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Influence of the lattice parameters on the magnetic properties of the weak ferromagnetic rare-earth cuprates $(R,R')_2CuO_4$

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Abstract

A systematic study of the magnetic properties of the T'-phase rare-earth cuprates $(R,R')_2CuO_4$ (R,R' = rare earths) is presented. Attention is focused in those compounds with lattice parameters a = d(Cu-O(1)-Cu) smaller than 3.91 Å, where weak ferromagnetism (WF) is first detected. It is observed that, although a diminution of the Cu-O(1)-Cu distance enhances monotonically the local WF component, it does not happen the same for the macroscopic WF characteristics, which reach a maximum at $a_1 = 3.885$ Å, decreasing thereafter. The same point marks the onset for coercive fields in M versus H loops. The results are interpreted in a framework which considers arrangements of oxygen distortions within the CuO(1)₂ planes, origin of WF, as creating a non-unique WF component in those planes. © 1997 Elsevier Science B.V. All rights reserved.

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1. Introduction

Up to now there have been many reports on solid solutions in the form $(R,R')_2CuO_4$ (R and R' = lanthanides) [1–3]. Their crystal structure changes from that of La₂CuO₄-type (T phase) to the T' phase [4] (for R = R' = Tb and heavier rare earths can only be synthesized under high pressure [5]). Both structures have in common the presence of CuO(1)₂ planes separated by $R_2O(2)_2$ blocks.

In these CuO(1)₂ planes, the Cu moments are strongly antiferromagnetic (AF) exchange coupled with J > 1000 K [6]. This causes two-dimensional (2D) AF correlations to develop as the temperature decreases. At a temperature, T_N , between 250 and 280 K, a crossover to a 3D AF order occurs [7–9]. When the lattice parameter, *a*, decreases beyond a certain value the Cu–O(1)–Cu distance becomes too small and distortions in the CuO(1)₂ planes take place. These distortions are associated to oxygen atoms displacements perpendicular to their bonds [2]. This allows a Dzyaloshinskii– Moriya-type antisymmetric exchange interaction between the Cu moments [10], leading to weak

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ferromagnetism (WF) [11, 12]. WF is observed in all the R_2CuO_4 cuprates for R = Gd and heavier rare earths [10]. The importance of studying this family of compounds is that in a few cases a suitable electron doping of the CuO(1)₂ planes leads to superconductivity (SC). In the T'-phase R_2CuO_4 for R = Nd, Pr, Sm and Eu, SC is achieved by an appropiate doping with Th or Ce and annealing in reducing atmosphere [13, 14]. However, for R = Gd and heavier rare earths SC cannot be induced [15, 16]. It is interesting to notice that in this type of compounds SC and WF do not coexist and the boundary is found at R = Eu, Gd [17]. This has been used to speculate about the mutual exclusion between WF and SC [10].

The study of solid solutions $(R,R')_2CuO_4$ permits, through the variation of the rare earth, a variation of the Cu-O(1)-Cu distance. In this way, the boundary between a perfect AF arrangement and weak ferromagnetism as a function of the lattice parameter a = d(Cu-O(1)-Cu) has been determined to be near $a_0 = 3.910 \text{ Å}$ [1, 3, 10]. This value of a_0 is very similar for the disappearance of SC with electron doping. In fact, Eu₂CuO₄, lies just at this boundary, because samples grown in alumina crucibles and CuO flux do not show WF, but when they are grown in Pt crucible and PbO or CuO flux [18] they show WF when field cooled (FC) [19]. Furthermore, SC can only be obtained in the Ce-doped samples when they are grown in alumina crucible and CuO flux. Since there is no systematic study of the magnetic properties of these systems for lattice parameters smaller than a_0 , the aim of this work will be to show the evolution of WF below this boundary. The data have been completed with others extracted from the available literature.

2. Sample preparation and characterization

Ceramic samples were prepared as follows: (a) the lanthanide oxides were dried in air at 900°C, (b) stoichiometric amounts of Ln_2O_3 and CuO were mixed and reacted at 900°C for about 24 h in alumina crucibles, (c) the mixtures were repeatedly pulverized and sintered at 1000°C for about 1 h, (d) the mixtures were pressed into $\frac{3}{8}$ in pellets at ~ 4 kbar, then placed in alumina crucibles and

Table 1	
Synthesized	compounds

Compound	Nominal concentration	
Eu _{1.8} Gd _{0.2} CuO ₄	x = 0.2	
$Eu_{2-x}Y_xCuO_4$	$0 \le x \le 0.6$	
$Gd_{2-x}Eu_{x}CuO_{4}$	$0.3 \le x \le 0.55$	
$Gd_{2-x}Tb_{x}CuO_{4}$	$1 \leq x \leq 1.6$	
$Gd_{2-x}Y_{x}CuO_{4}$	$0.3 \le x \le 0.4$	
$Sm_{2-x}Y_{x}CuO_{4}$	$0.2 \le x \le 0.8$	
NdYCuO ₄	x = 1	

able	2	
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Т

 R_2CuO_4 compound with pure T' phase

Compound	x without impurities
$Eu_{2-x}Y_xCuO_4$	x = 0, 0.01, 0.05, 0.2, 0.4
$Gd_{2-x}Tb_xCuO_4$ $Gd_{2-x}Eu_xCuO_4$	x = 1, 1.2, 1.4, 1.45, 1.5 $x = 0.2$
$\mathrm{Sm}_{2-x}\mathrm{Y}_{x}\mathrm{CuO}_{4}$	x = 0.2, 0.3, 0.4

sintered in air for ~ 48 h at temperatures between 1050 and 1150°C depending on the compound. Table 1 shows the synthesized compounds. Phase purity and lattice parameters were determined by X-ray powder diffraction. ESR was used to determine the presence of other phases such as Ln₂CuO₅ and Ln_2O_3 . None of them showed Ln_2CuO_5 , but in some of them Ln_2O_3 could be detected. Table 2 lists the samples which were found free of second phases, within the accuracy of the experiments. Rietveld refinement of the X-ray diffraction data showed that the lattice parameters of the samples vary monotonically with the substitution of the rare earth, covering a range of lattice parameters up to the limit where the sample synthesis required high pressure. The lattice parameters for compounds synthesized under pressure were obtained from the literature. The DC magnetization was measured with a vibrating-sample magnetometer in the temperature range 77 < T (K) < 300 and magnetic fields up to 10 KOe.

3. Analysis and results

Compounds with lattice parameter, a, smaller than $a_0 = 3.91$ Å show the following WF signatures:

a peak in the real and imaginary parts of the ACcomplex magnetic susceptibility and a low-field microwave absorption corresponding to the WF resonance mode [3, 10]. Both features are observed at $T \approx 280$ K independently of the rare earth. This temperature agrees with the AF ordering temperature, $T_{\rm N}$, found in neutron diffraction experiments for all this family of compounds [7-9]. Another typical WF signature is the non-linear behaviour of the DC-magnetization with the applied field [20], which persists even in a temperature range above $T_{\rm N}$ [21]. This has been attributed to an induced WF above T_N by the external field [21]. For the R_2CuO_4 compounds whose Cu sublattice is perfectly AF-arranged, the magnetic response is that of the \mathbb{R}^{3+} paramagnetic ions [10]. However, for compounds with lattice parameter a smaller than $a_0 = 3.91$ Å, the Cu moment, within the CuO(1)₂ planes, cants away from their perfect AF structure. This gives rise to a net magnetization of the $CuO(1)_2$ planes and to a polarization at the rareearth site [22], increasing the magnetization at low fields. Nevertheless, at sufficiently high fields the magnetization approaches a linear regime and the total DC-magnetization can be described in the following form:

$$M_{\rm DC}(T) = M_{\rm Cu} + \chi_{\rm d}(T) [H_{\rm a} + H_{\rm i}^{\rm R}], \qquad (1)$$

where, in analogy with the WF found in CoCO₃ [23] and GdCrO₃ [24], M_{Cu} is the net magnetization in the CuO(1)₂ planes due to the canting of the Cu moments, H_i^{R} the internal field at the rareearth site driven by M_{Cu} [22], H_a the applied magnetic field and $\chi_d(T) = (dM_{DC}(T))/(dH_a)|_{H_a \to \infty}$ the paramagnetic susceptibility of the rare-earth ions, which in some cases may take the form $\chi_d(T) = C_{\text{R}}/(T + \theta)$.

The spontaneous magnetization, defined as the value of $M_{de}(T)$ at $H_a = 0$ in Eq. (1),

$$M_{\rm s}(T) = M_{\rm Cu} + \chi_{\rm d}(T)H_{\rm i}^{\rm R}$$
⁽²⁾

contains M_{Cu} and H_i^R , which are the relevant parameters that characterize the WF behaviour of the compound. For those compounds where M_{Cu} and H_i^R are temperature and field independent, Eq. (1) predicts a linear relation between the sample DCmagnetization, $M_{DC}(T)$, and the applied field, H_a .

Therefore, from a plot of $M_{DC}(T)$ versus H_a at different temperatures one can fully determine $\chi_d(T) = (dM_{DC}(T))/(dH_a)|_{H_a \to \infty}$. On the other hand, the internal field, H_i^{R} , has usually been obtained by simply extrapolating Eq. (1) back to $M_{\rm DC}(T) = 0$; but, this last method has to be used with caution because such an extrapolation implies a drop of $M_{\rm Cu}$ in Eq. (1), which is only valid if $M_{\rm Cu} \ll$ $\chi_{\rm d}(T)H_{\rm i}^{\rm R}$. In the case of rare-earth ions like Tb³⁺ or Ho^{3+} (with effective magnetic moments of 10.6 and 9.72 $\mu_{\rm B}$, respectively) $C_{\rm R} = N \mu_{\rm eff}^2 \mu_{\rm B}^2 / 3k_{\rm B}T$ is quite large, and $M_{Cu} \ll \chi_d(T) H_i^R$ can apply. But, as C_R depends on μ_{eff}^2 , χ_d will strongly depend on the rare earth. For example, it is a factor 100 smaller for Sm^{3+} (with effective magnetic moment 1.5 μ_{B}); in such a case, $\chi_d(T)H_i$ is no longer much larger than $M_{\rm Cu}$, and the usual extrapolation of the high-field linear part of M versus H curves does not yield a correct value for H_i^{R} . The higher the rare-earth effective magnetic moment, the more accurate is the extrapolated value.

So, in order to measure M_{Cu} in such cases, magnetization data at several temperatures are needed. Eq. (1) can be rewritten as

$$\chi_{\rm d}^{-1}(T)[M_{\rm DC}(T) - \chi_{\rm d}(T)H_{\rm a}]$$

= $H_{\rm i}^{\rm R}(T) + C_{\rm R}^{-1}(T + \theta)M_{\rm Cu}(T);$ (3)

therefore, the left-hand side of Eq. (3) is expected to be a linear function of T. It is then possible to determine M_{Cu} from the slope and H_i^R from $T = -\theta$, as done by other authors [25, 26]. Anyway, it is important to notice that in the case of any dependence of M_{Cu} and H_i^R on H_a this method will underestimate them if they are not at their maximum values. The M_{Cu} values used in this work are obtained from the literature [25-29].

The DC-magnetization data on our samples were taken at a fixed temperature of 77 K and fields up to 10 kOe. Assuming that under these conditions M_{Cu} and H_i^R are temperature and field independent, i.e. they reached their maximum (saturation) values, we extracted from the linear behaviour of M_{DC} (77 K) versus H_a : (i) the rareearths susceptibility χ_d (77 K) = $(2 - x)\chi_R$ (77 K) + $(x)\chi_{R'}$ (77 K), from the slope and; (ii) the



Fig. 1. M versus H loop for $Sm_{1.6}Y_{0.4}CuO_4$ at 77 K.

spontaneous magnetization, M_s (77 K). Fig. 1 shows a typical $M_{\rm DC}$ (77 K) versus $H_{\rm a}$ for one of the samples, $Sm_{1.6}Y_{0.4}CuO_4$. Fig. 2a shows M_s (77 K) as a function of the lattice parameter, a, for all the samples studied in this work. One can observe in this figure that for the $Gd_{2-x}Tb_xCuO_4$ series $M_{\rm s}$ (77 K) drops as a decreases beyond a value $a_1 = 3.885$ Å. In Fig. 3 we show for the same compounds M_s (77 K), taken as the extrapolation to $H_{\rm a} = 0$ of linear fits of the $M_{\rm DC}$ (77 K) versus $H_{\rm a}$ curves between 10 kOe and different fields, $H_{\rm L}$. This is made in order to check its field dependence. It can be seen that it shows a little dependence on $H_{\rm L}$, indicating that the pure linear regime is not achieved at moderate fields; and therefore, a relative underestimation takes place. On the other hand, Fig. 2b shows the values of M_{Cu} as a function of a, which are obtained from the literature [25–29]. It is seen that $M_{\rm Cu}$, associated to the degree of canting of the copper moments, behaves monotonically with the lattice parameter up to a value $9.3 \times 10^{-3} \mu_{\rm B}$ /mol taken from Refs. [27, 28]. Substracting data of Fig. 2b from that of Fig. 2a and using the measured values for χ_d (77 K), H_i^R can now be extracted using Eq. (2) (M_{Cu} is not neglected now). Fig. 2c shows the internal fields, H_{i}^{R} , as a function of a, showing a similar trend to M_s . The value $a_1 = 3.885$ Å, at which the maxima of M_s and H_i^{R} are located, marks also a sharp change in the value of the measured coercive fields, H_c , as seen in Fig. 4.



Fig. 2. (a) Extrapolated magnetization, M_{s} , versus the *a* lattice parameters. The pure R_2CuO_4 and $Sm_{2-x}Gd_xCuO_4$ data are from Refs. [1, 20, 25–29]. (b) M_{Cu} versus the lattice parameter *a*. Data are from Refs. [1, 25–29]. (c) Average internal field at the rare-earth site, obtained as explained in the text, versus lattice parameter *a* for several $R_{2-x}R'_xCuO_4$ compounds. Lines are guide to eye.

4. Discussion

We part from the fact that the origin of WF is the distortion of the oxygen atoms in the $CuO(1)_2$ planes from their symmetric positions. The degree of distortion is modified through the substituted rare earth, as the R^{3+} ion size affects the volume of the lattice and, more concretely, to the *a* lattice parameter [3, 10]. The heavier is the rare earth the



Fig. 3. M_s versus the lowest extrapolation limit, H_L , in $Gd_{2-x}Tb_xCuO_4$. The symbols (\triangle), (\square), (\times), (+), and (\bigcirc) correspond to x = 1, 1.2, 1.4, 1.45, and 1.5, respectively.



Fig. 4. Coercive fields, H_c , versus lattice parameter *a* for several $(R,R')_2CuO_4$ compounds. The size of the markers is the order of the error. The Eu₁Tb₁CuO₄, Gd₂CuO₄, Sm_{0.2}Gd_{1.8}CuO₄, and Tb₂CuO₄ data are from Refs. [1, 10, 21, 25, 26], respectively.

smaller is the radius of \mathbb{R}^{3+} ; therefore, we will obtain cuprates of smaller lattice parameters, the oxygen distortion will be bigger and the canting of the copper moments will increase. This is in agreement with Fig. 2b where we see that M_{Cu} increases with the diminution of *a*. However, we should expect this to increase also all the other WF characteristics like H_i^{R} and M_s . From Fig. 2a and Fig. 2c it is clearly not the case. Concerning H_i^{R} , it seems to act less intensely on the rare-earth site. It has been suggested that the drop in H_i^R could be due to a change in the coupling of the rare-earth ions with the copper sublattice. The reduction in the coupling constant has been attributed to a change in the angle R-O(2)-Cu with the lattice parameter variations [29]. As M_{Cu} varies more or less linearly with respect to a between 3.86 < a (Å) < 3.90, it could be reasonable to hope a similar decrease of H_{i}^{R} . This is not observed, therefore, it does not seem to be the correct interpretation. Another possible cause for the fall of H_i^{R} could be that the net WF component within the $CuO(1)_2$ planes would not be completely saturated for the highest measuring fields, affecting the extrapolation of M_s , and subsequently, the value of $H_i^{\mathbf{R}}$. To check that, we had obtained Fig. 3, in which we had presented a variation of M_s on the extrapolation limits. Anyway, this is also unsatisfactory because the experienced variation of M_s is too small to compensate the observed one versus the lattice parameters. Even more, although the pure linear regime is not completely achieved within the used field ranges, it does not change too much after 10 kOe, values remaining quite similar; e.g., using a SQUID magnetometer, the measured internal field of Tb₂CuO₄ is very small, even at 50 kOe.

We think that the solution must be searched in the analysis of the distortion of the O(1) atoms. Neutron diffraction studies on single crystals of ¹⁵⁵Gd-enriched Gd₂CuO₄ found a long-range superstructure of the T' phase described by the Acam space group [30]. Such a superstructure implies the existence of a coherent sum of all the local WF contributions, giving a unique (macroscopic) WF component [31]. This WF component is the reason for the existence of H_i^R and M_s , so, if it is affected, changes in M_s and H_i^{R} will be measured. There are two ways of modifying the WF component in the $CuO(1)_2$ planes: (a) Modifying the degree of canting of the Cu moments, which we do with the substitution of different rare earths, changing the Cu–O(1)–Cu distances; the effect is an enhancement of the canted Cu structure and of the WF local component. (b) Modifying the superstructure, i.e., modifying the sum of the local WF components. In the case of Gd₂CuO₄ the existing superstructure maximizes this sum, so, any variation of it can only be reflected macroscopically by a fall of H_i^R , as the rare-earth site will be polarized by differently oriented WF regions. It must be noted that this internal field is obtained from macroscopical measurements (M_{DC}) ; therefore it is not a local field, but an average of all the local components acting on the rare-earth site. Regarding (a), if the lattice parameter decreases the WF characteristics should increase, so the fact pointed out in (b) could be playing a role below a_1 in the explanation of the decrease of the WF signatures. We have recently observed in Gd₂CuO₄ ceramic samples that by means of changes in their synthesis conditions its magnetic behaviour can be affected [32, 33] (amongst other changes, H_i^R is reduced), which we have interpreted with the existence of WF domains or clusters arising from a non-unique superstructure [33, 34]. Such non-uniformity would be due to a too-low synthesis temperature to stabilize the oxygen distortions. In this framework, as the a lattice parameter is lowered, the distortion becomes deeper, making its changes more difficult. For $a_1 = 3.885$ Å, which seems to be a critical value, the stabilization of a unique superstructure would be very difficult. The communication by Rouco et al. [27, 28] of the existence of several superstructures in Tm_2CuO_4 and Y_2CuO_4 supports this idea. Moreover, in the case of Y₂CuO₄, different magnetic behaviours are observed depending on the synthesis conditions; even for samples from a same batch, different magnetic dynamic responses are measured [29]. The parallelism with the aforementioned observations in Gd₂CuO₄ is remarkable, supporting the idea exposed here. For single crystals of this compound (with the Acam superstructure), no coercive fields are measured in M versus H loops [20]; however, we have observed in ceramic samples small coercive fields of about 15 Oe [33, 34], which depend also on the synthesis procedure. For this reason we have associated these coercive fields to the non-existence of a unique superstructure, resulting in WF clusters [33, 34]. In the case of R_2CuO_4 with smaller *a* parameters, the deepness of the distortion should enhance the effect, leading to larger H_c 's. This is what data show. The lattice parameter $a_1 = 3.885$ Å, at which the global H_i^{R} and M_s fall (revealing the non-uniformity of the WF component in the $CuO(1)_2$ planes) is also the starting point of the growth in H_c . The reason

would be that different organizations of the O(1) distortions have taken place, directing the resultants of the WF contributions to several directions. As the distortion becomes larger, the needed magnetic field for the reversal of the WF local sums needs to be increased.

5. Conclusions

The magnetic properties of the T' phase R_2CuO_4 compounds, R = rare earth, have been studied as a function of the a = d(Cu-O(1)-Cu) lattice parameter below $a_0 = 3.91$ Å, which is the onset point of a weak ferromagnetic (WF) character in these materials. It is observed that a reduction of the lattice parameters leads to an increase of the local WF component in the CuO(1)₂ planes, M_{Cu} , arising as the canting of the Cu antiferromagnetic structure. Nevertheless, some DC-magnetization-measured macroscopic WF signatures, like the internal field at the rare-earth site, H_i^R , and the extrapolated remanent magnetization, $M_{\rm s}$, do not behave monotonically, like M_{Cu} , but they reach a maximum at $a_1 = 3.885$ Å and drop for lower lattice parameters. a_1 is also the onset point of non-negligible coercive fields, H_c , which grow considerably under a_1 . This evolution of the WF signatures is interpreted as the loss of the superstructure which describes the distortion of the oxygen atoms from their symmetric positions in the T' phase. This superstructure would be substituted by locally arranged distorted CuO(1)₂ zones, creating WF clusters, contrary to a unique WF component existent for $a_0 > a > a_1$. The existence of such WF clusters would be the origin of the measured coercive fields.

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