Reply to Comment on "Nature and entropy content of the ordering transitions in RCo_2"

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In the previous Comment, M. Forker and coworkers claim that perturbed angular correlation (PAC) data leave no alternative to the conclusion that the spontaneous magnetisation of $PrCo_2$ and $NdCo_2$ undergoes a discontinuous, first-order phase transition at T_C. We show here that their claim is in clear contradiction with a wealth of experimental evidence, including our own. Finally, we propose a possible origin for the disagreement between their interpretation of the PAC results and the literature on this subject.

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I. INTRODUCTION

Before the publication by M. Forker *et al.* of "Perturbed angular correlation study of the magnetic phase transitions in the rare-earth cobalt Laves phases RCo_2" in 2003 [1] there was a general consensus in literature on the nature of the magnetic ordering phase transitions of the RCo_2 series. With a rather extense experimental base, the magnetic ordering in the Co Laves phases with R = Er, Ho, and Dy were always classified as first-order transitions (FOT), and the rest as second-order transitions (SOT). Initially there had been some controversy on the reason why $NdCo_2$ and $PrCo_2$ magnetic ordering would not be FOT, as the available models on the RCo_2 series [2, 3] would indicate. Apparently, the riddle had been clarified [4, 5] by introducing the effect of cell volume on the series expansion of the free energy.

However, a series of ^{111}Cd perturbed angular correlation (PAC) experiments led Forker *et al.* to partially review the literature on this subject, concluding that the "few detailed experimental studies of the phase transitions in the light RCo_2" available leave some doubts "as to the classification of the transitions of $NdCo_2$ and $PrCo_2$ as SOT's."

The accumulation of experimental results suggesting the SOT character of $NdCo_2$ and $PrCo_2$ transitions is in our opinion rather conclussive, but one might be forced to admit that several of those *evidences* in literature are reached by *inspection* of temperature dependent experimental data, and some of them may be not as conclusive as would be desirable. In Forker *et al.*'s opinion, [1] the clear doubts on the SOT character of the $NdCo_2$ and $PrCo_2$ Curie transitions are fully solved by PAC measurements. As they expose again on their previous Comment, PAC leaves no alternative to the conclusion that the spontaneous magnetization of $PrCo_2$ and $NdCo_2$ undergoes a discontinuous, first-order phase transition at T_C.

This forceful conclusion by Forker *et al.* as well as the large magnetocaloric effect of RCo_2 FOT's were the motivations to perform our differential scanning calorimetry (DSC) study on RCo_2 [6–8]. As it is known, DSC is well suited to determine the thermodynamic character of a phase transition because proper integration of the calorimetric signal yields the latent heat in FOTs while in a SOT, the signal reflects a continuous change of entropy. Moreover, the application of a magnetic field also helps to discriminate SOTs from FOTs by field-dependent DSC measurements. Our experimental results indicating a SOT character for $NdCo_2$ and $PrCo_2$ were clearly in disagreement with Forker *et al.*'s conclusion, which is stated again in their previous comment.

This reply is organised as follows: in section II we will show that Forker *et al.*'s claim is in clear contradiction with a wealth of experimental evidence in literature. In section III, we analize the dependence of the critical temperature on the magnetic field, and we apply the Banerjee criterion to RCo_2 magnetization data (R = Nd, Pr, Er, Ho). Finally, in section IV we propose a possible origin for the disagreement between the interpretation of the PAC results and the literature on this subject. Finally, in section V, we summarize our results.

II. PREVIOUS RESULTS

To clarify the present status of the subject, it is important to review some of the previous literature on the subject, and Forker *et al.*'s view on it.

Specific heat: of particular importance are the adiabatic calorimetry data of $NdCo_2$ and $PrCo_2$ published by Deenadas et al [9] in 1972. Specific heat measurement is a crucial experiment on this subject, although the work passed unnoticed by Forker et al. both in Ref. 1 as in their previous comment. To put it simply, the C_p curves are as incompatible with a FOT character for

$NdCo_2$ and $PrCo_2$ Curie transitions as our DSC results [6, 7]. One would argue on these measurements (as the previous comment does on our DSC) that the prototypical SOT shape of $NdCo_2$ and $PrCo_2$ specific heat curves at T_C may be caused by a (rather capricious) distribution of inhomogeneous phases with critical temperatures that *obfuscate* [10] the FOT peaked shape. But this strange phenomenon is shown false by one of the the samples itself: $NdCo_2$ offers a first-order spin reorientation transition (SRT) just $\sim$ 60 K below the magnetic ordering transition as a perfect *witness* on how much a FOT is affected by sample quality *on the very same specimen*. Both $NdCo_2$ samples, the one measured in Ref. 9 and our own [6] show a very abrupt, clearly first-order peak at $T_{SRT} = 40$K. The FWHM of the FOT peaks are < 1K and 0.5K in Deenadas *et al.* and our DSC, respectively, ruling out the hypothesis about a broad distribution of inhomogeneous phases with different critical temperatures in our samples. Of course there is some T_C distribution, but, as experiment show, it is not so broad to fully *obfuscate* a FOT peak. In contrast, a nicely broad, λ-shaped, SOT-like peak is observed at the magnetic ordering, which spreads as much as several decades around the Curie temperature. Indeed, the FWHM of the λ-peak at T_C is $\sim$15 times larger than the FWHM at T_{SRT} in both works. Although $PrCo_2$ does not offer such a *witness* peak, the shape of the specific heat and DSC curves at the order transition is hard to reconcile with a FOT character.

Those offered by calorimetry are not the only sound arguments pointing in the same sense.

Transport properties: $NdCo_2$ and $PrCo_2$ do not present the abrupt drop in resistivity shown in the Er, Ho and Dy Laves Co phases, as clearly shown by several studies of the transport properties of the RCo_2 series: see for example the recent reviews by Duc and Brommer, [11] and Gratz and Markosyan [12]) but very specially those by Hauser *et al.* [13], Deenadas et al. [9], and Duc *et al.* [4], which in our opinion can only be interpreted as a clear evidence of a SOT in $PrCo_2$ and $NdCo_2$ at the Curie temperature. Indeed the authors of the cited works do identify $PrCo_2$ and $NdCo_2$ magnetic orderings as SOTs in every case, contrary to Forker et al.'s reinterpretation of their works.

Mössbauer spectroscopy: There are also temperature-dependent Mössbauer experiments [14] on $NdCo_2$, which are of especial interest here, as PAC is also a hyperfine technique. Unfortunately, the number of spectra shown in Ref. 14 is scarce (the temperature step is of the order of 10 K) and therefore it is difficult to affirm anything on the order of the transition. But contrary to the point of view expressed by Forker *et al.* (as if the results were positive to a FOT) the fact that Atzmony et al. do measure in $NdCo_2$ one spectra in the middle of the transition (attaining about 50% of the saturation signal) in the vicinity of T_C is much more probable for a SOT than for a FOT.

Structure and magnetostriction: It is possible to agree with Forker *et al.* that a FOT is not excluded (as it is not a SOT, either) by the some of the studies on the temperature dependence of the lattice parameters and the crystal distortions[15] previous to the publication of Ref. 1. More recently, Ouyang *et al.* [16] present a very detailed temperature dependent study showing the continuous character of the changes of the lattice constants at the Curie temperature, in strong contrast with those taking place at T_{SRT}. Ouyang and coworkers also show $NdCo_2$ anisotropic magnetostriction, with similar characteristics: a FOT is hard to reconcile with Ouyang *et al.*' data at T_C, as the anisotropic magnetostriction constant λ_{100} presents a discontinuous drop at T_{SRT} which is absent at T_C. Indeed, a previous neutron scattering work[17] already was very clear about the SOT character of $NdCo_2$ and $PrCo_2$ as the thermal and magnetic strains are very similar to those of Tb and very different of those of Dy, Er and Ho. We can not agree with Forker *et al.* in their comments about the implications of this work in this subject.

Theory: The first models on the critical behaviour of the RCo_2 compounds [2, 3], which assumed a rigid structure of the Co band through the series suggested that the low T_C of $NdCo_2$ and $PrCo_2$ should correspond to a FOT. However, later calculation showed that the lanthanide contraction along the series drastically change the mechanism of the Co-moment formation at T_C from light- to heavy- rare-earth RCo_2 compounds. Khmelevskyi and Mohn are as clear as this: *a first-order phase transition in the light-rare earth compounds $PrCo_2$, $NdCo_2$ [...] is impossible because the Itinerant Electron Metamagnetism conditions are not fulfilled for the d subsystem and the Co atoms carry a magnetic moment caused by spontaneous polarization due to the exchange interaction within the Co d band. The second-order transition in these compounds is thus a consequence of the internal properties of the d subsystem [...]*.

However, the interpretation of the PAC data on $NdCo_2$ and $PrCo_2$ presented in Ref. 1 is that *there is no way to avoid the conclusion* that in $PrCo_2$ and $NdCo_2$ the spontaneous magnetization undergoes a FOT at T_C. Given the disagreement of PAC results with previous literature, it would really be of interest to find some clear criteria to solve the riddle.

III. FOT-SOT DISCRIMINATION CRITERIA

T_C field dependence: A typical behavior of magnetic FOTs is the dependence of T_C with the applied field. The shift of the $T_C(H)$ has been clearly observed in many systems showing magnetic FOTs, including MnAs[18], $Gd_5Si_{4-x}Ge_x$ [19], manganites[20], pyrochlores[21], and the RCo_2 (R=Er, Dy, and Ho) [22, 23], among others.

This behavior is indeed directly connected with the FOT character of the magnetic transition, as first pointed out by Meyer and Taglang. [24]. During the sixties a general theory of magnetic ordering FOTs was developed

[18, 25, 26], including different mechanisms leading to first order transitions. All those works coincide to predict that as soon as the Curie transition is a SOT, T_C does not depend on the applied field, while a $\partial T_C/\partial H \neq 0$ is predicted in the FOT case.

In Fig. 1 we show the shift of the critical temperatures ($\Delta T_C(H) = T_C(H) - T_C(H=0)$) as a function of applied magnetic field measured on the RCo_2 series. The ΔT_C for R = Ho (■), Er (•), Nd (▽), and Pr (□) have been obtained from our DSC measurements [7], while $DyCo_2$ (⊙) data are taken from magnetostriction measurements by del Moral *et al.* [23], which expands to much higher fields than our DSC data on $DyCo_2$. The straight lines are linear fits to the observed values. The following slopes are obtained: 2.0 ± 0.1K/T for $ErCo_2$, 3.7 ± 0.1K/T for $HoCo_2$, 9.1 ± 0.1K/T for $DyCo_2$, -0.04 ± 0.08K/T for $PrCo_2$ and 0.06 ± 0.08K/T for $NdCo_2$. Clearly, the $PrCo_2$ and $NdCo_2$ values are compatible with T_C =cte, as a visual inspection of Fig. 1 suggests. The spin reorientation temperature of $NdCo_2$ shows a very well defined linear dependence of T_C with the magnetic field, with slope $\partial T_{SRT}/\partial H = 3.1 \pm 0.3$K/T, as it is shown in the inset of Fig. 1. In our opinion, the contrast between $\partial T_{SRT}/\partial H$ and $\partial T_C/\partial H = 0$, obtained in a single measurement series on the same sample, combined with the specific heat and DSC different shapes at T_{SRT} and T_C is an extremely strong argument in favor of a FOT SRT and a SOT magnetic ordering in $NdCo_2$ and, by extension, in $PrCo_2$.

Banerjee criterion: In 1964, Banerjee [27] condensed Landau-Lifshitz [28–32] and Bean-Rodbell [18] criteria providing a tool to distinguish magnetic FOTs from SOTs by purely magnetic methods [33]: the presence or absence of a negative slope segment on the isotherm plots of H/M *vs.* M^2 near the critical temperature indicates a FOT or a SOT transition, respectively. Recently, this criterion has been applied with success to several systems[20, 21, 34]. In fact, Banerjee criterion is equivalent to test the S-shape of an Arrott plot (M^2 *vs.* H/M),

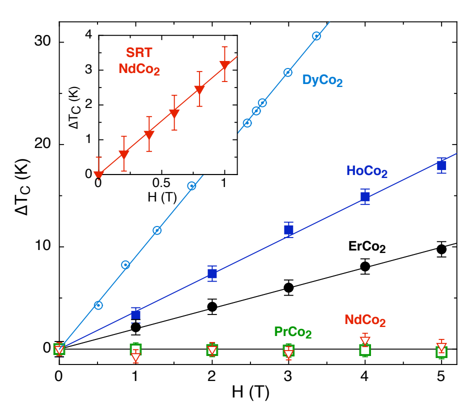

FIG. 1: Variation of the critical temperature with the applied magnetic field in the RCo_2 series. The inset shows data for the spin reorientation transition in $NdCo_2$.

which has already been used as a test for magnetic FOTs in heavy rare-earths RCo_2[11].

In Fig. 2, we show the H/M *vs.* M^2 plot of isotherms of $NdCo_2$ (♦), $PrCo_2$ (□), $ErCo_2$ (•), and $HoCo_2$ (■). The $ErCo_2$ and $HoCo_2$ have been measured at $T = 37$ K and $T = 84$ K, which are ~ 4 K above T_C in order to evidence the effect of the field-induced FOT at about $H = 1$ T. The magnetization curves of $NdCo_2$ and $PrCo_2$ have been measured at their corresponding T_C ($T = 41.6$ K for $PrCo_2$ and $T = 100$ K for $NdCo_2$), as it is clear from Fig. 1 that a field $H \leq 5$ T would not induce the transition at a higher temperature.

The same samples as those used in Refs. 7, 8 and 35 have been used, and their detailed structural and magnetic characterization has been already given. From Fig.2 it is evident that $ErCo_2$ and $HoCo_2$ fulfill Banerjee criterion for FOTs, as a negative slope region coincide with the field-induced transition. $PrCo_2$ and $NdCo_2$ present an homogeneously increasing slope along the whole curve. Therefore, the magnetic ordering transition of $PrCo_2$ and $NdCo_2$ must be identified as a SOT. A complete study of the Banerjee criterion applied to RCo_2, including magnetization measurements at different temperatures and fields above and below the critical temperature is in progress and will be published elsewhere. The magnetization curves of $ErCo_2$ and $NdCo_2$ are shown for comparison as an inset of Fig.2. $NdCo_2$ data have been multiplied in the inset by a factor 2 for clarity.

IV. XMCD MEASUREMENTS AND THE RCO_2 ELECTRONIC STRUCTURE

RCo_2 are systems with a complex electronic structure. This electronic complexity may be at the origin of the controversy between Forker's interpretation of PAC measurements and every other significant piece of experimen-

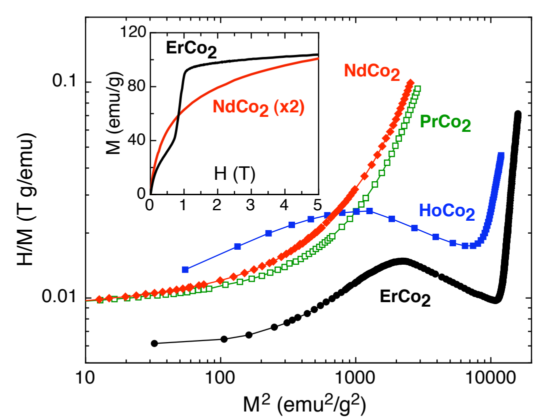

FIG. 2: Double-log $H = M$ vs. M^2 plot of isotherms of $NdCo_2$, $PrCo_2$, $ErCo_2$, and $HoCo_2$ measured at temperatures slightly above $T_C^{H=0}$. Only the FOTs do show a negative slope section. The inset shows the magnetization curves of $ErCo_2$ and $2{\cdot}NdCo_2$.

tal work on $NdCo_2$ and $PrCo_2$ magnetic transition known to us.

Probably, the pivotal role in the subject we are dealing with is played by the fact that PAC measurements sample a very particular component of the RCo_2 magnetization, as the dominant contribution to the ^{111}Cd hyperfine field comes from the s electron spin polarization, as explained in the previous comment [10]. Forker *et al.* assume the following hypothesis: *the s electron spin polarization is induced by (and proportional to) the polarized 3d-band*, and their interpretation is based on this fact. They assume that a PAC measure of the s band magnetic polarization is proportional to the Co 3d moment. We have performed experiments on $ErCo_2$ of another magnetic local probe, as it is x-ray magnetic circular dichroism (XMCD). The XMCD spectra have been recorded at the Co $L_{2,3}$ and K edges (corresponding to Co 2p→3d and Co 1s→4p transitions, respectively) and at the $M_{4,5}$ Er edges (corresponding to Er 3d→4f transitions). The experiments have been performed at 4.0.2 beamline at ALS and ID08 and ID12 beamlines at ESRF. The experimental details are given elsewhere [35–37].

Although it is well established that $L_{2,3}$ and $M_{4,5}$ edges probe the 3d Co and the 4f Er magnetization, respectively, what is observed in the K Co edge is the polarization of the Co sp band[38, 39], which is strongly hybridized with the 5d rare earth band [36, 37, 40]. In particular, in RCo_2 the effect of the rare-earth moments on the Co sp polarization is very strong, as our dichroic measurements show: the XMCD spectra shown in Fig. 3 evidences this fact. In Fig. 3 we compare the dichroic spectra obtained at 90K (well above T_C, upper panels) and at 5 K (well below T_C, lower panels), under an applied field of 1T, at the Co $L_{2,3}$ edge (left panels), the Co K edge (central panels), and the Er M_5 edge (right panels).

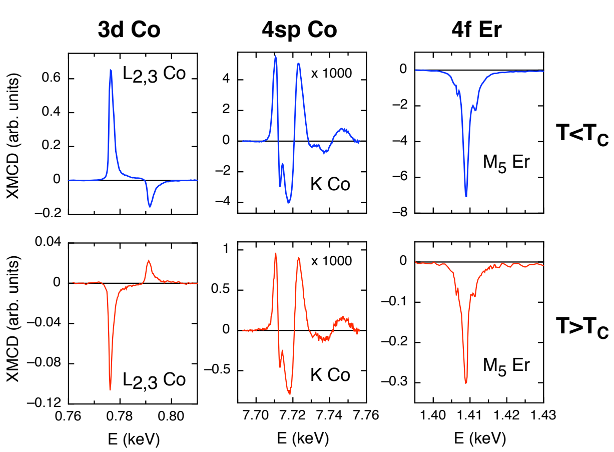

FIG. 3: XMCD spectra recorded on RCo_2 at the Co $L_{2,3}$ edges (left panels), Co K edge (central panels), and the Er M_5 edge (right panels). The XMCD spectra have been obtained at 90K (upper panels) and at 5 K (lower panels) under an applied field of 1T.

First we focus in the fact that Er M_5 and Co $L_{2,3}$ edges, thus Er 4f and Co 3d electrons respectively, show the expected behavior of a ferrimagnet under a moderate applied field, as indeed $ErCo_2$ is. The magnetization of the sublattice with the largest magnetic moment (Er) is, in average, always parallel to the applied magnetic field, thus maintaining its sign unaffected by the phase transition. Note that, as expected, the Er M_5 XMCD magnitude is strongly enhanced, as it is the magnetization. The magnetization of the Co 3d band is probed by the XMCD measured at the Co $L_{2,3}$ edges.

The Co 3d magnetization, with a much smaller magnetic moment than the Er ions, changes its sign through the phase transition, as expected in a ferrimagnet. Surprisingly, the polarization of the Co sp band, probed by the Co K edge XMCD does not behave proportionally to the Co 3d moments. As the two central panels of Fig. 3 show, the Co sp-band induced magnetic moment *does not* change sign upon the ferrimagnetic transition. The sp Co magnetization has the same sign both above and below T_C, just as the 4f rare-earth magnetic moment. Moreover, the shape of the measured K-edge spectra is strongly different to the Co metal one. This clearly shows that the sp Co band is strongly hybridized with the rare-earth 5d band, and it is much more influenced by the 4f moment than by the Co 3d one, as it has been suggested in recent literature [36, 37, 40, 41]. This fact offers a clue to understand the apparent disagreement of the PAC results with our [6, 7] and the rest of experimental results already cited about the nature of the $NdCo_2$ and $PrCo_2$ transition. In fact, two factors may be important:

- As shown by our XMCD results, the sp band magnetic polarization is not proportional to the polarized Co 3d-band, but it is strongly influenced by the 5d rare-earth moments. This has been well stablished by K-edge XMCD measurements in rare-earth intermetallics, and it rules out the base hypothesis for PAC interpretation by Forker et al.
- Moreover, PAC spectra is a local probe, and ^{111}Cd occupies the rare-earth site in RCo_2. Therefore, as the polarization of the Co 3d-band is strongly influenced by the 5d rare-earth moments, which are induced by the well localized 4f ones by intraatomic exchange, one would expect that the local polarization of the sp band is strongly affected by the absence of the rare-earth ion at the probing site. If this is the case, ^{111}Cd PAC in RCo_2 would give a perturbed signal, which may not be easy to correlate with the sp polarization of the parent undoped material. However, PAC is not our field of expertise and we leave this discussion open to the hyperfine-probe community.

V. CONCLUSIONS

We have shown that the SOT character of the $NdCo_2$ and $PrCo_2$ transitions from paramagnetism to ferromagnetism is established out of any reasonable doubt, except for the fact that the temperature evolution of the PAC spectra remains to be explained. It is clear from literature that PAC spectroscopy has been used with success to study phase transitions (see references cited in Refs. 1 and 10) in other systems. We do not doubt of the general validity of the technique, but RCo_2 is probably a not very favorable system, due to the complexity of its electronic structure (at the edge of Co moment formation) and to the fact that ^{111}Cd PAC in RCo_2 occupies the R site, locally affecting the Co sp moment in a very strong way.

We deal here with the interpretation of a series of PAC results, which is in disagreement with every other relevant published result on the question. In our opinion, this gives rise to a problem for the interpretation of PAC on this particular subject. But it is not justified to take it as a proof of its unique capabilities to tackle the subject. This positioning disregards every previous piece of work on the FOT *vs.* SOT classification of the $NdCo_2$ and $PrCo_2$ Curie transitions.

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