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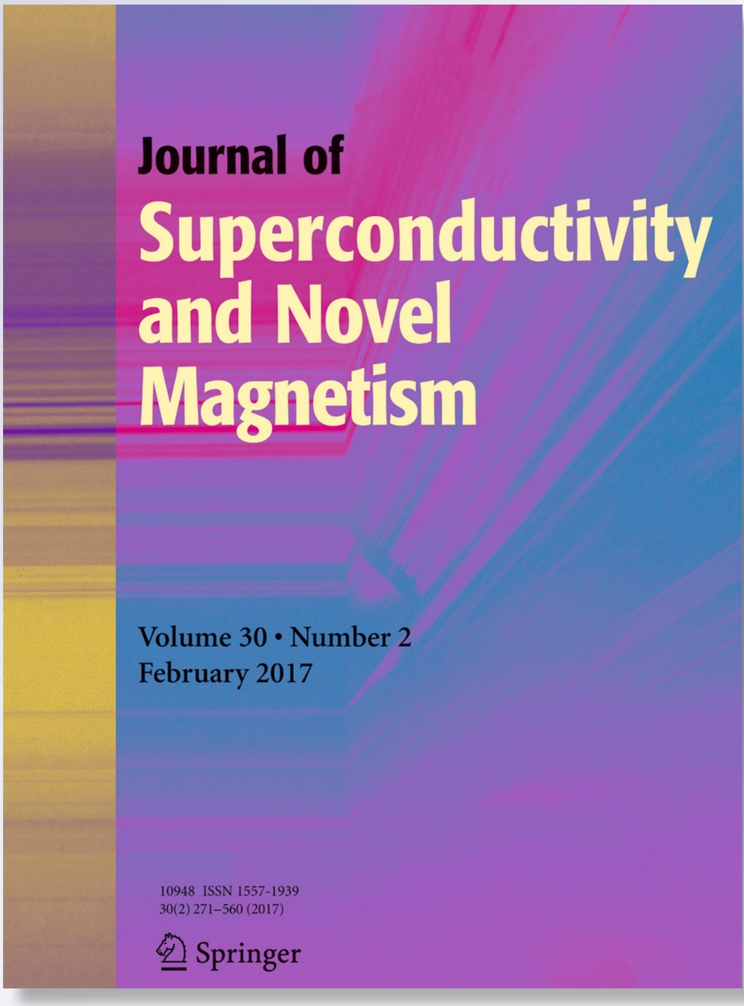

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ORIGINAL PAPER

Search for New Superconductors: an Electro-Magnetic Phase Transition in an Iron Meteorite Inclusion at 117 K

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Abstract The discovery of superconductivity in pnictides and iron chalcogenides inspires the search for new iron-based superconducting phases. Iron-rich meteorites present a unique opportunity for this search because they contain a broad range of compounds produced under extreme growth conditions. We investigated a natural iron sulfide-based material (troilite) inclusion with its associated minerals in the iron meteorite (Fe, Ni), Tlacotepec. Tlacotepec cooled over the course of 10^6–10^7 years in an asteroidal core under high pressure while insoluble sulfur-rich materials segregated into inclusions within the Fe–Ni core, synthesizing minerals under conditions not possible in the laboratory. The search for superconductivity in these heterogeneous materials requires a technique capable of detecting minute amounts of a superconducting phase embedded in a non-superconducting matrix. We used magnetic field modulated microwave spectroscopy (MFMMS), the most sensitive, selective, and non-destructive technique, to search for superconductivity in heterogeneous systems. Here, we report the observation of an electro-magnetic phase transition (EMPT) at 117 K that produces a unique MFMMS response. A pronounced and reproducible peak proves the appearance of an EMPT at 117 K. The temperature of this transition is not influenced by moderate magnetic fields up to 1400 Oe. Further, hysteretic isothermal field sweep loops are typical of the field sweep loops caused by flux trapping in high T_c superconductors. Although the compound responsible for the peak in the MFMMS spectra was not identified, our results indicate that it is a material heterogeneously distributed over the inclusion and possibly an iron sulfide-based phase.

Keywords Superconductivity · Extraterrestrial materials · Microwave absorption · Iron sulfide

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1 Introduction

The discovery of iron-based superconductors, a new class of superconducting compounds with an unconventional pairing mechanism, has rekindled the interest in the search for new superconducting materials [1–3]. Superconducting iron chalcogenides like undoped β-FeSe [4], $Fe(Se_{1-x}Te_x)$ [5, 6] or S-substituted FeTe [7] are of particular interest. The superconducting transition temperature T_c of FeSe can be significantly increased by applying high external pressure [8]. Several attempts were made to increase the transition temperature either by high pressure, high temperature synthesis, or by substituting selenium with sulfur that has a smaller atomic radius [9, 10]. However, in this study, we search for superconductivity in naturally occurring materials instead of synthesizing new compounds. This approach has two advantages: First, we can investigate materials that have crystallized under very extreme conditions like very long cooling time, high pressure, unusual chemical compositions, and high temperature, which are difficult or impossible to obtain in a laboratory. Second, due to the intrinsic inhomogeneity of these compounds, it is possible to investigate a large variety of material phases without the need to synthesize them.

The magnetic field modulated microwave spectroscopy (MFMMS) used in this study probes the magnetic field derivative of the electromagnetic absorption in a cavity at microwave frequencies. Because the electrical [11] and magnetic properties [12] change simultaneously at the critical temperature, all superconducting materials respond strongly in MFMMS-temperature scans. By contrast, most other materials do not respond at all even if they undergo a phase transition like ferromagnetic, anti-ferromagnetic, or metal-insulator transitions. However, in a few rare cases, other electro-magnetic materials, in which the microwave absorption can be influenced by a magnetic field (due to electro-magnetic effects or a change in skin depth), have a MFMMS response as well. In this manuscript, we use the rather broad term electro-magnetic phase transition (EMPT) to describe any phase transition (superconducting or not) detected by MFMMS.

MFMMS (also referred to as magnetic field modulated microwave absorption or differential/field-dependent microwave absorption) is an established technique [13–15] for detecting superconductivity, although it is not as common as the standard techniques. Because it is contactless, it is very useful for investigating fragile [16] or air-sensitive samples [17]. In a series of extensive studies, we have shown earlier that MFMMS has the unprecedented sensitivity of 10^{12} cc of a superconducting material embedded in a non-superconducting matrix [18, 19]. For these reasons, we chose MFMMS to search for traces of superconductivity in naturally occurring, inhomogeneous materials, such as meteorites and minerals.

Here, we report on an electro-magnetic phase transition (EMPT) at 117 K in an iron sulfide mineral extracted from the core of an iron meteorite, which causes a MFMMS response that is typical of a superconductor.

2 Materials

Iron meteorites are crystallized at the core of protoplanetary bodies under extreme pressure and temperature [20–25]. They predominately contain iron and nickel (15.9 %) and platinum (0.05 %) [22] but also contain a large array of minerals, such as pnictides. The Tlacotepec meteorite, which this study focuses on, is an iron IVB meteorite. It has an inferred kamacite nucleation temperature of 4750 °C and cooled at a rate of 500 °C per million years at a pressure below 1 GPa [22] with an internal temperature of more than 1487 °C, which provided fast planetary differentiation time scales. The presence of excess ^{107}Ag from extinct ^{107}Pd ($t_{1/2} = 6.5 \times 10^6$) during its solidification requires a time scale for formation and differentiation of less than 10^7 years [26–28]. During formation, sulfur-rich liquid continuously segregated under high pressure into droplets, forming inclusions. Troilite is the most common inclusion in the Tlacotepec meteorite with some inclusion sizes in the centimeter range. The meteorite also contains chromite, sphalerite, phosphate bearing sulfides, silica, chromium nitride, rare earth elements, and native copper [29]. These conditions offer a unique opportunity to search for the presence of superconductivity in unconventional, inhomogeneous natural systems, which does not exist in terrestrial materials. However, the inhomogeneity of these materials requires a sensitive and selective technique, to detect the presence of a superconducting phase.

3 MFMMS

MFMMS is based on a modified electron paramagnetic resonance spectrometer [18, 19]. The material under investigation is placed in a quartz tube mounted in a continuous flow Helium cryostat, which allows for changing the temperature between 3.5 and 300 K. The sample is positioned in a 9.4 GHz microwave cavity at the maximum (minimum) of the microwave magnetic (electric) field. A DC magnetic field (0–9000 Oe) is applied with an electromagnet. A pair of Helmholtz coils allows applying a collinear AC field between -100 and 100 Oe with a 100 kHz modulation frequency. For a typical MFMMS temperature scan, a small (15 Oe) DC field is applied together with a 15 Oe AC field modulated at 100 kHz, while the sample temperature is continuously ramped between two temperatures (across the expected T_c). The change in reflected microwave power from the cavity due to the AC magnetic field is read out via a lock-in technique.

The MFMMS response depends on the materials under investigation [18]. In particular, for superconducting materials in a DC magnetic field, as the temperature is swept through the T_c, the AC magnetic field forces the material periodically in and out of the superconducting state. This "derivative" type measurement gives rise to the characteristic MFMMS peak and the high sensitivity. This characteristic behavior was observed in many superconductors, independently of the type (elemental, A15, MgB_2, cuprates, etc).

Refer to [19] for a short introduction and to [18] for an extensive discussion of the MFMMS technique.

4 Results and Discussion

The MFMMS was designed, tested, and optimized to detect superconducting materials. A superconducting phase transition is indicated by a peak in the MFMMS temperature scans. For superconducting materials that effectively pin superconducting vortices the peak looks more like a step up with a small peak superimposed at the right, but in all cases it has a sharp right flank and its onset is at the transition temperature T_c.

The MFMMS was designed to suppress the response of non-superconducting materials, thus the spectra of the vast majority of non-superconducting materials are featureless. However, in a few rare cases (i.e., Mn doped GaAs), a peak in the MFMMS response is obtained due to magnetoresistive effects or due to a magnetic field induced change in the skin depth [18]. To discriminate superconducting materials from these rare cases, complementary criteria are used as follows:

(i) The response of all superconducting materials has the same sign. If a material produces a dip instead of a peak compared to a reference superconductor, then superconductivity can be ruled out.

(ii) Applied DC magnetic fields smaller than the upper critical field H_{c2} should have little effect on the MFMMS peak onset, which occurs at the superconducting transition temperature.

(iii) By considering the chirality of isothermal hysteretic field scan loops (FSLs), it is possible to distinguish the diamagnetic response of superconducting materials from the response of other magnetic materials. The chirality is clockwise for all known superconductors. By contrast, we only know of one published report [30] of clockwise chirality in a non-SC material. However, in this case, the clockwise chirality was probably caused by a switching event, which resulted in a sign change of the microwave signal. Furthermore, the overall shape of the FSLs is atypical of a superconducting material, meaning this signal can be distinguished from the FSL of a superconducting material despite the clockwise chirality.

Figure 1a shows a photograph of the sample under investigation. It is approximately one quarter of a Troilite inclusion from the iron meteorite Tlacotepec. We quarried out 0.5 cc of materials from the front side of the Troilite inclusion, crushed it in a mortar, and separated it into different samples for MFMMS analysis. Figure 1b shows a photograph of a sample tube (Tla-1).

Figure 1c shows the MFMMS-spectrum of the material at 15 Oe. The overall shape is an almost flat negative response from room temperature to 145 K followed by a rounded, s-shaped step down to 60 K. Most important, there is a small peak with an onset temperature of 117 K. This peak has the same sign as a reference superconductor MgB_2 that was added to one of the samples. At a higher magnetic DC offset field, the 117 K peak becomes more pronounced (Fig. 1d). We have investigated 22 different samples in total, 15 of which showed a peak with an onset temperature around 117 K in MFMMS with a DC field of 15 Oe (Fig. 1e). The repeatable and reproducible peak at 117 K in the MFMMS spectrum is indicative of an EMPT which satisfies criterion (i).

The shape of the background resembles the MFMMS spectra of Fe_3O_4 (magnetite) found in both synthetic powders and in micrometeorites [19]. In other materials though, similar magnetite-like MFMMS signal was absent with a predominantly flat background as in the case of Allende and Murchison meteorites and lunar and Martian rocks. The above-mentioned suggest that, in this sample, the background probably arises from traces of Fe_3O_4 [19]. Note that in an inhomogeneous material, it is very difficult to uniquely identify all possible backgrounds and their field dependence.

To investigate further the origin of the signal, we subdivided this sample repeatedly and found that similar peaks appeared in the subdivisions at the same temperature but of lower magnitude. This implies that the EMPT phase is distributed throughout the inclusion. Furthermore, the subdivided samples that do show a peak also comply with the complementary criteria mentioned earlier. However, in some of the subdivided samples, we observed a step up and step down background, which produced a peak in the combined sample. Though these samples showed small peaks as well, this could imply that some part of the peak we observe is created by the summation of step transitions.

A series of MFMMS spectra from the sample Tla-1, with increasing magnetic DC offset field are shown in Fig. 2. The peak onset temperatures remain constant for different DC offset fields, although the peak heights and the background change drastically. Further, the peak is largely suppressed above 1400 Oe applied field at which the background signal becomes dominant. The fact that the peak position is independent from the background is evidence that the peak and the background have different origins. We should stress that the intensity of the peak may be affected by the field dependence of that background and that we cannot rule out absolutely the possibility that the peak is caused by the superposition of signals from different compounds. This

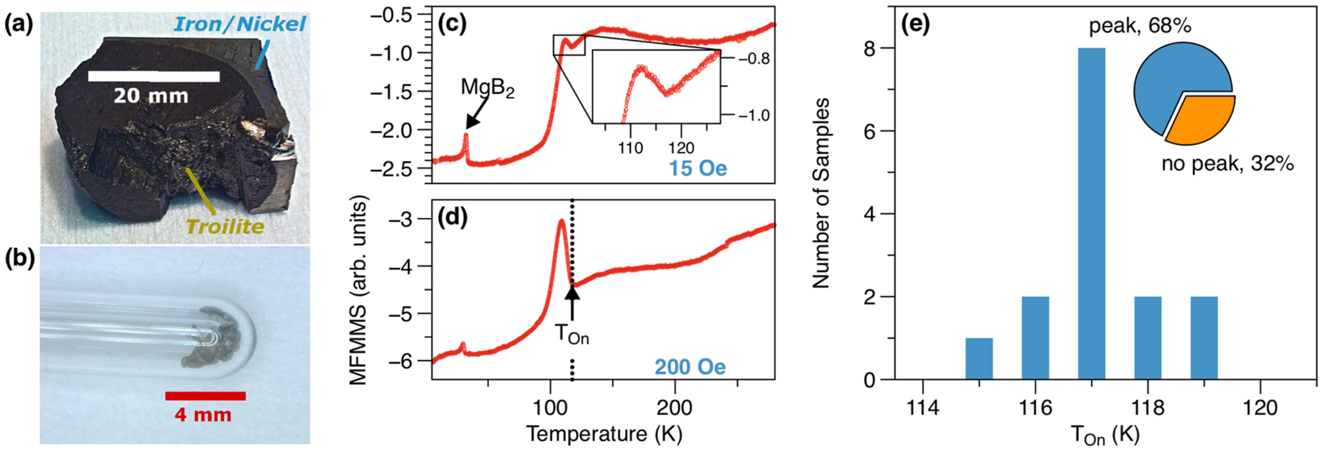

Fig. 1 Magnetic field modulated microwave spectroscopy (MFMMS) temperature scans of Troilite. **a** Photograph of the Tlacotepec Troilite inclusion under investigation. **b** Photograph of the sample Tla-1. **c**, **d** MFMMS of Tla-1 with different DC offset fields (*blue labels*). The onset temperature (T_{On}) is indicated by a *vertical dotted line*. **e** Distribution of T_{On} of different sample tubes. In total, 22 sample tubes were investigated and 15 had a peak in vicinity of 117 K in MFMMS with 15 Oe applied DC field

makes estimating the volume fraction of the material that causes the peak very difficult.

Because the MFMMS of a superconductor arises from changes of the penetrating magnetic flux induced by a small (15 Oe) magnetic AC field, it can be suppressed by a relatively small DC applied magnetic field (see chapter 4 in Ramírez et al. [18]). Note that the decrease in peak intensity with DC field is not due to the suppression of superconductivity. It is due to the fact that the relative change in the penetrating magnetic flux induced by the small AC field used (15 Oe) can no longer be detected by MFMMS. It is very likely that a superconductor with a T_c at 117 K would have a very high upper critical field H_{c2} (significantly above 20 T), as found in other known high T_c superconductors. Therefore, the data in Fig. 2 suggest that criterion (ii) also holds.

To investigate the chirality (as mentioned earlier), we have acquired hysteretic FSLs following a procedure with field cooling and asymmetric field scans similar to the ones used in cuprates and spin-glass studies [31–33]. The sample was cooled from 150 K to the intended set-temperature in a low 10 Oe DC field. After a 30 min wait for temperature stabilization, the field was set to −50 Oe and then two consecutive FSLs between −50 and 600 Oe were measured. Finally, the sample was heated to 150 K, which allowed the low field cool to the next set temperature.

Figure 3 summarizes the results from the FSLs method in the Tlacotepec Tla-1 sample. Figure 3a shows the MFMMS spectrum with a pronounced peak indicating an EMPT. We acquired FSLs at different temperatures above and below the peak. FSLs typical for a set temperature above the EMPT are shown in Fig. 3b: they have a small hysteresis, i.e., the up-sweep curve lies above the down-sweep curve, and the hysteresis of the second loop is slightly reduced. Both down-sweep curves coincide. Figure 3c, d show FSLs acquired at temperatures below the peak: the first up-sweep curve starts at larger values than the other curves, i.e., the first FSL is open, while the consecutive FSLs are closed. Hence, there is a training effect in the FSLs associated with the EMPT indicated by the peak in the MFMMS spectra. In order to clearly associate the EMPT with the unknown phase, the background signal above the transition was subtracted as shown in Fig. 3e, f. This is justified by the results showed in Fig. 2, which demonstrates that the peak and the background are of different origin, although if the FSLs of the background material changed drastically over this temperature range, this subtraction could be adding that difference to the response of the material causing the EMPT.

These background removed FSLs (BRFSLs) (Fig. 3e, f) are very similar to the spin-glass behavior observed in granular high T_c superconductors and magnetic systems [31–33]. Starting at negative field values, they increase to a maximum and develop a small, negative slope for higher positive fields. More importantly, the maximum of the up-sweep curve of the first BRFSL is at a lower field value than the maxima of the down-sweep curve and the other consecutive curves. Sastry et al. [32] compared the FSL characteristics of spin-glasses and vortex-glasses of granular high T_c superconductors. They found very similar FSLs in both cases with one clear distinction: for a superconductor, the maximum of the first up-sweep curve is at lower field values than the maximum of the down-sweep curve, while it is the opposite for a spin-glass. For superconductors, the difference in the field positions of the up-and down-sweep maxima was associated with flux trapping [34]. During the first up-sweep after cool down, magnetic flux is trapped in the superconductor and the maxima of the consecutive field sweeps are shifted to higher field values. For spin-glasses, the first up-sweep changes the spin configuration and the consecutive down-sweep curve is shifted to lower field values, as in every ferromagnetic system.

According to the Josephson Junction network model for superconducting clusters [31, 35], the position of the maximum in the first up-sweep curve is related to H_{c1}*, the magnetic field at which magnetic flux quanta start to enter the loops of the Josephson Junction network: $S = (\phi_0/2)H_{c1}*$, where S is the average surface area of the loops and ϕ_0 the magnetic flux quantum. In our study, the magnetic fields corresponding to the maxima in the first up-sweep curves are more than one order of magnitude larger than in Blazey et al. [31]. However, these fields correspond to loop areas of 0.04–0.07 μm^2 comparable to the 0.1 μm^2, which were determined by measuring Shapiro steps at YBCO samples [36], and could be realistic.

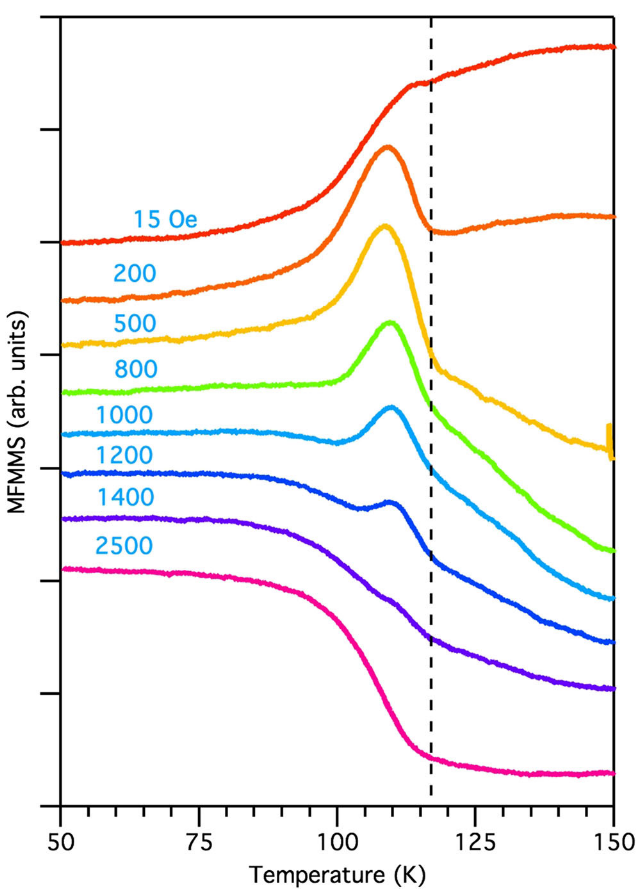

Fig. 2 Magnetic field modulated microwave spectroscopy (MFMMS) data of Tla-1, with different magnetic DC offset fields (*blue labels*). The spectra are shifted vertically for clarity. The *vertical dashed line* at 117 K is a guide to the eye that indicates the onset temperature

The BRFSLs in Fig. 3e, f have a clockwise chirality and satisfy criteria (iii). Although the chirality criteria is not universal, it is unlikely that a non-superconducting material shows all the characteristics of a granular high T_c superconductor in such a complex cooling and field sweep sequence.

Several caveats should be emphasized regarding the possibility of this being evidence for superconductivity in a meteorite.

(i) In all MFMMS temperature scans to date, the peak was on a background which varied from sample to sample. This implies that the inclusions consist of a variety of material phases with different microwave responses. We cannot exclude that the background deforms the peak considerably. However, it is safe to assume that the peak is associated with an EMPT for following reasons: First, the peak has a sharp onset and is reproducible for different samples. Second, in the temperature scan series with varying magnetic DC fields (shown in Fig. 2), the onset position is constant, while the peak height varies continuously, and third, the training effect indicated by the gap in the FSLs (shown in Fig. 3) is associated with the peak in the temperature scans.

(ii) Beside the MFMMS, we have used SQUID and AC susceptibility to characterize the magnetic response. Although these measurements provide no evidence, the MFMMS provides clear indications for an EMPT in vicinity of 117 K. The lack of EMPT signature in some measurements is similar to earlier studies of very small superconducting fractions in lithographically prepared [18] and in small quantities of superconducting powders mixed into mostly magnetic samples [19]. In both of these cases, the sensitivity of the MFMMS was much higher than SQUID magnetometry.

(iii) Since the chemistry of meteorites is very complex, it is possible that they contain a superposition of several unidentified, unknown phases, which mimic the response of a superconductor. However, we have performed extensive exploration of many different materials system and have not observed a superposition of non-superconducting phases that mimic a superconductor as well as these samples do.

We are presently using a "divide and conquer" methodology, in which the sample is subdivided multiple times until the response becomes unique. These measurements presumably will permit to isolate the material phase causing the peak. Then, it will be possible to identify the responsible compound and to test for zero resistance and the Meissner effect.

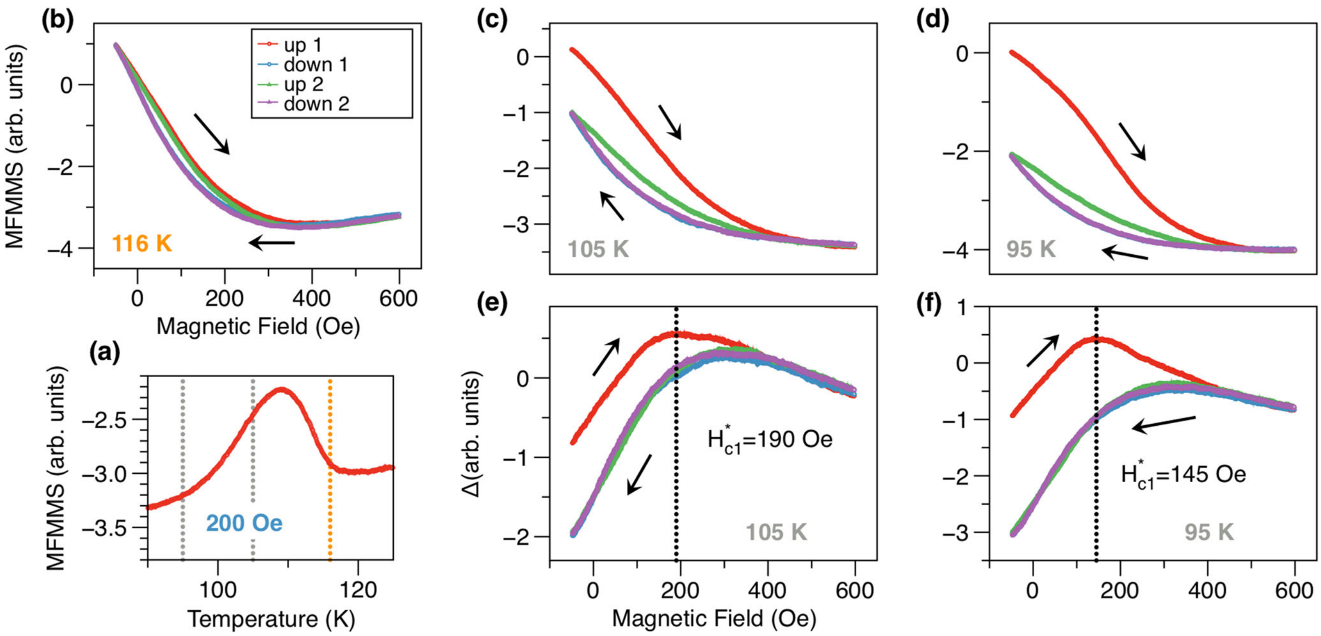

Fig. 3 Field scan study of Tla-1. **a** MFMMS-spectrum, *blue label* indicates DC offset field, *vertical lines* indicate set temperatures of field scan loops (FSLs). **b–d** Two consecutive FSLs acquired at set temperatures indicated by labels. **e–f** Background removed FSLs (BRFSLs): FSLs at 116 K (**b**) subtracted from FSLs at 105 and 95 K, respectively. The *dotted vertical lines* indicate the maxima of the first up sweep curves ($H_{c1}{}^*$)

5 Conclusions

Using the selective, non-destructive, highly sensitive MFMMS technique, we have identified an EMPT at 117 K in a Troilite (iron sulfide) sample, which originates from an inclusion of the iron meteorite Tlacotepec. Further, the EMPT complies with three MFMMS criteria indicative of a superconducting transition: (1) The MFMMS response has the same sign as a superconductor. (2) Moderate magnetic fields up to 1400 Oe do not shift the peak onset in the temperature scans. (3) The chirality of the isothermal BRFSLs is clockwise indicating the diamagnetic response of a superconductor and the BRFSLs are very similar to FSLs of granular high Tc superconductors reported by others. However, as stated previously, these signatures could be produced by a combination of background signals; so, until we can isolate the phase or phases responsible for this signal, we will not be able to measure and determine the signal's origin with certainty.

We could not detect a magnetic transition at 117 K using SQUID or AC susceptibility measurements. Regardless of origin, a phase transition detected in MFMMS is always of magnetic nature. Therefore, if the phase transition cannot be detected via SQUID or AC susceptibility measurements, it is below the sensitivity threshold of those systems. This implies that the EMPT occurs only in a small volume fraction of the materials under investigation. Because we have detected an EMPT in 68 % of the investigated samples, we know that this volume fraction is distributed over the whole inclusion.

Preliminary energy-dispersive x-ray spectroscopy indicates that the materials under investigation consist predominantly of iron and sulfur. However, it is well known that Troilite minerals contain a variety of trace elements so we cannot determine absolutely what materials are causing the EMPT.

A variety of iron chalcogenides material phases were reported to be superconducting, and during the publication process of this manuscript, we have learned of a superconducting FeS phase with a transition temperature of 5 K [37]. Therefore, it is possible that a small superconducting material phase has formed in the meteorite inclusion under the extreme growth conditions, and taking into account that superconducting iron chalcogenides are very sensitive to a variation in stoichiometry [38], it is plausible that this material phase is only a very small volume fraction in the Troilite inclusion.

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Authors' Contributions This is a highly collaborative research. I.K.S. generated the idea to develop the MFMMS for the systematic search for superconductivity. The equipment was set up and tested by I.K.S., A.C.B. and J.G.R.. I.K.S. started the collaboration with M.T., who provided the samples originally obtained from the Field Museum of Natural History. M.T. suggested the Tlacotepec materials to search for superconductivity. S.G. generated the idea to search for superconductors in extraterrestrial materials and made most of the measurements and data analysis. In particular, S.G. discovered the similarities with the field sweep studies of spin-glasses and granular superconductors. S.G. wrote the first version of the manuscript. J.G.R., A.C.B., and J.W. contributed to the measurements and data analysis. S.G., J.G.R., A.C.B., J.W., M.T., and I.K.S. interpreted the results and wrote the manuscript.

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