Enhancement of Spin Pumping from CoFeB to Sb$_2$Te$_3$ Layers by Crystal Orientation Control

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Sb$_2$Te$_3$ is well known as a phase-change material, and is expected to exhibit high spin–charge current conversion because of its strong spin–orbit interactions. Reversible phase-change characteristics enable manipulation of physical properties depending on phase. Therefore, it is anticipated that its spin properties can also be tuned by controlling the phases of Sb$_2$Te$_3$, promising great potential for novel spintronic devices. However, the effects of crystallinity on the spin properties of Sb$_2$Te$_3$ have not yet been investigated. Herein, the spin relaxation induced by the spin-pumping effect in CoFeB/Sb$_2$Te$_3$ bilayers is examined using ferromagnetic resonance (FMR) at 300 K for crystalline and amorphous Sb$_2$Te$_3$. FMR signal width enhancement is observed for the highly oriented crystalline Sb$_2$Te$_3$ film at thickness less than 7 nm. In contrast, the FMR signal widths of the amorphous sample are independent of the layer thickness. The significant increase in linewidth suggests more efficient spin injection from the ferromagnetic CoFeB layer into crystalline Sb$_2$Te$_3$ than into amorphous Sb$_2$Te$_3$. This stark contrast between the spin dynamics of the amorphous and crystalline Sb$_2$Te$_3$ films supports the idea that spin conduction can be tuned by controlling the crystal orientation of Sb$_2$Te$_3$ films.

1. Introduction

Layered chalcogenides such as Sb$_2$Te$_3$ and Bi$_2$Te$_3$ have served as important components of phase-change materials (PCMs) for nonvolatile memory applications that utilize the difference in resistivity between amorphous and crystalline phases. At the same time, these materials have strong spin–orbit coupling (SOC), which is supposed to convert charge current to spin current with high efficiency; thus, such materials are expected to be suitable for spintronic devices. In spintronic devices, the generation, control, and detection of spin currents (angular momentum flows) are crucial because they are necessary to effectively realize the advantages of spin-based magnetic memory and logic devices. So far, materials that can generate spin currents that have been investigated include heavy metals with intrinsic strong SOC, Cu and Au alloys with diluted heavy metals, and 2D electron gases at semiconductor surfaces. Recently, highly efficient spin current generation has also been reported in 2D systems with Rashba surfaces and 3D topological insulators (TIs). The techniques for generation, manipulation, and detection of spin currents in these materials include spin pumping, spin torque ferromagnetic resonance (ST-FMR), and nonlocal spin valve (NLSV). Here, spin pumping is commonly investigated using a simple heterostructure consisting of ferromagnetic (FM) and nonmagnetic (NM) layers. In this method, FMR excites the precession of magnetization in the FM layer, causing spin accumulation at the FM/NM interface, and the diffusion of these spins induces a spin current. The spin current flowing into the NM layer is scattered owing to the presence of strong SOC and is detected as a voltage drop in the NM, which is known as the inverse spin Hall effect (ISHE). In contrast, the FMR signal is broadened as the damping parameter changes depending on the amount of the spin current injected into the NM layer from FM layer. In this method, the FM layer plays a role as both source and detector of the spin current. Using this measurement to detect the spin-pumping effect has been reported for heavy metals such as Pt and W, oxide insulators, and semiconductors. Recently, it has been experimentally reported that damping enhancement can be realized in FM/TI bilayers. Therefore, the observation of spin-pumping effects using FMR is an effective technique for studying spin generation and detection materials.

In this study, we investigated the spin dynamics of FM metal CoFeB and Sb$_2$Te$_3$ using systematic FMR measurements. Here, note that Sb$_2$Te$_3$ is a PCM that exhibits a reversible transition between a layered crystalline phase and a disordered amorphous phase, generating physical contrasts such as optical reflectivity and electrical resistivity. This property means that PCMs have...
the capability to control charge currents. However, to date, the tunability of the spin current has not been investigated. In this work, we controlled the Sb$_2$Te$_3$ phase by tuning the fabrication process and obtained both crystalline and amorphous films. The results of our experiments were then compared with results for typical heavy-metal-based CoFeB/Pt bilayers. Enhancement of the FMR signal width was observed only for the highly oriented crystalline Sb$_2$Te$_3$ sample when the film thickness was less than 7 nm, whereas the FMR signal widths of the amorphous films were almost independent of film thickness. The FMR linewidth of crystalline Sb$_2$Te$_3$ was found to be more than twice that of the typical heavy metal Pt with the same 5 nm film thickness. Furthermore, it is notable that the maximum linewidth for crystalline Sb$_2$Te$_3$ occurs at 4 nm in thickness. The results are discussed based on atomic ordering as well as the topological properties of Sb$_2$Te$_3$ films. These findings will facilitate the realization of spintronic devices based on PCs and novel devices that utilize reversible switching of unique properties with the presence of crystalline structures.

As the FM, a 10 nm-thick ($d_{CoFeB}$) CoFeB film was deposited using the Co$_{40}$Fe$_{60}$B$_{20}$ alloy target at room temperature after the growth of Sb$_2$Te$_3$ in the same chamber without breaking the vacuum. The CoFeB/Sb$_2$Te$_3$ interface was examined by X-ray photoelectron spectroscopy (not shown) and no interfacial reaction was observed, suggesting that the FMR signal purely reflects the spin pumping from FM to TI layer. Note that because the Pt film was grown in a different deposition chamber, Ar sputter-cleaning of the Pt surface was performed for 1 min at 50 W before CoFeB deposition to eliminate surface contamination. Each sample was covered with a 3 nm-thick MgO-cap layer to prevent oxidation. Note that the MgO-cap is fabricated on all samples to eliminate the effects of interfacial magnetic anisotropy caused by nonmagnetic material.$^{[26]}$ The thicknesses of the spin-pump materials were varied as 3, 4, 5, 7, and 10 nm, nearly in the same order as their spin diffusion lengths at which spin injection is most efficiently achieved. X-ray diffraction (XRD) measurements and transmission electron microscopy (TEM) were conducted to investigate the crystallinity as well as the microstructures of the Sb$_2$Te$_3$ layer.

To study the relaxation of magnetization by spin pumping, FMR measurements using a TE011 microwave cavity were performed at a microwave frequency $\nu_{mic}$ of 9.6 GHz. The samples were inserted into the cavity on a glass rod, and the derivative absorption FMR spectra with respect to the DC magnetic field $H_{ext}$ were measured by the lock-in technique using a small AC magnetic field 10 G modulated at 100 kHz, which was superimposed on the DC magnetic field. Note that the modulation field dependence of the FMR signal was also measured from 1 to 30 G to confirm the shapes of the FMR signals, as they could be distorted by too large modulation field. It was found that the FMR signals did not exhibit significant changes regardless of the magnetic field magnitude, as shown in Figure S1, Supporting Information. Thus, a magnetic field magnitude of 10 G was used in this work. All FMR experiments were performed at room temperature.

2. Results and Discussion

Sb$_2$Te$_3$ has a layered crystal structure with van der Waals gaps, as shown in Figure 1a. Figure 1b shows the XRD patterns of the 5 and 50 nm-thick crystalline Sb$_2$Te$_3$ films as well as the simulated XRD pattern under the assumption of (001) preferred orientation. The 50 nm-thick Sb$_2$Te$_3$ film exhibits an ideal (00l)-textured XRD pattern without showing any nonoriented peaks. It should be noted that even though the peak intensities become much weaker for the 5 nm-thick film, the peak positions are essentially identical to the thicker sample as well as to the simulated pattern, indicating that the quality of the Sb$_2$Te$_3$ films is preserved even for ultrathin films with thicknesses less than 10 nm. Figure 1c compares the full widths at half-maximum (FWHMs) of the (0015) peaks for the 5 and 50 nm-thick films. The 5 nm-thick Sb$_2$Te$_3$ film shows a FWHM nearly 10 times larger than that of the 50 nm-thick film.

Figure 1. a) Crystal structure of Sb$_2$Te$_3$. b) XRD patterns of 5 nm-thick (red) and 50 nm-thick (green) crystalline Sb$_2$Te$_3$ films. The simulated XRD pattern of the (001) preferred orientation is also shown. c) Magnified image of (0015) peaks. The FWHMs of two films with different thicknesses are compared.
This result is reasonable because the FWHM is known to be inversely proportional to the size of the crystal grain according to the Scherrer equation. Therefore, these results suggest that the quality of the fabricated crystalline Sb$_2$Te$_3$ film is guaranteed in the thinner limit. It should be noted that apart from sputtering, metal–organic chemical vapor deposition (MOCVD) is another potential technique for industrial fabrication and there have been some studies reporting the growth of Sb$_2$Te$_3$ film by MOCVD, where depending on the growth condition either randomly oriented polycrystalline or highly oriented crystalline films were reported.$^{[27,28]}$ Sputter-grown Sb$_2$Te$_3$ film in this study is a highly oriented structure like the study by Rimoldi et al.,$^{[29]}$ in which surface is terminated by Te atoms.

**Figure 2a** shows the XRD patterns of the 50 nm-thick crystalline and amorphous Sb$_2$Te$_3$ films. Small peaks are observed in the amorphous film, indicating that even though the growth was performed at room temperature, the Sb$_2$Te$_3$ film was not fully amorphized (disordered) and contained nanometer-scale fine crystal grains. However, as the intensities are much weaker than those of the highly oriented crystalline film, and moreover, as the observed peaks do not originate from (00l) planes, but rather from nonoriented lattice planes such as (015) typically observed in the ideal powder sample, the two films are inherently different in terms of atomic ordering. In this article, even though the Sb$_2$Te$_3$ film grown at room temperature is highly disordered nanocrystalline structure, the word “amorphous” is nominally used to avoid reduced readability. For the same reason, the highly oriented film is referred as “crystalline” film. Figure 2b–e shows cross-sectional TEM images of the crystalline and amorphous samples, respectively, where the CoFeB ferromagnet (10 nm) and MgO cap (3 nm) layers are also observable. As shown in Figure 2b,d, the crystalline Sb$_2$Te$_3$ film is highly oriented and is characterized by lattice fringes parallel to the substrate surface. In contrast, the amorphous Sb$_2$Te$_3$ film shows a disordered microstructure, where some crystal grains with 2–3 nm size can be seen (Figure 2c,e), which is consistent with the XRD result (Figure 2a).

**Figure 3a** shows a schematic of spin pumping in the CoFeB/Sb$_2$Te$_3$ heterostructure film. The angular momentum, originating from the resonant precession of the magnetization in CoFeB, is pumped into Sb$_2$Te$_3$, leading to spin accumulation at the interface. The accumulated spin diffuses into Sb$_2$Te$_3$ and dissipates in the order of the spin diffusion length, giving rise to the spin current $j_s$. The spin relaxation corresponds to the decay of the generated spin current, and the larger the SOC, the faster is the spin relaxation. The spin relaxation in Sb$_2$Te$_3$ causes a loss of angular momentum in CoFeB, resulting in an increase in the damping. Therefore, the damping constant derived from the FMR measurement represents the magnitude of the spin current flowing into Sb$_2$Te$_3$. Figure 3b–d compares the FMR spectra of crystalline Sb$_2$Te$_3$, amorphous Sb$_2$Te$_3$, and Pt heterostructure samples, respectively, with that of the CoFeB (10 nm) single-layer sample. All samples exhibit linewidth broadening compared with the resonance signal of the CoFeB control sample. The resonant linewidth $\Delta H$ was obtained by fitting the FMR spectra with the first derivative of the Lorentzian function, as shown in Figure 3b–d, and was estimated to be $166.0 \pm 4.6$, $72.6 \pm 3.6$, and $77.7 \pm 0.1$ G for the crystalline Sb$_2$Te$_3$, amorphous Sb$_2$Te$_3$, and Pt samples, respectively. Note that the magnetic inhomogeneity is practically absent because all spectra could be well fitted by the single Lorentzian function. $\Delta H$ of the crystalline Sb$_2$Te$_3$ sample is much larger than those of the amorphous counterpart and conventional heavy metal, indicating that the crystal orientation of the Sb$_2$Te$_3$ film plays an important role in the spin injection efficiency.

In parallel, FMR measurements were also performed to investigate the effects of magnetic anisotropy by rotating the external magnetic field along a polar angle between the in-plane and out-of-plane directions. As shown in Figure 5S, Supporting Information, the resonance field shifted to a higher field magnitude for both amorphous and crystalline Sb$_2$Te$_3$ films with decreasing external magnetic field angle. This is the same trend as for the CoFeB single film, which arises owing to the easy plane of magnetization. It should be noted that the linewidth of the crystalline Sb$_2$Te$_3$ sample was always broader compared with its amorphous counterpart at all measured angles, indicating that the observed peak broadening in the crystalline sample is due to long-range atomic order of highly oriented films and that the magnetic anisotropy effect does not play an essential role for spin injection efficiency.
Figure 3. a) Schematic illustration of spin pumping at CoFeB/Sb$_2$Te$_3$ interface. b–d) FMR spectra of crystalline Sb$_2$Te$_3$ (b), amorphous Sb$_2$Te$_3$ (c), and Pt (d) samples. The FMR spectrum of the reference sample (CoFeB (10 nm)/MgO (3 nm)) is also shown for comparison. Dotted curves: fitting curves of Lorentzian derivative model.

Figure 4. a,b) Film thickness dependence of FMR spectra for crystalline (a) and amorphous (b) Sb$_2$Te$_3$ heterostructure samples. The FMR spectrum of the CoFeB single layer is shown with dotted lines in each figure as a reference.

constant $\alpha$, represented as $\Delta H = 2\alpha\omega/\gamma$, where $\omega$ and $\gamma$ are the resonant frequency and gyromagnetic ratio, respectively. Here, $\Delta \alpha$ is defined as $\Delta \alpha = \alpha_{\text{CoFeB}}/\alpha_{\text{Sb$_2$Te$_3$}} - 1$, where $\alpha_{\text{CoFeB}}$ and $\alpha_{\text{Sb$_2$Te$_3$}}$ are the damping constants of the CoFeB/Sb$_2$Te$_3$ bilayer and CoFeB single layer, respectively. The linewidth and damping constant differences for the 4 nm-thick crystalline film had maximum values of $225.0 \pm 0.3$ G and $(2.64 \pm 0.01) \times 10^{-2}$, respectively. It is known that, in transition metals, where the thickness of the spin-pump layer is less than its spin diffusion length, backflow of spin occurs from the opposite interface[29] and the increase in the damping constant is suppressed (Figure S3, Supporting Information). In contrast, the damping constant $\Delta \alpha$ increased for crystalline Sb$_2$Te$_3$ when the thickness was less than 7 nm, suggesting that the thickness dependence tendency of the damping constant is different from that in conventional transition metals. Note that the thickness dependence of sheet resistance $R_S$ for the amorphous and crystalline Sb$_2$Te$_3$ films shows a similar trend while preserving a constant resistance contrast of about two orders of magnitude, as shown in the inset of Figure 5a. This means that the qualities of the Sb$_2$Te$_3$ film, e.g., its phase-change function, are well maintained even in thinner films.

Here, the origin of the significant enhancement of $\Delta H$ and $\Delta \alpha$ observed in crystalline Sb$_2$Te$_3$ samples less than 7 nm-thick is discussed. Fanchiang et al. reported the abrupt increase in $\Delta H$ for the Bi$_2$Se$_3$ TI film grown on the ferromagnetic insulator yttrium iron garnet (YIG) for film thicknesses less than 7 nm.[22] They attributed the origin of the enhanced magnetic anisotropy to hybridization between topological surface states (TSS) and the magnetic layer YIG. As Sb$_2$Te$_3$ is a typical topological insulator, as is Bi$_2$Se$_3$, a similar enhancement is expected. Moreover, they also found an abrupt drop of $\Delta \alpha$ in the thickness range between 3 and 7 nm. It was explained that the Dirac cone opens for the thinner films because of the hybridization of wave functions between the top and bottom surfaces, resulting in the disappearance of the TSS.[10,31] This threshold thickness was in good agreement with the experimental observations obtained by angle-resolved photoelectron spectroscopy (ARPES) as well as
Figure 5. a) FMR linewidth $\Delta H$ and difference of the damping parameter $\Delta \alpha$ ($= \mu_{\text{CoFeB}}/\mu_{\text{SbTe}} - \mu_{\text{CoFeB}}$). The inset shows the dependence of sheet resistance on thickness for Sb$_2$Te$_3$. b) Effective magnetization $4\pi M_{\text{eff}}$ as a function of Sb$_2$Te$_3$ thickness. The inset represents the direction of the external magnetic field applied to the samples, which is the in-plane direction. The dashed lines in both figures represent values for the CoFeB single film.

with theoretical calculations.$^{[13]}$ Figure 5a shows a similar trend in that $\Delta H$ and $\Delta \alpha$ sharply increased as the film became thinner for the crystalline sample, i.e., surface effects became dominant. Furthermore, these values decreased again for the 3 nm-thick film, suggesting the disappearance of TSS. It should be noted that this behavior was not observed for the amorphous film; moreover, the thickness dependence was almost negligible, similar to that for heavy metals. The results for the Pt reference sample are shown in Figure S3, Supporting Information. As the long-range order is absent in the amorphous Sb$_2$Te$_3$ film, TSS was not observed, even for the thinner samples. Moreover, surface sensitive topological states have been recently reported theoretically, where the Dirac cone appears at the Te-terminated (001) surface, while the band structure becomes either metallic or insulating at side surfaces.$^{[13]}$ Therefore, even though some small crystal grains were observed in the amorphous film, the orientation of crystals is random and the atomic order of Sb$_2$Te$_3$ at the interface is not uniform resulting in absence of the linewidth broadening in the amorphous film. In addition, $\Delta H$ of the crystalline Sb$_2$Te$_3$ film is comparable with the amorphous film at thickness greater than 7 nm film, suggesting that the bulk effect becomes more dominant than the surface effect for the thicker films. Although other measurements should be performed to confirm the topological properties of the crystalline Sb$_2$Te$_3$ film used in this study, the observed clear contrast of $\Delta H$ and $\Delta \alpha$ between two phases would be beneficial for realizing a novel device utilizing the Sb$_2$Te$_3$/CoFeB heterostructure.

Figure 5b shows the relationship between the effective magnetization $4\pi M_{\text{eff}}$ and the Sb$_2$Te$_3$ film thickness. $4\pi M_{\text{eff}}$ exhibits a relationship with the resonance frequency and the resonance field, $\omega = |g| \sqrt{H_{\text{res}}/H_{\text{res}}+4\pi M_{\text{eff}}}$. It was found that the effective magnetization tends to decrease with decreasing Sb$_2$Te$_3$ film thickness for both crystalline and amorphous samples. The $4\pi M_{\text{eff}}$ of the amorphous film is smaller than that of the CoFeB single-layer sample (13.9 kG) over the entire thickness range, while the crystalline sample showed a crossover of $4\pi M_{\text{eff}}$ with the CoFeB reference at around 5 nm of thickness. Here, depending on the magnitude of $4\pi M_{\text{eff}}$ relative to the effective magnetization of the CoFeB single layer, $4\pi M_{\text{CoFeB}}$, the magnetic anisotropy is changed, namely, perpendicular magnetic anisotropy (PMA) is present when $4\pi M_{\text{CoFeB}} > 4\pi M_{\text{eff}}$, and an easy-plane magnetic anisotropy is enhanced for the opposite case $4\pi M_{\text{CoFeB}} < 4\pi M_{\text{eff}}$. As the 10 nm-thick CoFeB single layer is known to exhibit easy-plane magnetic anisotropy, it was anticipated that the decrease in $4\pi M_{\text{eff}}$ for the amorphous Sb$_2$Te$_3$ sample was due to the appearance of the PMA component through formation of the heterostructure. According to the TEM image in Figure 2c, the surface of CoFeB grown onto the amorphous Sb$_2$Te$_3$ film was rougher compared with the crystalline sample, indicating substantially lower $4\pi M_{\text{eff}}$ for the amorphous samples. Moreover, the increasing stress and the roughness become more and more significant for the thinner films that could cause further reduction of $4\pi M_{\text{eff}}$ seen in the thinner limit (Figure 5b). In crystalline Sb$_2$Te$_3$, in-plane magnetic anisotropy (IMA) exists when the Sb$_2$Te$_3$ film thickness is greater than 5 nm, resulting in the increment of $4\pi M_{\text{eff}}$ over the reference value of the CoFeB single layer. Assuming the existence of TSS in the crystalline Sb$_2$Te$_3$ sample, the surface spins of the Sb$_2$Te$_3$ lie in the in-plane direction due to spin–momentum locking. At the Sb$_2$Te$_3$/CoFeB interface, the CoFeB spins align in the in-plane direction owing to the exchange interaction between the ferromagnetic CoFeB and the Sb$_2$Te$_3$ surface spins, which can additionally contribute to the IMA of CoFeB film. This can also explain the weakened effective magnetization of thinner Sb$_2$Te$_3$ films where the TSS no longer exists because of the hybridization of wave functions. In addition, similar to the case for amorphous Sb$_2$Te$_3$, the PMA component starts to appear due to the presence of stress and roughness for the thinner crystalline films, as shown in Figure 5b.

3. Conclusion

We have investigated the spin-pumping effect in CoFeB/Sb$_2$Te$_3$ bilayers using the FMR technique at room temperature for highly oriented crystalline and amorphous phases of Sb$_2$Te$_3$. A sharp increase in the linewidth of the resonance signal was
observed at a thicknesses less than 7 nm only in the crystalline Sb$_2$Te$_3$ film, whereas almost no change was observed for the amorphous Sb$_2$Te$_3$ film regardless of film thickness. Moreover, the spin-pumping efficiency was enhanced by more than twofold for the highly oriented crystalline Sb$_2$Te$_3$ film compared with the conventional Pt film. In particular, the enhancement of the linewidth is most remarkable at 4 nm thickness for the crystalline Sb$_2$Te$_3$ samples; this feature is not observed for amorphous Sb$_2$Te$_3$ samples. No such reports have been made previously for heavy metals typically used in spin Hall materials such as Pt, W, and Ta. In addition, for the effective magnetization of Sb$_2$Te$_3$ samples, a change in the magnetic anisotropy direction depending on Sb$_2$Te$_3$ thickness was observed, implying the possibility of interactions between TSS in crystalline Sb$_2$Te$_3$ and ferromagnetic CoFeB films. These results strongly suggest that it is possible to modulate the amount of injected spin by controlling the crystall structure, which provides a new perspective on spin conduction control using phase-change phenomena in conventional spintronics research. This may lead to the development of phase-change spintronics devices using charge and spin degrees of freedom. The next research step will require detection of voltage converted from spin current, which is motivated by the increased damping constant due to the crystalline structure of Sb$_2$Te$_3$. In fact, realization of spin-to-charge conversion has been reported in the crystalline Sb$_2$Te$_3$ at room temperature. Moreover, the topological nature of crystalline and amorphous Sb$_2$Te$_3$ films will be necessary to be measured and compared using transport measurements, i.e., weak antilocalization effect. From the viewpoint of fundamental physics, it would be interesting to simultaneously investigate the topological function and spin property phenomena of the material because Sb$_2$Te$_3$ is also typically used as a TI material.

4. Experimental Section

The bilayer samples used in this study consisted of spin-pump materials and FMs at whose interfaces spin-pumping phenomena could be observed. Crystalline and amorphous Sb$_2$Te$_3$ films were fabricated as spin-pump materials. A Pt film 5 nm thick was also prepared as a reference, as it is a typical heavy-metal-based spin-pump material with strong SOC. Thin film samples were deposited by radiofrequency magnetron sputtering (QAM-4, ULVAC KYUSHU Corp.) on a single-crystal Si substrate (525 nm) covered with thermally oxidized SiO$_2$ (300 nm). For the growth of a crystalline Sb$_2$Te$_3$ film, a 3 nm-thick (2 nm thick in the case of 3 nm films) amorphous Sb$_2$Te$_3$ film was deposited in advance at 300 K. Subsequently, the sample was heated to 503 K in the sputter chamber, causing crystallization, followed by growth of the rest of the film at high temperature. A detailed description of the Sb$_2$Te$_3$ growth process is available elsewhere. The amorphous Sb$_2$Te$_3$ and Pt films were grown at room temperature.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

Keywords

ferromagnetic resonance, phase-change materials, spin pumping, spintronics, topological insulators

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