

Orthogonality point and intersublattice exchange in $\text{Dy}_2\text{Fe}_{17}$ determined in strong magnetic fieldsY. Skourski,^{1,*} M. D. Kuz'min,² K. P. Skokov,³ N. Shayanfar,³ A. V. Andreev,⁴ S. Zherlitsyn,¹ S. Yasin,^{1,†} L. Zvyagina,¹ O. Drachenko,⁵ O. Portugall,⁵ and J. Wosnitza^{1,6}¹*Hochfeld-Magnetlabor Dresden (HLD-EMFL) and Würzburg-Dresden Cluster of Excellence ct.qmat, Helmholtz-Zentrum Dresden-Rossendorf, 01328 Dresden, Germany*²*Aix-Marseille Université, IM2NP, UMR CNRS 7334, 13397 Marseille Cédex 20, France*³*Technische Universität Darmstadt, Institute of Materials Science, 64287 Darmstadt, Germany*⁴*Institute of Physics ASCR, Na Slovance 2, 18221 Prague, Czech Republic*⁵*Laboratoire National des Champs Magnétiques Intenses (LNCMI-EMFL), CNRS-UGA-UPS-INSA, 31400 Toulouse, France*⁶*Institut für Festkörper- und Materialphysik, Technische Universität Dresden, 01062 Dresden, Germany*

(Received 27 March 2025; accepted 23 May 2025; published 10 June 2025)

We report on magnetization measurements of single crystalline $\text{Dy}_2\text{Fe}_{17}$ in pulsed magnetic fields up to ~ 127 T. To produce the fields, we used (i) a single- and (ii) a double-coil magnet generating pulses in the 10 ms range, as well as (iii) semidestructive single-turn coils creating pulses of much shorter duration, ~ 5 μs . We observed steplike transitions at 64.3 and 77.7 T, for fields applied along [100] and [120], respectively. We confirmed the latter transition by ultrasound-velocity measurements using the double-coil magnet. Our data allowed us to locate the orthogonality point of $\text{Dy}_2\text{Fe}_{17}$ at ($\mu_0 H_{\perp} = 90$ T, $M_{\perp} = 31$ $\mu_B/\text{f.u.}$) and determine the intersublattice exchange field, $\mu_0 H_{ex} = 215$ T.

DOI: [10.1103/scy5-m3j8](https://doi.org/10.1103/scy5-m3j8)

I. INTRODUCTION

Rare-earth-iron (R -Fe) intermetallic compounds present a large class of magnetic materials with high application potential [1–4]. Iron-rich systems are particularly suitable for applications, for the obvious reason that the main constituent in the material is inexpensive and abundant. It is mainly the compounds containing light rare earths with iron that show strong magnetic performance, since the magnetic moments of the R and Fe sublattices are aligned, i.e., they add up. The application potential of R -Fe intermetallics with heavy rare earths, such as Dy used as anisotropy booster in $(\text{Nd,Dy})_2\text{Fe}_{14}\text{B}$ [5,6], is limited by the high cost and by the fact that the moments of the heavy rare earth are antiparallel to those of iron, which leads to a reduction of net magnetization. This fact, however, makes heavy R -Fe compounds attractive for basic research since it enables a direct measurement of the of R -Fe exchange—the interaction responsible for the antiparallel alignment of the R and Fe sublattice moments. For such a measurement, a strong magnetic field is needed to overcome the antiparallel alignment or at least to modify it significantly. To date, only pulsed-field magnets can produce such large fields.

$R_2\text{Fe}_{17}$ alloys occupy a special place among the iron-rich R -Fe intermetallics. They are model systems, owing to their existence as pure 2-17 binaries over the entire lanthanide series. Consequently, it was $R_2\text{Fe}_{17}$ with heavy rare earths, on which many research groups spent considerable efforts aiming at the determination of the intersublattice R -Fe exchange [7–13]. One should bear in mind the severe constraint imposed on such high-field studies by the fact that $R_2\text{Fe}_{17}$ (as well as other intermetallics) are good conductors of electricity. So it does not suffice that the applied magnetic field is strong enough to overcome the R -Fe exchange. For a successful high-field measurement it is also necessary that the field be changing slowly, in order to avoid unwanted eddy-current effects. As a matter of fact, the main limiting factor—the energy dissipated in a pulsed magnet—is proportional to the pulse duration times the square of the maximum field. So one faces a dilemma of the field either not being large enough to reach the sought anomaly or changing too rapidly and thus precluding any reliable magnetization measurement.

Much of the early work on the intersublattice exchange was carried out in long-pulsed, quasistationary magnetic fields not exceeding 40 T [14]. Since this field is not sufficient to modify the ferrimagnetic ground state of the $R_2\text{Fe}_{17}$ compounds, the early experiments were conducted on diluted materials such as $R_2\text{Fe}_{17-x}\text{Al}_x$, where the threshold fields are lower. The results were subsequently extrapolated to $x = 0$ (see Ref. [15] for a summary of early data). This approach was supplemented with an ingenious technique (the free powder method) that enabled the researchers to work without single crystals and to eliminate the effect of magnetic anisotropy by allowing the powder particles to freely rotate in the applied magnetic field, aligning their magnetic moments with the field direction.

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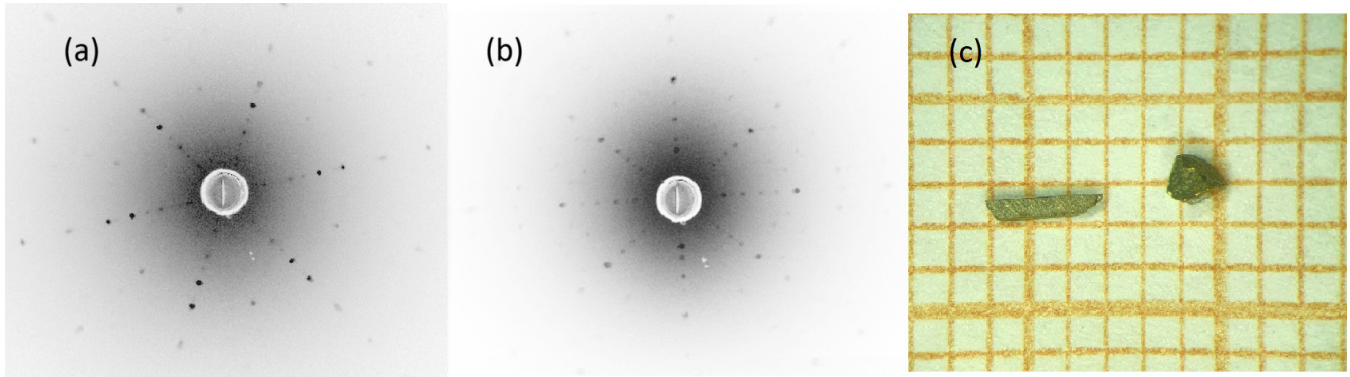


FIG. 1. (a), (b) Laue diffraction patterns of a $\text{Dy}_2\text{Fe}_{17}$ single crystal along the c and a directions, respectively. (c) Photograph of the crystals used in the experiment.

Following the advent of nondestructive 60 T magnets with pulse durations of around 10^{-2} s [16–18], we and other groups carried out measurements directly on single-crystalline samples, such as $\text{Ho}_2\text{Fe}_{17}$ [11], $\text{Er}_2\text{Fe}_{17}$ [12], and $\text{Tm}_2\text{Fe}_{17}$ [13]. However, materials such as $\text{Tb}_2\text{Fe}_{17}$ and $\text{Dy}_2\text{Fe}_{17}$, which are of interest due to their strong anisotropy, proved hard to deal with. The field required for the destruction of the antiparallel orientation of the Fe and R sublattice moments is proportional to $(1 - 1/g_J)$, which for the heavy lanthanides decreases with atomic number. Here, g_J denotes the Landé factor. This means, for example, that the threshold field in $\text{Tb}_2\text{Fe}_{17}$ ($g_J = 3/2$) is twice as large as in $\text{Er}_2\text{Fe}_{17}$ ($g_J = 6/5$). The difference is further enhanced by another, weaker effect—the falling dependence of the R -Fe exchange field (see Fig. 6) caused by the lanthanide contraction.

Here, we present magnetization measurements of $\text{Dy}_2\text{Fe}_{17}$ in fields up to 85 T produced by two different nondestructive magnets. We observe two first-order phase transitions for magnetic fields applied along the hexagonal a and b axes, i.e., along [100] and [120], respectively. We also present magnetization data up to about 130 T obtained using semidestructive coils and address the possibilities and challenges associated with such experiments.

II. EXPERIMENTAL

We prepared $\text{Dy}_2\text{Fe}_{17}$ by arc melting under a protective argon atmosphere using a stoichiometric mixture of 99.9%-pure metals in a triarc furnace on a water-cooled copper hearth. For better homogeneity, the alloy buttons were turned over several times and then kept in a molten state for about one hour. We grew the $\text{Dy}_2\text{Fe}_{17}$ crystal from those molten buttons using the Czochralski method at a pulling speed of 10 mm/h using a tungsten rod as crystallization nucleus. The obtained single crystal was about 4 mm in diameter and 25 mm long. X-ray back-scattering Laue patterns showed its high quality (see Fig. 1). For the magnetization measurements we used smaller pieces of this crystal that fitted in our pick-up coils. We determined the phase purity and lattice parameters by standard x-ray diffractometry on powder prepared from a piece of the crystal.

We carried out magnetization measurements using three different pulsed magnets. This comprises a single-coil nonde-

structive magnet (i) at Osaka University [19], which produced a field up to 68 T with a pulse duration of about 15 ms (see the red curve in Fig. 2). Further, we used a double-coil nondestructive magnet (ii) at the Dresden High Magnetic Field Laboratory (HLD) at the Helmholtz-Zentrum Dresden-Rossendorf generating field pulses as shown by the blue curve in Fig. 2. The generated field is a superposition of a broad (~ 100 ms) pedestal pulse produced by an outer coil and a much narrower (~ 10 ms) pulse produced by an inner coil [20]. Jones *et al.* explained the advantages of multiple-coil magnets [21].

Finally, we generated very high (close to 130 T), but also much shorter (about 6 μs , inset of Fig. 2) pulses using semidestructive single-turn coils (iii) at the Laboratoire National des Champs Magnétiques Intenses (LNCMI) [22]. The term “semidestructive” here means that the coil producing the field is destroyed during the pulse, while the sample, pick-up coils, and the cryogenic environment remain intact [23]. The used single-turn coils had an aspect ratio of 1:1 (length = diameter = 12 mm) presenting a compromise between the field strength and homogeneity. The pulse duration was set so that the peak current is reached just before the destruction of the coil. The coil was powered from a bank of ten 40 kV capacitors, each of them equipped with its own low-inductance rail-gap switch. The discharge was initiated by a very short (10 ns rise time) 100 kV pulse applied to the ignition contacts of the rail gaps.

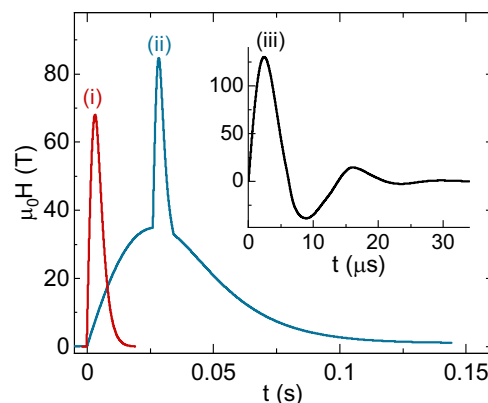


FIG. 2. Time-dependent magnetic fields generated by the three different pulsed-field magnets.

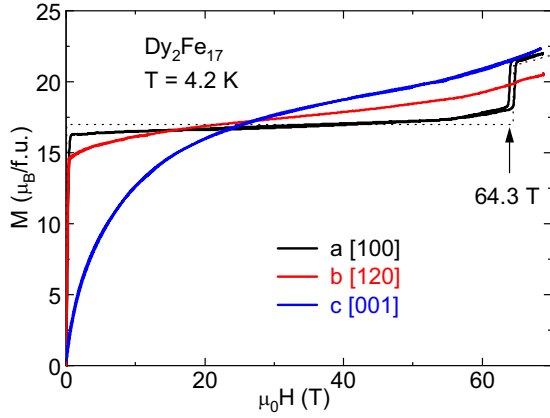


FIG. 3. Magnetization curves of $\text{Dy}_2\text{Fe}_{17}$ measured with field applied along the main crystallographic directions using the single-coil magnet (i). The dotted line represents simulation result.

In the nondestructive magnets, we measured the magnetization by means of the induction method, using coaxial pick-up coil systems similar to the one described in Ref. [11]. The magnetometer for the single-turn coils consisted of two identical pick-up coils located side-by-side and connected in series with their polarities opposite to each other.

For all magnetization measurements, both in the nondestructive and semidestructive coils, we made two equal-field pulses, one with and one without a sample in one of the coils. The background response of the empty coils was subtracted from the signal obtained with the sample in place.

We performed ultrasound measurements in the double-coil magnet (ii) using a pulse-echo phase-sensitive detection technique [24]. We carried out the experiments in longitudinal geometry, i.e., both the polarization vector $\mathbf{u}$ and the propagation vector $\mathbf{k}$ of the sound wave were parallel to the b axis. We glued two piezoelectric film transducers, working in the frequency range of 100 MHz, onto facets on two opposite sides of the sample. The magnetic field was also applied along the b axis.

All pulsed-field experiments reported in this work were carried out under nonisothermal, nearly adiabatic conditions. All indicated temperatures are initial values, measured just before the field pulse. These initial temperatures were set far below the Curie temperature, $T_c \approx 380$ K [25,26].

III. RESULTS AND DISCUSSION

We present our magnetization data measured in magnet (1) in Fig. 3. The magnetization for field applied along the hard direction c shows no spontaneous component, confirming the high crystal quality and correct orientation. For field applied along the easy axis a , we observe a spontaneous magnetization of about $17 \mu_B/\text{f.u.}$ followed by a nearly flat plateau, more than 50 T wide. For field applied along the b direction, we find a spontaneous magnetization of $14.8 \mu_B/\text{f.u.}$, which corresponds well to the projection of the easy-axis value onto b , reduced by a factor of $\sqrt{3}/2$.

We observe a steplike increase of the a -axis magnetization at a field of 64.3 T. Here, the applied magnetic field prevails over the intersublattice exchange interaction, making the Dy

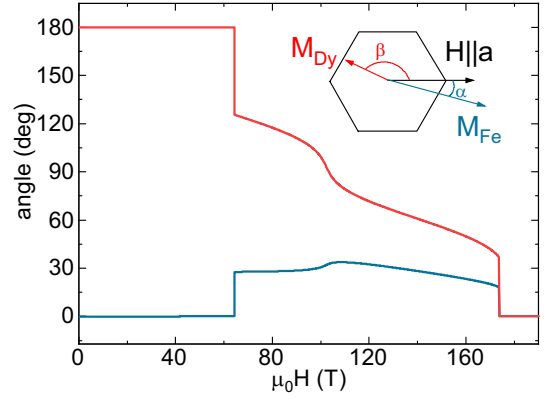


FIG. 4. Simulated evolution of the Fe and Dy sublattice angles with respect to the applied magnetic field.

sublattice moment abandon its low-field orientation antiparallel to the Fe moments and the applied field and turn to the next minimum in the sixfold anisotropy energy profile in the basal plane. The moment of the Fe sublattice reorients as well, deviating slightly from its initial orientation along the applied field. Interestingly, the step is preceded by a slightly increasing ramp starting at about 55 T. The evolution of the Fe and Dy sublattice angles with respect to the applied magnetic field is illustrated in Fig. 4. A similar ramp was previously observed in $\text{Ho}_2\text{Fe}_{17}$ [11] and $\text{Er}_2\text{Fe}_{17}$ [12], but not in $\text{Er}_2\text{Co}_{17}$ [27].

According to the model presented in Refs. [11,12], the b -axis magnetization should also exhibit a field-induced step. Obviously, the critical field of that transition is beyond 68 T. Hence, we extended the field range of our investigations to fields of up to 85 T by using the double-coil magnet (ii). This was also motivated by our aim to locate the so-called orthogonality point in the magnetization curve, where the R -sublattice magnetization makes a right angle with the applied field. At this point, the anisotropy of the sublattice effectively cancels out [28], which would enable us to determine the intersublattice exchange field. As shown in Ref. [28],

$$M_{\perp} = \sqrt{M_{\text{Fe}}^2 - M_{\text{Dy}}^2} \quad (1)$$

is the theoretical value of magnetization at the orthogonality point. The corresponding magnetic field, $\mu_0 H_{\perp}$, is what we want to determine experimentally.

We take the free-ion magnetization for the Dy sublattice, $M_{\text{Dy}} = 20 \mu_B/\text{f.u.}$ From the measured spontaneous magnetization of $M_s = 17 \mu_B/\text{f.u.}$ we can estimate the iron sublattice moment $M_{\text{Fe}} = M_s + M_{\text{Dy}} = 37 \mu_B/\text{f.u.}$ Equation (1) then yields

$$M_{\perp} = 31 \mu_B/\text{f.u.} \quad (2)$$

Considering the data shown in Fig. 3, it is obvious that we need magnetic fields way beyond 70 T to reach such a magnetization.

Therefore, we measured the b -axis magnetization in the 85 T double-coil magnet (ii). Discontinuities of the field-sweep rate in the double-coil magnet lead to a somewhat noisier signal. In particular, switching noise during the activation of the inner coil affects the recorded dM/dt ,

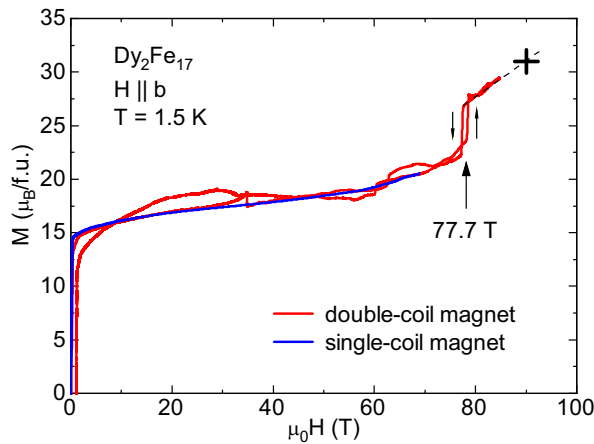


FIG. 5. Magnetization of $\text{Dy}_2\text{Fe}_{17}$ measured in pulsed fields applied along the b direction using the double-coil magnet (ii) in comparison with the single-coil (i) data already shown in Fig. 3. The dashed line shows a linear extrapolation of the data above about 78 T. The cross indicates the orthogonality point.

resulting—after integration—in some discrepancies. Nevertheless, the measured magnetization agrees well with that obtained in the single-coil magnet (Fig. 5). We clearly observe a magnetization jump (first-order phase transition) at about 77.7 T. Moreover, we now can estimate the orthogonality point by extrapolating the data above about 78 T to $\mu_0 H_{\perp} = 90$ T. Hence, we determine the exchange field on Dy,

$$\mu_0 H_{ex} = 2\mu_0 H_{\perp} \frac{M_{\text{Fe}}}{M_{\perp}} = 215 \text{ T}. \quad (3)$$

This is consistent with an earlier estimate [8]. Yet, our determination of H_{ex} is more direct, since it involves no fitting. The factor 2 in Eq. (3) stands for $g_J/2(g_J - 1)$ of Dy^{3+} , with $g_J = \frac{4}{3}$.

Figure 6 summarizes $\mu_0 H_{ex}$ in the R_2Fe_{17} series as a function of R . The values of $\mu_0 H_{ex}$ for $R = \text{Sm}$ [30] and Gd [31] were deduced from inelastic neutron scattering, whereas those for $R = \text{Dy}$ (this work) and for Ho, Er, and Tm [11–13] originate from pulsed-field magnetization measurements. The apparent kink in Fig. 6 is not related to the difference in experimental techniques, but rather to the structural

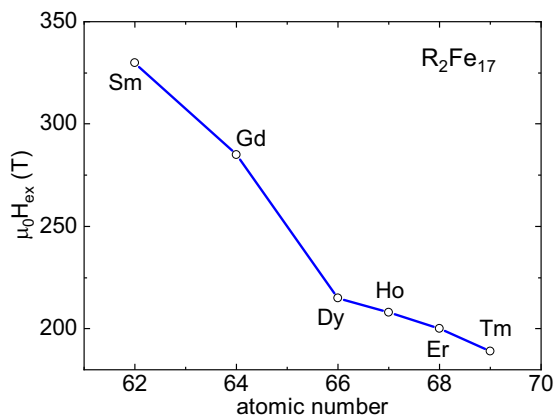


FIG. 6. Exchange field in R_2Fe_{17} vs the atomic number of R .

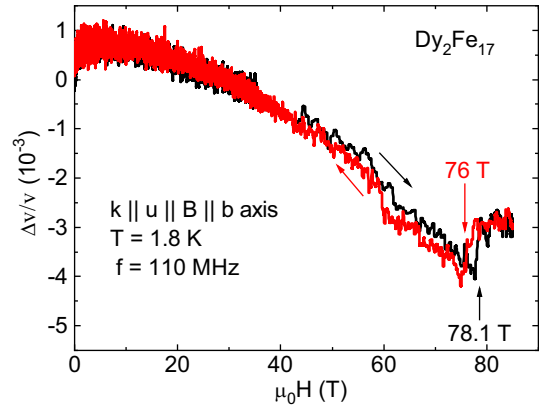


FIG. 7. Relative change of the longitudinal ultrasound velocity along the b axis of $\text{Dy}_2\text{Fe}_{17}$ measured for fields applied along b using the double-coil magnet (ii).

difference between $\text{Sm}_2\text{Fe}_{17}$ and $\text{Gd}_2\text{Fe}_{17}$ on the one hand (both $\text{Th}_2\text{Zn}_{17}$ -type structure), and $\text{Dy}_2\text{Fe}_{17}$ to $\text{Tm}_2\text{Fe}_{17}$ on the other hand (isomorphic to $\text{Th}_2\text{Ni}_{17}$).

To confirm the 77 T anomaly, we utilized the exceptional sensitivity of the ultrasound technique to phase transitions that couple to the lattice [29]. We measured the ultrasound velocity of the longitudinal acoustic mode c_{11} in $\text{Dy}_2\text{Fe}_{17}$ up to 85 T using magnet (ii). Figure 7 shows the relative change of the ultrasound velocity as a function of magnetic field applied along the b direction. We observed a remarkable softening of this acoustic mode with increasing magnetic field up to about 77 T followed by a jump at the phase transition also detected in the magnetization (Fig. 5). The phase transition is accompanied by a 2 T wide hysteresis, consistent with its first-order nature. Above the phase transition the sound velocity remains nearly constant. The significant decrease of the sound velocity by about 0.5% from zero magnetic field just before the transition and the notable jumplike anomaly (about 0.1%) at the phase transition evidence the strong magnetoelastic coupling in $\text{Dy}_2\text{Fe}_{17}$.

The model [11,12] predicts further magnetization jumps at even higher fields. Raising the upper field limit above 100 T implies the use of alternative magnetic-field generators, such as single-turn coils [22] producing very short pulses. This technique is generally not well suited for bulk metals, as their magnetic response is dominated by eddy currents. As a compromise, one can perform measurements on metallic powder embedded in an insulating matrix [32,33], but this is not suitable for strongly anisotropic materials such as $\text{Dy}_2\text{Fe}_{17}$. Encouraging news came recently from the Tokyo group [34], who had succeeded in measuring the magnetization of an YbB_{12} single crystal in a single-turn coil. Yet, YbB_{12} only becomes conducting (and anisotropic) above 55 T. Besides, the field applied to the single crystal in Ref. [34] was limited to 90 T. Still, we decided to try out the single-turn coil technique (iii) on single crystals of $\text{Dy}_2\text{Fe}_{17}$.

After testing several sample geometries, we selected a platelet-shaped crystal with dimensions of approximately $3 \times 1 \times 0.1$ mm. This geometry minimized eddy-current effects, while maintaining a reasonable sample volume and magnetic moment. The data acquired on this sample are

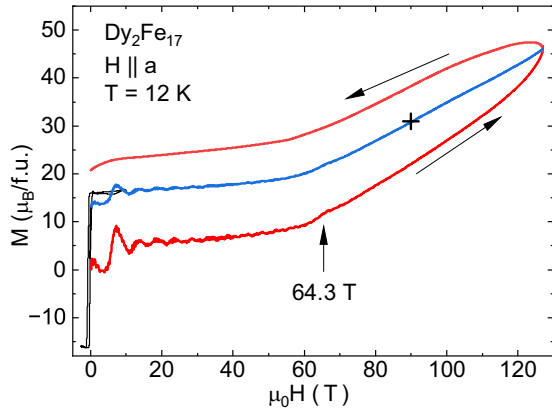


FIG. 8. Magnetization of $\text{Dy}_2\text{Fe}_{17}$ measured in pulsed field applied along the a direction using a single-turn coil (iii). The red line shows the data as measured, whereas the blue line is the result corrected for eddy currents (see text).

shown in Fig. 8 (red line). These data can be further processed based on the considerations discussed below.

The difference between the up- and down-sweep branches of the measured signals is an artifact due to eddy currents. We can minimize the eddy-current effect by averaging the two branches using the empirical formula,

$$M = wM_{\uparrow} + (1 - w)M_{\downarrow}, \quad (4)$$

with

$$w = 0.3 + 0.2 \frac{H}{H_{\max}}. \quad (5)$$

Thereby, $M_{\uparrow}$ and $M_{\downarrow}$ are the integrated pick-up signals measured during up and down sweep, respectively. The result of the averaging is the blue curve in Fig. 8. The weight function (5) was constructed on the basis of the following considerations.

The weight factor w should equal 0.5 at the maximum of the field range, $H = H_{\max}$. While this corresponds to a simple averaging of $M_{\uparrow}$ and $M_{\downarrow}$, it ensures the elimination of spurious effects in the curvature at the high-field end of the magnetization curve. However, this approach does not apply equally well across the entire field range due to the difference in sweep rates during the up and down sweep of the field.

At $H = 0$, w was set to 0.3. This value was chosen to account for the different sweep rates for increasing and decreasing field and to align the beginning of the blue curve with a reference measurement (black line near the origin of Fig. 8). We obtained this reference using the same pick-up coil setup and single-turn coil under nondestructive conditions by charging the capacitors to only 5 kV. This is significantly lower than the 40 kV used during the semidestructive measurements and, thus, less affected by spurious effects. We then linearly interpolated the weight $w(H)$ between $H = 0$ and $H = H_{\max}$.

Overall, M vs H in Fig. 8 reasonably reflects the expected and partially measured magnetization with the exception of the first ten teslas, where the signal is spoiled by the noise from the ignition pulse. Yet, this initial interval is covered by the reference curve. A more serious observation is the smearing out of the anomaly (magnetization step) at about 64 T, to the extent that it is no longer discernible in the down-sweep branch. The likely cause is an insufficient spatial homogeneity of the field produced by the single-turn coils. During the down sweep of the field, the field homogeneity is expectedly reduced, since then the coil is already significantly deformed. Nevertheless, by preparing a properly shaped sample and using a sophisticated pick-up coil system, we can reasonably well determine the magnetization in a metallic material in microsecond pulses to beyond 120 T. In the present case, for $\text{Dy}_2\text{Fe}_{17}$, this allows us to reproduce the orthogonality point at 90 T with reasonable accuracy (cross in Fig. 8).

IV. SUMMARY

By using state-of-the-art pulsed magnets and carefully designed pick-up-coil magnetometers, we were able to measure the magnetization of a metallic rare-earth-iron compound in very high magnetic fields. For that, we used nondestructive coils up to 85 T and a semidestructive single-turn coil to reach 127 T. Although the detected signal in the latter case showed some spurious effects caused by eddy currents and reduced homogeneity of the pulsed field, we still extracted reasonable magnetization data up to this extreme-field limit. This allowed us to determine directly field-induced first-order phase transitions in the magnetization of $\text{Dy}_2\text{Fe}_{17}$ at 64.3 T ($H||a$) and 77.7 T ($H||b$). We further found the orthogonality point in this metallic hard magnet, situated at $\mu_0 H_{\perp} = 90$ T with $M_{\perp} = 31 \mu_B/\text{f.u.}$ Hence, we determined the intersublattice exchange field on Dy, $\mu_0 H_{\text{ex}} = 215$ T.

ACKNOWLEDGMENTS

The work was supported by the Deutsche Forschungsgemeinschaft (DFG) through the Würzburg-Dresden Cluster of Excellence on Complexity and Topology in Quantum Matter—*ct.qmat* (EXC 2147, Project No. 390858490) and SFB 1143, by the ANR-DFG under Grant Fermi-NES, by the Czech Science Foundation under Project No. 25-16339S, by the Materials Growth and Measurement Laboratory (MGML [35]), and by Hochfeld-Magnetlabor Dresden (HLD) at HZDR and LNCMI, both members of the European Magnetic Field Laboratory (EMFL).

DATA AVAILABILITY

The data that support the findings of this article are not publicly available. The data are available from the authors upon reasonable request.

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