.2}$  is  $8.9 \mu_B/Gd$ , i.e. much larger than the theoretical value predicted for a free trivalent Gd ion ( $7.94 \mu_B$ ) as previously observed for  $Gd_2In$  ( $9.6 \mu_B/Gd$  [3],  $9.09 \mu_B/Gd$  [7]). This large excess of moment was also observed in pure Gd and in many Gd compounds diluted with non-magnetic metals. It is assigned to the spin polarization of the 5d(Gd) conduction electrons. Thus the 5d-4f interactions are believed to play an important role in the magnetic order as well as in the field-induced metamagnetic transition. This is supported for  $Gd_2In$  by band structure calculations using different methods [16,17] which show that the density of states near the Fermi level is mainly due to the presence of Gd 5d states which form a narrow band. Notably, Singh et al. have clearly highlighted the role of the Gd 5d moments in the metamagnetic behavior of  $Gd_2In$ .

Concerning  $X=Sn$  and  $Pb$ , the magnetic behavior differs more significantly with the substitution than for Al and Ga (Fig. 4). Both compounds exhibit a ferromagnetic transition but at higher temperature than  $Gd_2In$  since  $T_C=255$  K for  $X=Sn$  and  $T_C=243$  K for  $X=Pb$ . It is noticeable that the small modification of the non-magnetic network yields a stronger variation of  $T_C$  than the dilution of the magnetic Gd network [9], emphasizing the importance

of the electronic effect in this substitution. The Curie temperature depends on the magnetic moment and the exchange coupling which is function of the density of states at the Fermi level as well as the distance between rare earth atoms (RKKY interactions). As we may assume that Gd-Gd distances and magnetic moments barely vary through In substitution, the evolution of  $T_C$  is likely due to the modification of the DOS structure near the Fermi level. This would explain the modification of  $T_C$  when replacing In by a non isoelectronic element whereas  $T_C$  stays almost constant for the isoelectronic metals Al and Ga. Further DFT (Density Functional Theory) calculations need to be done to confirm this assumption and to understand the role of the dopant atom in the change of the electronic structure.

The most striking change is observed for the AFM transition since its temperature is considerably reduced for  $X=\text{Pb}$  and even suppressed for  $X=\text{Sn}$  (see Fig. 4). In other words, the In substitution by elements with one more p electron induces an increase of the Curie temperature and a decrease or even a vanishing of  $T'$ . Also, it is clear that the low temperature state of  $\text{Gd}_2\text{In}_{0.8}\text{Pb}_{0.2}$  is not purely AFM which strongly supports the establishment of a conical magnetic structure. The low temperature magnetic behavior is very similar to those of the isotypes  $\text{R}_2\text{In}$  ( $R=\text{Tb}, \text{Er}$ ) [18] and diluted  $\text{Gd}_{2-x}(\text{La}, \text{Y})_x\text{In}$  [9,8] compounds. These results confirm the strong competition between the exchange interactions and the proximity of an instability between a FM and an AFM ground state.

The field dependence of the magnetization were further investigated and are depicted in Figs. 5 and 6 for  $X=\text{Al}, \text{Ga}$  and  $X=\text{Sn}, \text{Pb}$ , respectively. For  $X=\text{Al}$  and  $\text{Ga}$ , we observe below  $T'$  a linear increase of the magnetization at low fields followed by a metamagnetic transition and eventually a tendency to saturate at high fields. This metamagnetic behavior is comparable to that of  $\text{Gd}_2\text{In}$ , as expected considering their similar  $M(T)$  behavior. Above

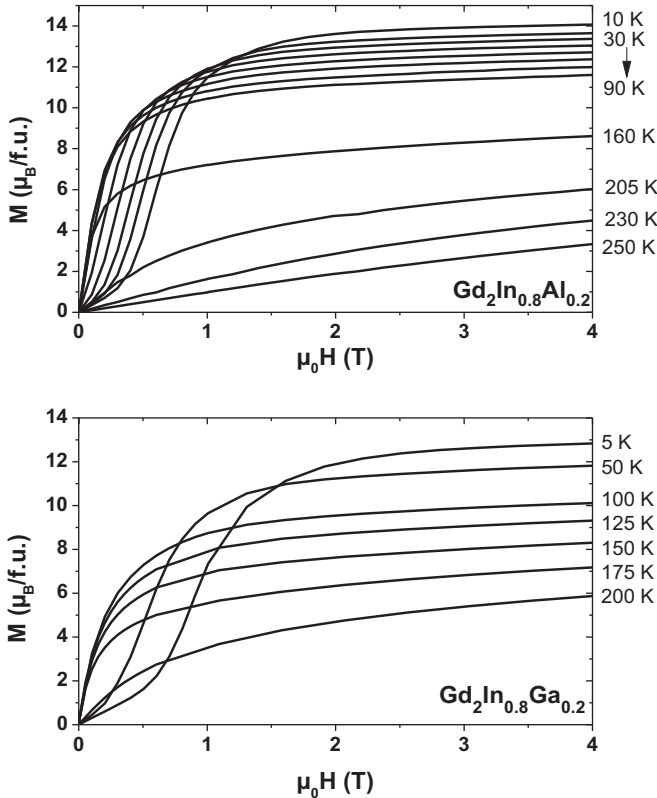


Fig. 5. Field dependence of the magnetization of  $\text{Gd}_2\text{In}_{0.8}\text{Al}_{0.2}$  and  $\text{Gd}_2\text{In}_{0.8}\text{Ga}_{0.2}$  at various temperatures.

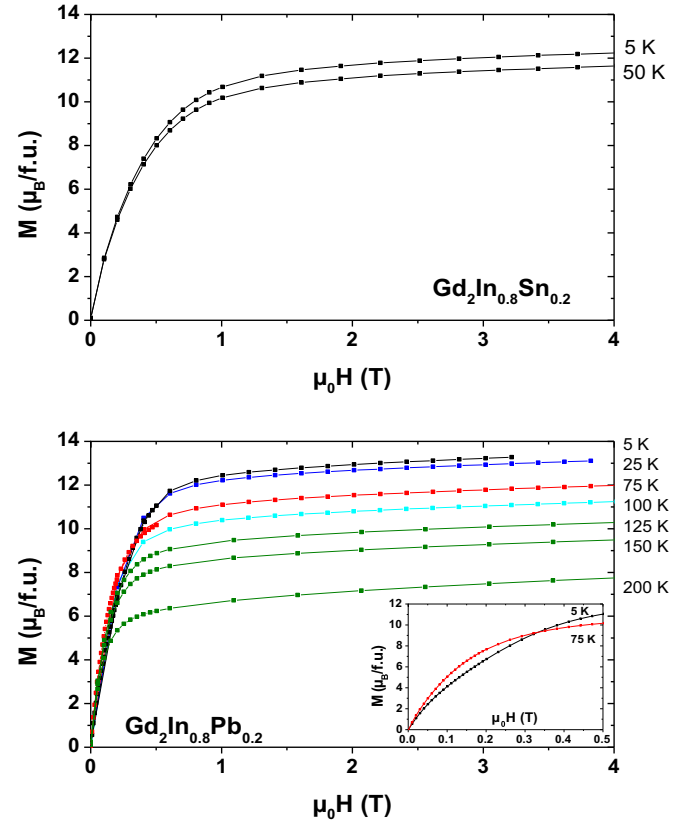


Fig. 6. Field dependence of the magnetization of  $\text{Gd}_2\text{In}_{0.8}\text{Sn}_{0.2}$  and  $\text{Gd}_2\text{In}_{0.8}\text{Pb}_{0.2}$  at various temperatures. The inset in the lower figure shows the low field part of the isothermal magnetization curves at 5 and 75 K for  $\text{Gd}_2\text{In}_{0.8}\text{Pb}_{0.2}$ .

$T'$ , both compounds display a ferro like behavior with a negative curvature over the whole field range, in agreement with the  $M(T)$  measurements. At low temperature, the critical field  $H_C$ , determined at the inflection point of the  $M(H)$ , is found to be at 6 kOe and 9 kOe for  $X=\text{Al}$  and  $\text{Ga}$ , respectively. As  $H_C$  was measured around 12 kOe for  $\text{Gd}_2\text{In}$ , the evolution of the critical field is coherent with the decrease of the temperature of the transition from F to AFM order. For  $\text{Gd}_2\text{In}_{0.8}\text{Sn}_{0.2}$  the  $M(H)$  curves are very similar at 5 K and 50 K confirming that this pseudo binary exhibits the same magnetic order over the whole temperature range below  $T_C$  (Fig. 6). The feature of the curves is compatible with a simple ferromagnetic or an helical ferromagnetic state. On the other hand the magnetic behavior of  $\text{Gd}_2\text{In}_{0.8}\text{Pb}_{0.2}$  is somehow different. The  $M(H)$  feature at low fields and low temperatures is rather linear compared to that at temperature above  $T'$ . In particular it is noticeable that the magnetization value at low fields is higher at 75 K than at 5 or 25 K and both curves cross each other around 3 kOe (see inset of Fig. 6). This results agrees well with the  $M(T)$  curve and may suggest the presence of an helical FM structure at low temperatures and a FM structure above  $T'$ .

## 5. Magnetocaloric properties

Magnetocaloric effect (MCE) was investigated for two selected compounds, namely  $\text{Gd}_2\text{In}_{0.8}\text{Al}_{0.2}$  and  $\text{Gd}_2\text{In}_{0.8}\text{Pb}_{0.2}$ . The isothermal magnetic entropy change  $\Delta S_m$  was calculated by means of isothermal magnetization data and the use of the Maxwell relation. The temperature dependence of  $\Delta S_m(T)$  are plotted in Fig. 7 for magnetic fields variations between 1 and 4 T. The MCE of both compounds displays a negative minimum in the vicinity of their Curie temperature as expected for a ferromagnetic phase

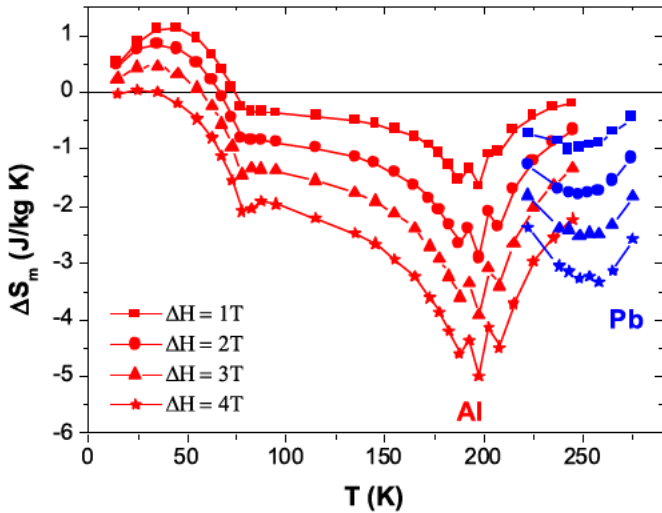


Fig. 7. Temperature dependence of the isothermal magnetic entropy change for  $\text{Gd}_2\text{In}_{0.8}\text{Al}_{0.2}$  and  $\text{Gd}_2\text{In}_{0.8}\text{Pb}_{0.2}$  in red and blue symbols, respectively.

transition. These values reach  $5.0 \text{ J K}^{-1} \text{ kg}^{-1}$  and  $3.3 \text{ J K}^{-1} \text{ kg}^{-1}$  for  $X=\text{Al}$  and  $X=\text{Pb}$  respectively, for an applied field of 4 T. The value is smaller for  $X=\text{Pb}$  because of a higher  $T_C$  but also likely due to less homogeneity of the sample inducing a widening of the maximum of  $\Delta S_m$ . However, the maximum of entropy change found for  $X=\text{Al}$  is close to that of  $\text{Gd}_2\text{In}$  having the same  $T_C$ , i.e.  $4.5 \text{ J K}^{-1} \text{ kg}^{-1}$  or  $3.7 \text{ J K}^{-1} \text{ kg}^{-1}$  according to Bhattacharyya et al. [19] or Ilyn et al. [7] for an applied field of 4 T.

The magnetocaloric effect of  $\text{Gd}_2\text{In}_{0.8}\text{Al}_{0.2}$  was measured down to low temperatures (see Fig. 7). As expected a second anomaly is observed near  $T'$  with a sharp decrease of  $|\Delta S_m(T)|$  with decreasing temperature. Beside, the sign of  $\Delta S_m(T)$  changes below  $T'$  in particular for the low applied magnetic fields. This inverse MCE is consistent with the occurrence of the antiferromagnetic state and is just more pronounced that the metamagnetic state is preserved when applying the field. A similar behavior was observed for  $\text{Gd}_2\text{In}$  with the presence of a maximum of the positive  $\Delta S_m(T)$  below  $T'$ .

## 6. Conclusion

The substitution of In by some non magnetic elements in the binary  $\text{Gd}_2\text{In}$  has been investigated in order to modify its magnetocaloric effect in a quest of finding suitable refrigerant materials for magnetic cooling applications. In this way, we succeed to prepare  $\text{Gd}_2\text{In}_{0.8}\text{X}_{0.2}$  samples for  $X=\text{Al}$ , Ga, Sn and Pb by melting the constituent elements. We showed that the substitution of

isoelectronic elements as Al and Ga for In does not change the Curie temperature whereas the second transition at  $T'$  decreases significantly as observed for  $\text{Gd}_2\text{In}$  under increasing pressure. On the contrary, the substitution of non isoelectronic elements as Sn and Pb for In induces an increase of the Curie temperature in parallel to a strong diminution or even a vanishing of the transition at  $T'$ . These substitutions clearly evidence the strong competition between the ferromagnetic and the antiferromagnetic interactions since the ground state can be easily modified from one to the other magnetic state only by a small change in composition of the non magnetic element. DFT calculations would be helpful to compare the electronic density of states near the Fermi level between those compounds to better understand the role of the conduction electrons and dopant elements in the evolution of the magnetic properties with composition.

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