I. INTRODUCTION

Topological insulators (TIs) are 3D materials with a band gap in the bulk-electronic structure but with gapless, delocalized, surface states. The surface-states are protected by time-reversal symmetry (TRS), and cannot be gapped without changing substantially the bulk-electronic structure. This special relation between the bulk and surface states is the basic difference between these systems and systems showing ordinary surface states. The protected surface states are believed to be robust to disorder, interactions, and thermal fluctuations, potentially leading to room-temperature de-vice applications, potentially leading to room-temperature de-

In this context, an important issue is the stability of the TI surface states to the presence of impurities.

In the bulk, it was shown that a TI with magnetic impurities can have long range magnetic order in the metallic and insulating phases. On the surface, long range order can also be formed independent of the bulk magnetic ordering, as the RKKY interaction induced by the Dirac fermions is generally ferromagnetic when the Fermi energy ($E_F$) is close to the Dirac point. Both effects can lead to breaking of TRS, resulting in a gap opening at the Dirac point that makes the surface Dirac fermions massive. Chen et al. have shown that the Dirac gap can be observed in magnetically doped samples with or without bulk ferromagnetism. Furthermore, it was found that $E_F$ can be tuned into the surface-state gap which in turn may allow the observation of many interesting topological phenomena, such as the image magnetic monopole induced by an electric point charge. Theoretical work on magnetically doped TIs has also been very active. These studies predict a variety of effects due to the RKKY interactions between different magnetic impurities mediated by the surface states. For example, Zhu et al. predict that such interaction consists of Heisenberg-like, Ising-like, and Dzyaloshinskii-Moriya-like terms. They devise a new way to control surface magnetism using electrical field or even by changing the average spacing between different impurities at the surface, e.g., by changing their doping level. However, most importantly this surface magnetism is predicted to be dramatically different from the bulk due to the different type of magnetic interactions at the surface.

In this paper we investigate magnetically (Fe) doped Bi$_2$Se$_3$, at different doping levels. Using zero field (ZF) muon spin relaxation ($\mu$SR) and bulk magnetization measurements, we find that even at low doping levels, the full volume of the material becomes magnetic at a relatively high temperature (∼250K). We identify two magnetic regimes; the first between ∼10 and 250K and the second below ∼10K. The nature of the magnetism in both regimes, as well as the effective size of the magnetic moment per doping Fe, are independent of the doping level. However, the dynamics due to magnetic fluctuations increase at lower doping. Moreover, near ∼250K, the size of local magnetic field at the muon site increases more sharply as the temperature is decreased for higher Fe doping. These results indicate that Fe-Fe interactions dominate the magnetism at high temperatures, while the single Fe ion properties dominate in the low temperature regime. These studies form a basis for future studies of the magnetism near the surface using depth resolved low energy $\mu$SR, which may shed light on the interplay between magnetism and topological surfaces states in this important class of materials.
II. EXPERIMENTAL

Single crystals of Bi$_{2-x}$Fe$_x$Se$_3$, with $x = 0, 0.1, 0.2$ and 0.3, were grown from a melt using the Bridgman method. Corresponding amounts of Bi, Fe, Se of minimum purity 99.99% were mixed and sealed in evacuated silica ampules. The ampules were annealed at 820°C over 10 h for homogenization. The melt was then cooled down to 640°C at a rate of 4°C/h and then quenched into cold water. Well-formed silvery crystal rods were obtained, which could be easily cleaved into plates with flat shiny surfaces. The magnetization measurement reported here were obtained using a Physical Properties Measurement System (PPMS, Quantum Design).

The Bi$_{2-x}$Fe$_x$Se$_3$ ($x = 0.2, 0.3$) crystals were characterized at room temperature by powder X-ray diffraction (XRD) using a D8 Advance Bruker AXS diffraclometer with Cu K$_\alpha$ radiation. Additionally, the Bi$_{2-x}$Fe$_x$Se$_3$ ($x = 0.2$) crystal was studied by means of neutron powder diffraction (NPD) at the SINQ spallation source at the Paul Scherrer Institute (PSI, Switzerland), using the high-resolution diffractometer for thermal neutrons HRPT [2] (wavelengths λ = 1.494 Å and 1.155 Å). The NPD patterns were measured at temperatures in the range 2 – 300 K. For the diffraction measurements, part of the crystal was powderized and loaded into a vanadium container with an indium seal in a He glove box. Refinement of the crystal structure parameters was done using FULLPROF software [15], with its internal tables for neutron scattering lengths.

The μSR experiments were performed on the DOLLY spectrometer at PSI, Switzerland. In these experiments 100% polarized (along the beam direction, $z$) positive muons are implanted in the sample. In our measurements single crystals were suspended on an aluminized Mylar tape and mounted into a He gas flow cryostat. The crystals were aligned such that the initial polarization of implanted muons is along the $c$-axis (and the applied field in non-zero field measurements). Each implanted muon decays (lifetime $\tau_\beta = 2.2$ μsec) emitting a positron preferentially in the direction of its polarization at the time of decay. Using appropriately positioned detectors, one measures the asymmetry of the muon beta decay along $z$ as a function of time $A(t)$, which is proportional to the time evolution of the muon spin polarization. $A(t)$ depends on the distribution of internal magnetic fields and their temporal fluctuations.

III. RESULTS

A. Magnetization measurements

In Fig. 1 we show the magnetic susceptibility, $\chi$, as a function of temperature. In this figure we present both zero field cooled (ZFC) and field cooled (FC) measurements in a magnetic field of $B = 100$ mT applied perpendicular to the crystallographic c-axis ($B \perp c$). The mother compound Bi$_2$Se$_3$ gives a small diamagnetic signal with almost no temperature dependence. In contrast, Fe doping makes Bi$_{2-x}$Fe$_x$Se$_3$ magnetic below $\sim 250$K, confirmed by a clear split between the ZFC and FC susceptibility. The magnetic nature seems to change at temperatures below $\sim 10$ K, where a small abrupt upturn is observed for all doping levels measured (see insets of Fig. 1). Similar behaviour is observed in measurements with the magnetic field applied parallel to the $c$-axis ($B \parallel c$), but with a more pronounced upturn below $\sim 10$ and smaller $\chi$ values in general.

Measurements of the hysteresis loops in all four samples are shown in Figs. 2 (B $\perp c$) and Fig. 3 (B $\parallel c$). These were measured at two different temperatures; 2 and 35 K to better show the difference above and below 10 K. The diamagnetic and temperature independent nature of the mother compound Bi$_2$Se$_3$ ($x = 0$) is evident in the absence of hysteresis and the negative slope of $M$ vs. $B$ yielding $\chi \sim -1^{-6}$ emu/g. However, for all doped samples we observe a clear opening of the hysteresis when the field is applied perpendicular to the $c$-axis (Fig. 2). In contrast, we find that the hysteresis loops are much narrower when the field is applied along the $c$-axis (Fig. 3). This strong anisotropy indicates that the magnetic easy axis in Bi$_{2-x}$Fe$_x$Se$_3$ lies within the Bi layer and almost perpendicular to the $c$-axis. Furthermore, the hysteresis loops seem to be narrower below 10 K pointing to the possibility of a different orientation of the magnetic easy axis between the high and low temperature regimes.

Note, in the $B \perp c$ measurements (Fig. 2) the remanence depends only weakly on temperature, but the saturation of the magnetization differs significantly. In addition, we find that the magnetization saturates gradually.
FIG. 2: The magnetization as a function of magnetic field ($B \parallel c$) for the different Fe doping. The red squares and blue circles are measurements at $T = 2$ and 35 K, respectively. The solid lines are a guide to the eye.

FIG. 3: The magnetization as a function of magnetic field ($B \perp c$) for the different Fe doping. The red squares and blue circles are measurements at $T = 2$ and 35 K, respectively. The solid lines are a guide to the eye.

magnetic inclusion of Fe-Se binary compounds.

B. NPD and XRD measurements

Both room-temperature NPD and XRD experiments show that the samples consist mainly of the rombohedral Bi$_{2-x}$Fe$_x$Se$_3$ phase (space group R-3m). However, we also detect a small impurity phase of Fe$_3$Se$_4$ (about 5% mass), which has a monoclinic structure and orders ferrimagnetically below $T_c \sim 314$ K. Difference Fourier density map analysis reveals that Fe substitutes Bi atoms in the structure. The refined Fe occupancy is in agreement with the nominal composition, but the deduced values contain large systematic errors. This is due to the presence of the small impurity phase and a strong preferred orientation of the flat-shape crystallites in the powder along (001)-direction. This in turn affects their packing and the resulting diffraction patterns seen in both XRD and NPD. We account for this artifact by using the March formula in the FULLPROF refinement analysis. Note, since the scattering lengths of Bi and Fe are quite close (8.53 fm and 9.45 fm, respectively), NPD is not very sensitive to the Bi/Fe-occupancy ratio. Therefore, their sum was simply fixed at 2. High statistics NPD data, which were collected at two different temperatures 2K and 20K, do not exhibit any statistically significant difference due to possible magnetic contribution. This may be due to a small magnetic moment on the Fe ions or due to a disordered nature of the magnetic phase in these compounds. The crystal structure parameters for the main phase refined from XRD (300K) and NPD (2K and 300K) are presented in the Table[1]

C. $\mu$SR measurements

Compared to conventional dc magnetization measurements, $\mu$SR is a much more sensitive method to investigate the magnetism of these compounds. It also has two very important advantages: (I) detecting intrinsic magnetism even in zero applied magnetic fields and (II) since it is a local probe method, the contribution of impurity phases is proportional to their volume fraction and can be easily disentangled from the contribution of the studied compound. Typical $\mu$SR asymmetries measured in the $x = 0.2$ sample are shown in Fig. 4. Similar behaviour was observed in the other two Fe doped compounds. Note, in zero applied field, the asymmetry relaxes to finite value. A priori, one cannot determine the source of this relaxation. It can be due to either a distribution of internal static fields, dynamic fields, or a combination of the two. However, it is evident in Fig. 4 that application of an external magnetic field along the initial direction of the muon spin (so-called longitudinal field or LF-$\mu$SR) decouples the relaxation. This decoupling, together with the appearance of “wiggles” in the relaxation curves when the field is applied, are evidence
|                  | XRD (300K) | NPD (300K) | NPD (2K) |
|------------------|------------|------------|----------|
| a (Å)            | 4.14072(10)| 4.13914(4) | 4.12518(3) |
| c (Å)            | 28.64692(60)| 28.63151(56)| 28.47718(40) |
| Bi (0, 0, 0)     | 0.40057(6) | 0.40101(14)| 0.40125(9) |
| Bi(l) (Å²)       | 0.997(52)  | 1.743(67)  | 0.498(45) |
| Se2 (0, 0, 0)    | 0.20984(10)| 0.21071(8)  | 0.21062(6) |
| Bse2 (Å²)        | 3.173(144) | 1.393(97)  | 0.745(65) |
| Rp, Rwp, Rexp    | 6.55, 8.46, 3.77 | 3.80, 4.75, 3.65 | 3.46, 4.46, 2.00 |
| χ²               | 5.04       | 1.69       | 4.99     |

TABLE I: Crystal structure parameters of Bi$_2$–xFe$_x$Se$_3$ (x = 0.2) obtained from XRD (300 K) and NPD (300 and 2 K) refined in the space group R-3m (no. 166) with Se1 in (3a) position (0, 0, 0), Bi and Fe in (6c) position (0, 0, z), and Se2 in (6c) position (0, 0, z). B$_{Bi}$ and B$_{Se2}$ are the atomic displacement parameters of Bi and Se2 atoms, respectively. $R_p$, $R_{wp}$ and $R_{exp}$ are the reliability factors and $\chi^2$ is the global chi-square (Bragg contribution).

![Figure 4](image_url)  

**FIG. 4:** The asymmetry measured in the $x = 0.2$ compound with different applied longitudinal magnetic fields and temperatures: (a) 35K, (b) 5K and (c) 1.6K. The lines are fits to Eq. (1).

...of the quasi-static nature of the local fields experienced by the implanted muons, i.e., the local field is predominantly static with a smaller dynamic component. In this case the relaxation can be described using a phenomenological function; a Lorentzian Kubo-Toyabe (LKT) multiplied by an exponential. The LKT accounts for the relaxation due to the distribution of static fields while the exponential accounts for the depolarization due to the small dynamic component. The asymmetries in Fig. 4 were fit to a function of the form

$$A(t) = A_0 P_{LKT}(B, \Delta, t)e^{-t/T_1},$$  

(1)

where $A_0$ is the initial asymmetry, $P_{LKT}(B, \Delta, t)$ is the depolarization function due to a Lorentzian distribution of static fields with width $\Delta$ in the applied field $B$, and $1/T_1$ is the spin lattice relaxation rate due to the fluctuating transverse magnetic field component at the muon site. In order to obtain a reliable fit in Fig. 4, we assume a temperature and field independent value of $A_0$ and a field independent (but temperature dependent) value of $\Delta$, with only $1/T_1$ allowed to vary as a function of the applied field. These assumptions produce reliable fits and very good agreement with the measured asymmetries. Note that Eq. (1) account for the full signal, and therefore no impurity phase (e.g. Fe$_3$Se$_4$) contribution is detected in our measurements. This could be due to the small volume fraction of this phase and its correspondingly small contribution to our measured signal. Alternatively, the single crystals used for these measurements, which were cleaved from large crystal rods, are free from impurities.

Typically, the muon spin depolarization function due to a randomly oriented quasi-static local magnetic field exhibits a dip at early times, followed by a recovery to $\sim 1/3$ of the initial asymmetry which continues to relax in an exponential-like manner at longer times. The absence of a clear dip in the ZF measurements is due to the relatively small value of $\Delta$ ($\sim 1$MHz) and the dynamic component of the local field. This makes it difficult to fit reliably all our data to Eq. (1). Instead we parametrize the temperature dependence in all three Fe doped compounds using a function of the form,

$$A(t) = A_0 \left( \frac{2}{3} e^{-\lambda t} + \frac{1}{3} e^{-t/T_1} \right).$$  

(2)

Here, $\lambda$ is a parameter proportional to $\Delta$ (approximately $\lambda \sim 2\Delta$) and quantifies the width of the static field distribution. From these fits we extract the values of $\lambda$ and $1/T_1$ as a function of temperature shown in Fig. 5 for the three Fe doped samples. Note that, in agreement with the magnetization measurements, we observe a large increase in $\lambda$ below $\sim 250$ and $\sim 10$K. These coincide with broad and weak peaks in the values of $1/T_1$, indicating that the Fe doped Bi$_2$Se$_3$ undergoes two different types of magnetic ordering at these temperatures. However, while $\lambda$ below $\sim 250$ K seems to depend strongly on the doping level, it is almost independent of $x$ below $\sim 10$ K. In fact, the main difference between the doping levels appears just below 250 K, where the increase in $\lambda$ is sharper for higher doping. The spin lattice relaxation rate, $1/T_1$, depends more strongly on $x$. Its value increases with decreasing $x$, indicating that the fluctuations of local fields...
are faster for lower doping. In all three doping levels a sharp peak is observed at \( \sim 5 \) K, which we attribute to the magnetic phase transition below \( \sim 10 \) K. In contrast, the peak at high temperature is broader for all \( x \) values and it seems to shift to lower temperatures for larger Fe doping. We attribute this peak to the magnetic phase transition occurring below \( \sim 250 \) K. The width and quality of the results do not allow accurate determination of the exact transition temperature, nevertheless, they indicate a more disordered and possibly inhomogeneous magnetic transition (typically seen in dilute spin glasses).\(^{23}\)

In contrast to the doped samples, the muon spin relaxation rate in the mother compound is very small and has a very weak temperature dependence. Fitting the relaxation to an exponential function gives a rate of \( \sim 0.008 \) \( \mu s^{-1} \) (bottom panel of Fig. 5). The only source of relaxation in this case is expected to be dominated by the Bi nuclear magnetic moments. However, the observed relaxation is much smaller than one expects from dipolar fields of nuclear moments and therefore strongly indicates that the muons stopping sites are far away from Bi. In our case, the muons are most probably located in the van der Waals gap between layers of \( \text{Bi}_2\text{Se}_3 \).

\[ \text{FIG. 5: The temperature dependence of } \lambda \text{ (bottom) and } 1/T_1 \text{ (top) obtained from the ZF } \mu \text{SR data for the different Fe doping.} \]

\[ \text{FIG. 6: The saturation magnetization as a function of } x, \text{ the Fe doping level at } 35 \text{K and } 2 \text{K. The dashed lines are a linear fit of the data.} \]

\[ M_{\text{sat}} \text{ as a function of } x \text{ we find that the contribution of each Fe ion to the magnetization is equal (within error) at both temperatures and amounts to } 1.91 \pm 0.06 \text{ emu/g. From } M_{\text{sat}} \text{ we estimate a magnetic moment per Fe of } 0.34(7) \text{ and } 0.25(4) \text{ } \mu_B \text{ at } 2 \text{ and } 35 \text{K, respectively. Note, however, this contribution must decrease for much lower doping in order to approach the non-magnetic } x \to 0 \text{ limit.} \]

The \( \mu \text{SR} \) results, summarized in Fig. 6 show that the width of the static field distribution (or \( \lambda \)), which is proportional to the local magnetization, depends only weakly on \( x \) below \( \sim 50 \) K. Since the muon is a local probe it is mostly sensitive to the dipolar field from its nearest neighbour magnetic moment. Therefore, the \( x \) independent \( \lambda \), together with our previous conclusion regarding the \( x \) independent size of Fe magnetic moment, imply that the implanted muons stop at a similar average distance from a neighbouring Fe in all samples. However, one expects that the implanted muons have higher probability to stop closer to a Fe ion for larger \( x \). This contradiction may be explained if the muon stops within the van der Waals gap in the material, as we concluded from the small relaxation rates measured in the mother compound. In such a case the distribution of dipolar fields experienced by the muons from the Fe ions, which occupy a site in the Bi layer, does not depend strongly on \( x \). The width of this distribution can be calculated\(^{23}\) by distributing \( x/2 \) Fe ions randomly on a monolayer of a triangular lattice (of Bi, lattice constant \( a \)), and summing up their contribution on muons which are located 4.76 Å away from it (within the van der Waals gap). Although the proposed model is very simple, we find that for an average Fe moment of \( 0.34\mu_B \) we obtain values of \( \lambda \) which are consistent with the experimental results.

The measurements presented here indicate that the low and high temperature magnetic phases differ in the underlying interactions. The high temperature regime seems to be associated with Fe-Fe coupling, and therefore depends on \( x \) which affects the average distance be-
tween Fe ions. In contrast, the weak x dependence at low temperature is strong evidence that the magnetization is dominated by the Fe single ion properties and its interaction with the Bi$_2$Se$_3$ lattice, e.g. crystal field etc. Hence, as the temperature is decreased the smaller single ion interaction with the lattice becomes more relevant, altering the high temperature size and ordering of the Fe moments. This is consistent with the strong anisotropy detected in magnetization measurement, which show a strong tendency of the Fe moments to align perpendicular to the c-axis, especially at low temperatures. Moreover, considering the low density of Fe ions, and therefore the fairly long range (a few a) magnetic Fe-Fe interaction, it is most probably mediated by charge carriers which extend over these length scales. Such effects have been observed in Fe doped Bi$_2$Te$_3$, though with magnetic transition at much lower temperatures (< 15 K$^{23}$) which also depend strongly on x. This was attributed to donor type Fe doping in both Bi$_2$Te$_3$ and Bi$_2$Se$_3$. However, in that study it was found that Bi$_{2-x}$Fe$_x$Se$_3$ with x = 0.04 is non-magnetic. Furthermore, that the magnetic transition temperatures in Bi$_{2-x}$Fe$_x$Te$_3$ depends strongly on x and that the easy axis is along c. Finally, it is important to point out here that recently Lasiak et al$^{25}$ predicted that in a magnetically doped TI the magnetic moments order ferromagnetically at zero temperature, but undergo a first order transition to a spin density wave (SDW) at higher temperature before they reach the paramagnetic phase. Our results may be consistent with this model, though it is unlikely that the high temperature phase, which can be detected clearly in dc magnetization measurements, is due to a SDW ordering. Such ordering is typically associated with small bulk magnetization.

V. SUMMARY AND CONCLUSIONS

In conclusion, we find that even at Fe doping levels as low as 5%, the full volume of the Bi$_2$Se$_3$ becomes magnetic at a relatively high temperature of ~ 250K. We identify two magnetic regimes; the first between ~ 10 – 250K and the second below ~ 10K. Bulk magnetization measurements show that the average size of Fe magnetic moment depends on temperatures but is almost independent of doping, and that the at the Fe moments align perpendicular to the crystallographic c-axis. Our $\mu$SR results indicate that the low temperature regime is independent of doping level and is more likely determined by Fe single ion properties and crystal field effects. In contrast, the nature of the magnetism in the high temperature regime differs, such that Fe-Fe interactions become more important, and therefore the local magnetization depends more on doping. The observed magnetism is homogeneous in the full volume of the sample and cannot be attributed to magnetic impurities. Finally, we conclude that the appearance of local static magnetic field at high temperatures and low doping is strong evidence that the Fe-Fe interactions are dominated by conduction electrons, e.g. RKKY type interaction. Therefore, near the surface, we expect that the magnetization will be affected differently by the topological surface states in the two temperature regimes. At low temperatures, the broken crystal symmetry will affect crystal field, while the change in the nature of conduction carriers due to topological surface states will affect the high temperature magnetism. Low energy $\mu$SR measurements are underway to explore the magnetism and its dependence on surface proximity. The results presented here will provide an excellent point of reference to detect any changes in the nature of the magnetism near the surface of Fe doped Bi$_2$Se$_3$.

* Electronic address: zaher.salam@psi.ch

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