Domain wall pinning and hard magnetic phase in Co-doped bulk single crystalline Fe$_3$GeTe$_2$

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We report the effects of cobalt doping on the magnetic properties of two-dimensional van der Waals ferromagnet Fe$_3$GeTe$_2$. Single crystals of (Fe$_{1-x}$Co$_x$)$_3$GeTe$_2$ with $x=0.0-0.78$ were successfully synthesized and characterized with x-ray diffraction, energy dispersive x-ray spectroscopy and magnetization measurements. Both the Curie-Weiss temperature and ferromagnetic (FM) ordered moment of Fe$_3$GeTe$_2$ are gradually suppressed upon Co doping. A kink in zero-field-cooling low field M(T) curve which is previously explained as an antiferromagnetic transition is observed for samples with $x=0.0-0.58$. Our detailed magnetization measurements and theoretical calculations strongly suggest that this kink is originated from the pinning of magnetic domain walls. The domain pinning effects are suddenly enhanced when the doping concentration of cobalt is around 50%, both the coercive field $H$ and the magnetic remanence to saturated magnetization ratio $M_r/M_s$ are largely improved and a hard magnetic phase emerges in bulk single crystal samples. The strong doping dependent magnetic properties suggest more spintronic applications of Fe$_3$GeTe$_2$.

INTRODUCTION

Two-dimensional (2D) van der Waals (vdW) ferromagnetic materials have recently drawn great attentions for their potential 2D magnetic, magnetoelectric and magneto-optic applications. For example, the layer-dependent intrinsic 2D ferromagnetism has been demonstrated in two insulating vdw materials Cr$_3$Ge$_2$Te$_6$ and CrI$_3$. The following application of CrI$_3$ in making spintronic devices has revealed surprisingly giant tunnelling magnetoresistance and the possibility to push magnetic information storage to the atomically thin limit. Comparing with insulators, vDW magnetic metals are preferred for building spintronic heterostructures as their metallic nature enabling the interplay of both spin and charge degrees of freedom. Fe$_3$GeTe$_2$ (FGT) serves as a rare metallic example of itinerant ferromagnetic vDW materials. It has a hexagonal crystal structure with the layered Fe$_3$Ge substructure sandwiched by two layers of Te atoms and a van der Waals gap in between. Early research finds ferromagnetic order with Fe moments aligned along the c axis below Curie temperature in bulk FGT ($T_c \approx 160$ K-230 K). Recent reports show that itinerant ferromagnetism persists in FGT down to the monolayer with an out-of-plane magnetocrystalline anisotropy and tunable FM characteristics, making FGT a promising candidate for spintronic applications. According to current reports, the bulk FGT single crystal has a ferromagnetic state with very small magnetic remanence to saturated magnetization ($M_R/M_s$) ratio and coercivity at all temperatures which limit its application in spintronic architectures. The only way to obtain a hard magnetic phase is making either nanoflakes or few layer samples. On the other hand, Yi et al. suggest an antiferromagnetic (AFM) transition below 150 K for FGT based on the low field magnetization data and theoretical calculations. Therefore it is still controversial if there is an AFM ground state at low temperature.

Chemical substitution is an effective way to tune the properties and probe the underlying physics of magnetic materials. We noticed that both Fe$_3$GeTe$_2$ and Ni$_3$GeTe$_2$ form the same crystal structure while the intermediate element Cobalt failed to form a Co$_3$GeTe$_2$ phase according to current reports. This is unusual because normally the properties of cobalt such as Pauling’s electronegativity and ionic radius lie in the middle between iron and nickel. It would be interesting to see how the magnetic properties of Fe$_3$GeTe$_2$ can be tuned by Co doping, which may also provides insights about the controversial ground state of FGT.

In this paper, we report the magnetic properties of (Fe$_{1-x}$Co$_x$)$_3$GeTe$_2$ single crystals with $x=0.0-0.78$. Our results suggest the previously reported suspicious AFM-like transition in FGT is actually caused by the movement of magnetic domain walls in a pinning state. The domain pinning effect can be largely enhanced by Co-doping, which induces an intrinsically hard magnetic phase ($M_R/M_s$~0.9) in contrast with the soft magnetic phase in undoped FGT.

EXPERIMENTAL DETAILS

The single-crystalline samples of (Fe$_{1-x}$Co$_x$)$_3$GeTe$_2$ were prepared by the standard chemical vapor transport (CVT) method with iodine as the transport agent similar to previous reports. Crystals with typical dimensions of 1 mm×1 mm×0.1 mm are obtained with cobalt doping values up to $x=0.78$. Further efforts in growing crystals with larger $x$ failed and simply brought out products of CoTe$_{1.8}$ crystals. We characterized all samples with energy dispersive x-ray spectroscopy (EDS, Oxford X-Max 50). The descriptions in this paper about doping level $x$ all refer to the EDS values. The single crystal x-ray diffraction patterns were collected from a Bruker D8 Advance x-ray diffractometer using Cu $K_{\alpha}$ radiation. The magnetization measurements of our
FIG. 1: (a) The x-ray diffraction patterns measured on single crystals of (Fe$_{1-x}$Co)$_2$GeTe$_2$ showing (00L) diffraction peaks. (b) Doping dependence of c-axis lattice parameters. Inset shows one single crystal of x=0.58 imaged by a scanning electron microscope. (c) Temperature dependence of the magnetization M measured with H=0.5 T applied either parallel to the c-axis (H||c, solid symbols) or parallel to the ab-plane (H||ab, open symbols) for (Fe$_{1-x}$Co)$_2$GeTe$_2$. (d) Isothermal magnetization curves for different samples measured with H||c and H||ab up to 5 T at T=1.8 K. The curve with x=0.78 is fitted by equation (1). (e) Doping dependence of saturated magnetic moment per Fe calculated from isothermal magnetization curves.

samples were performed using a Quantum Design MPMS3.

We performed first-principles density functional theory calculations using the same methods described in our previous publications[23-25]. In brief, we used a van der Waals density functional (vdW-DF) method[26,27], with the optB86b functional[28] for the exchange part (optB86b-vdW) to optimize atomic structures of bulk FGT, which usually reveals good agreements of calculated structure-related properties with experimental values of two-dimensional materials[29-33]. Given optimized structures, we used the standard Perdew-Burke-Ernzerhof (PBE) functional[34] with the consideration of spin-orbit coupling (SOC) to account energy differences of all considered magnetic configurations, this scheme was found to share the qualitatively same results with the Heyd-Scuseria-Ernzerhof (HSE06) functional[35,36] in other magnetic 2D layers, e.g. CrI$_3$[24] and CrSCl$_2$[25].

RESULTS AND DISCUSSIONS

Figure 1(a) presents the x-ray diffraction data of three single crystals with x=0, x=0.58 and x=0.78 respectively. The peaks can be indexed by (00L) with even values. No impurity peaks are found within the instrument resolution. The c-axis lattice parameters derived from the x-ray data decrease monotonically with increasing x as shown in Fig. 1(b). These results indicate the successful introduction of cobalt into the FGT lattice.

Figure 1(c) shows the temperature dependent magnetization measurements in zero-field-cooling (ZFC) with magnetic field of 0.5 T applied either parallel or perpendicular to the ab-plane. The FM transition temperature $T_c$ of Fe$_3$GeTe$_2$ sample is around 200 K, then it is gradually suppressed with Co-doping. On the other hand the magnetic easy axis is along H||c for all samples while the magnetic anisotropy and the ordered moment of Fe gradually decrease with increasing x.

The isothermal magnetization curves at T=1.8 K are presented in Fig. 1(d). For crystals with x=0-0.58, the rapid saturated magnetizations confirm their ferromagnetic ground states. For x=0.78, the shape of M(H) curve resembles those observed in cluster glasses[37,38]. Therefore we fit the M(H) curve with a modified Langevin function represented by

$$M(H) = M_S L(\mu H/k_BT) + \chi H$$

Here $\mu$ is the average moment per cluster, $L(x)=coth(x)-1/x$ is the Langevin function, $M_S$ is the saturated moment, and $\chi$ is the paramagnetic susceptibility[37,38]. The fitting result gives an $M_S$ value of 0.153 $\mu_B$ for x=0.78. For other samples, the $M_S$ values were determined from the intercept of a linear fit of H>1 T data with H=0. The doping dependence of saturated magnetic moment per Fe/Co is shown in Fig. 1(e). The suppression of saturated moment is quite similar as that in nickel-substituted Fe$_3$GeTe$_2$[38]. One difference is that the cluster glass behavior starts at x=0.37 for Ni doping while the ferromagnetic state still seems to be robust at least for x=0.58 in the case of Co doping.

When the temperature dependent magnetizations are measured at a lower magnetic field such as 100 Oe, anoma-
which is possibly due to the formation of cluster spin glass. In Fig. 2(f) the FM transition temperature $T_\text{c}$ (minimum in the $\text{d}M/\text{d}T$ curve) and the anomalous ZFC kink temperature $T^*$ at $H=100 \text{ Oe}$ are plotted as a function of doping $x$.

In order to clarify the origin of $T^*$, the $x=0.58$ sample is chosen for detailed magnetization measurements. Three major features are found: (1) The kink gradually moves to low temperature with increasing magnetic field and finally disappears at $H=3 \text{ kOe}$ (Fig. 3(a)). (2) No kink is observed in FC curves under the same field (inset of Fig. 3(a)). (3) The $M(\text{H})$ curve at $T=1.8 \text{ K}$ with $H||c$ undergoes a steep magnetization jump at $H\approx2 \text{ kOe}$ as shown in the inset of Fig. 3(b). This jump gradually moves to lower field and finally disappears at $T=30 \text{ K}$. The kinks in $M(\text{T})$ curve and the jumps in $M(\text{H})$ curve (marked by black arrows in (a) and (b)) can be scaled together in a temperature vs field plot. (d) Hysteresis loops for $x=0.58$ with different maximum magnetic fields applied parallel to $c$-axis at $T=2 \text{ K}$.

Based on the above observations, there are two possible explanations for the kinks and jumps mentioned above, namely a spin-flop transition (from AFM to FM) or a pinning-depinning crossover of magnetic domain walls. We argue that a spin-flop transition is unlikely for two reasons. First of all, according to our theoretical calculations described in the previous section, the interlayer FM configuration is $0.81 \text{ meV/Fe}$ more stable than the interlayer AFM configuration, suggesting a FM groundstate, which indicates flipping of magnetic moment from an anti-parallel to a parallel configuration is, most likely, not a reason for the observed magnetic transition. Even if the interlayer magnetism appears to be AFM, owing some reason, e.g. a particular stacking. We find that this crossover or transition remains in Co-doped samples up to $x=0.58$ with occurring temperature $T^*$ approaching the FM transition temperature. For $x=0.78$ all $M(\text{T})$ curves show peaks at $T=9 \text{ K}$, which is possibly due to the formation of cluster spin glass. In Fig. 2(f) the FM transition temperature $T_\text{c}$ (minimum in the $\text{d}M/\text{d}T$ curve) and the anomalous ZFC kink temperature $T^*$ at $H=100 \text{ Oe}$ are plotted as a function of doping $x$.
difference implies that it may take roughly 10 Tesla to flip the interlayer magnetic moment, roughly two orders of magnitude larger than the 2 kOe field we observed in our experiment. Magnetic field at this strength would more likely to cause a movement or depinning of magnetic domains, rather than flop the spins. Secondly, the magnetization loop with maximum field of 1 kOe exhibits a linear feature with very weak hysteresis, while significant FM hysteresis appears in the loop with maximum field of 2.1 kOe (Fig. 3(d)). This means that if a spin-flop transition from AFM to FM really exists, it can not be tuned back when the field is cooled from 2.1 kOe. This behavior clearly contradicts the common features of spin-flop transitions.

So we propose a crossover from pinning to depinning of magnetic domain walls as the reason for the magnetization kinks shown in Fig. 2 and Fig. 3. When the sample is cooled under zero field, the magnetic domains start to be pinned below the crossover temperature $T^*$ with their total moment close to zero (keeping the lowest magnetostatic energy). Then applying a low field of 100 Oe at lowest temperature is not enough to move the pinned domains. With increasing temperature, thermal fluctuations gradually weaken the pinning force and finally completely depin the domains above $T^*$ with domain moment well aligned along the field direction. This explains why the kink of magnetization with decreasing temperature never occurs in FC curve. Because in FC process the domains are always pinned with the effective FM moment aligned along the cooling field. The thermo-hysteresis observed in the FC and FW curves is likely due to the domain structure dynamics when switching between pinning and depinning state. A recent scanning tunneling microscope (STM) study on FGT uses ferromagnetic Ni tips to mimic the FC and FW process[40]. The data show that the domain structure in FC process is different from that in FW process even at the same temperature, which naturally explains the thermal-hysteresis[41]. It should be mentioned that a spin-flop transition could also possibly generate the thermo-hysteresis[22], however previous neutron scattering studies on FGT do not support an AFM spin-configuration at low temperature[17].

The hysteresis loops are measured for all samples and reveal new doping induced magnetic properties. As shown in Fig. 4(a) and inset, it is evident that all $(\text{Fe}_{1-x}\text{Co}_x)_2\text{GeTe}_2$ samples with $x \leq 0.58$ are pinning type ferromagnets. Namely the initial magnetization of the sample is negligible but suddenly become significant beyond a certain field, this change in magnetization is reached by the movement of the pinned domain walls[20]. For $x=0.25$, both the coercive field $H_c$ ($\sim 200$ Oe) and the magnetic remanence to saturated magnetization ratio $M_R/M_S (<0.1)$ are very low, which belong to soft magnetic properties same as previous reports about $\text{Fe}_3\text{GeTe}_2$[17, 20]. However for samples with $0.46 \leq x \leq 0.58$, the hysteresis loops suddenly display a near square shape with greatly enhanced coercivities (Fig. 4(b), coercive field $\sim 1.5$ kOe). Meanwhile the calculated $M_R/M_S$ ratios are all larger than 0.8 from $x=0.46$ to $x=0.58$ with maximum value of 0.9 (Fig. 4(c)). These are all well-defined hard magnetic properties similar as that in the previously reported few layer samples or thin films of FGT[18, 19, 21]. These results suggest hard magnetic phases can also be induced by Co doping in bulk single crystals. For samples with $x \geq 0.68$, both the hard magnetic properties and pinning type magnet features gradually disappear. To summarize the results in Fig. 4, we have discovered that the coercive fields and $M_R/M_S$ values in $(\text{Fe}_{1-x}\text{Co}_x)_2\text{GeTe}_2$ are strongly doping dependent, hard magnetic phases can be realized at $0.46 \leq x \leq 0.58$.

![Hysteresis loops](image)

**FIG. 4:** (a) Hysteresis loops for samples with different doping concentrations at $T=2$ K and $H||c$. The inset shows the enlarged view of the dashed box area. The data were measured with $H_{\max} = \pm 50$ kOe. Doping dependent of coercive fields (b) and $M_R/M_S$ values (c) at $T=2$ K for $(\text{Fe}_{1-x}\text{Co}_x)_2\text{GeTe}_2$. The data are calculated from hysteresis loops.

A major source of hysteresis in ferromagnets is the pinning of magnetic domain walls[41]. Generally speaking in order to get a high coercive field $H_c$ in a pinning type magnet, it requires the formation of a large domain wall energy (DWE) and an effective network of pinning centers capable of locally increasing DWE to inhibit the domain wall movement[20]. The doping of Co should somehow greatly improve the DWE of $\text{Fe}_3\text{GeTe}_2$ thus induces hard magnetic properties. It should be mentioned that this improvement of DWE seems to only occur when Fe:Co=1:1. Samples with $x \leq 0.25$ and $x \geq 0.68$
all exhibit soft magnetic properties. We have repeated the above findings on more samples with slightly different synthesis procedures and nominal doping, the results show that the emergence of hard magnetic phase only depends on the doping concentration. Hard magnetic properties are crucial for the applications of 2D magnetic materials in spintronics. We have shown the possibility of getting a tunable hard magnetic phase through chemical doping in Fe$_3$GeTe$_2$ bulk single crystals instead of making few layer samples or thin films. These findings should shed new light on the research and application of itinerant 2D vdW ferromagnetic metal Fe$_3$GeTe$_2$.

**CONCLUSIONS**

In summary, a series of (Fe$_{1-x}$Co$_x$)$_3$GeTe$_2$ (x=0-0.78) single crystals have been successfully grown by CVT methods. All samples with x=0-0.58 are pinning type magnets and the previously reported AFM-like transition in Fe$_3$GeTe$_2$ should originate from the movement of pinned magnetic domains. Instead of making few layer samples or thin films, these hard magnetic properties can be realized in bulk single crystals of Fe$_3$GeTe$_2$ with Co doping.

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