Fe-Sn nanocrystalline films for flexible magnetic sensors with high thermal stability

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The interplay of magnetism and spin-orbit coupling on an Fe kagome lattice in Fe₃Sn₂ crystal produces a unique band structure leading to an order of magnitude larger anomalous Hall effect than in conventional ferromagnetic metals. In this work, we demonstrate that Fe-Sn nanocrystalline films also exhibit a large anomalous Hall effect, being applicable to magnetic sensors that satisfy both high sensitivity and thermal stability. In the films prepared by a co-sputtering technique at room temperature, the partial development of crystalline lattice order appears as nanocrystals of the Fe-Sn kagome layer. The tangent of Hall angle, the ratio of Hall resistivity to longitudinal resistivity, is maximized in the optimal alloy composition of close to Fe₃Sn₂, implying the possible contribution of the kagome origin even though the films are composed of nanocrystal and amorphous-like domains. These ferromagnetic Fe-Sn films possess great advantages as a Hall sensor over semiconductors in thermal stability owing to the weak temperature dependence of the anomalous Hall responses. Moreover, the room-temperature fabrication enables us to develop a mechanically flexible Hall sensor on an organic substrate. These demonstrations manifest the potential of ferromagnetic kagome metals as untapped reservoir for designing new functional devices.

Iron-based alloys and compounds have constituted the outstanding basis for applications, particularly with judicious utilization of their rich magnetism and magneto-transport characteristics1–4. To enrich their functionality further, extensive investigations have continued on iron-based ferromagnetic materials including ordered alloys5, oxides6 and nitrides7. In this study, we exemplify magnetic sensor functions in a ferromagnetic iron-tin alloy that is fabricated to harness massive Dirac bands of the kagome metal Fe₃Sn₂ (ref.8). Magnetic sensors are capable of electrically detecting a magnetic field9 and are becoming increasingly important towards the acceleration of Internet of Things. Their applications include monitoring of electric current via the Oersted field, electronic compasses and motion detection of mechanical parts in microdevices. In conventional semiconductor Hall sensors, the detection of a magnetic field B (termed instead of magnetic induction, hereafter) relies on the ordinary Hall effect, which converts a flow of electric current to the transverse Hall voltage $V_{yx}$ (ref.10). Since the output $V_{yx}$ is proportional to B and the injection current, good sensor performance under a constant input voltage is achieved in III–V semiconductors such as GaAs, InAs, and InSb with a high carrier mobility10. These semiconductor devices are constructed essentially on highly crystalline films with a low carrier density precisely tuned by high-temperature growth. Their bandgaps are, however, inevitably accompanied by substantial temperature (T) dependences of device characteristics. To ensure the stable operation in a wide T range, an external circuit that compensates the T dependence needs to be implemented.

Without changing the basic device structure, the semiconductor can be replaced by a ferromagnetic material if the anomalous Hall effect (AHE) is sufficiently large and provides a linear response to the applied B. $V_{yx}$ induced by AHE is a nominal function of magnetization M and is proportional to tangent of Hall angle, the ratio of Hall resistivity to longitudinal resistivity; materials design is thus better guided with tangent of Hall angle than mobility and carrier density. However, conventional ferromagnetic metals such as Fe, Co, and Ni show low tangent of Hall angle values of less than approximately 0.01 (ref.11), and are not suited to AHE-type Hall sensors. In the past two decades, significant progress has been made in the development of AHE materials and understanding of their physical origins11. Of particular interest is the intrinsic AHE where Berry curvature arising from electronic band topology acts as an effective magnetic field and can produce a large tangent of Hall angle and $V_{yx}$.  

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In this context, a ferromagnetic Fe-Sn compound, Fe₃Sn₂ (Curie temperature $T_C = 657$ K), is attracting attention because of its very large AHE at room temperature. The crystal structure consists of alternate stacking of stanene and a bilayer of Fe₃Sn with a kagome network of Fe, as illustrated in Fig. 1a. A recent angle-resolved photoemission spectroscopic study proposed that an interplay of the kagome lattice, which in analogy to graphene produces linearly dispersed bands and Dirac points, and spin-orbit coupling yields massive Dirac bands that concentrate Berry curvature. By positioning the Fermi level within the gap, e.g. with electrostatic gating and impurity doping, quantized AHE may be realized at room temperature. Fe₃Sn₂ is, however, the high-temperature phase stable above 607 °C; the bulk crystal is formed by a quenching technique. Recognizing the uniqueness of Fe₃Sn₂, we focus on the thin film of Fe-Sn kagome compounds as a candidate for AHE-type Hall sensors. Here we demonstrate magnetic sensor functions of nanocrystalline Fe-Sn alloy films prepared by room-temperature sputtering. Despite the lack of macroscopic lattice order, these films clearly bear characteristics of the crystalline Fe-Sn phase diagram and exhibit large and linear AHE responses as in the Fe₃Sn₂ bulk. This metal-based Hall device can outperform conventional semiconductor Hall sensors in thermal stability. The integration on a bendable polymer sheet thanks to the room-temperature fabrication is demonstrated for potential use in flexible electronics. These findings should accelerate challenges to exploitation of exotic physics hosted by iron and other transition-metal kagome compounds.

**Results**

Fe-Sn nanocrystalline films grown by co-sputtering at room temperature. FeₓSn₁₋ₓ alloy films were fabricated by a co-sputtering technique. The two elements were supplied from a single magnetron cathode by mounting Fe chips on a Sn target (Fig. 1c inset and also see Methods section). In the Fe-Sn binary members, antiferromagnetic FeSn (Néel temperature $T_N = 368$ K) and ferromagnetic Fe₃Sn₂ are displayed in the lower side. The device structure is shown schematically. An excitation current $I$ was injected to an Fe-Sn film on an insulating substrate, and longitudinal voltage $V_x$ and transverse Hall voltage $V_y$ were measured. A magnetic field $B$ is applied perpendicularly to the film plane. (c) Fe content $x$ in FeₓSn₁₋ₓ films was controlled by changing the Fe chip configuration on the Sn target. Error bars (standard deviations for $x$) are smaller than the symbols. A photograph when six Fe chips are placed is shown in the inset. The scale bar shows 10 mm. Blue, green, and, red broken lines correspond to $x = 0.50$ (FeSn), 0.60 (Fe₃Sn₂), and 0.75 (FeSn), respectively. (d) XRD patterns for FeₓSn₁₋ₓ films with $x = 0.54, 0.62, \text{and } 0.78$ grown on sapphire (0001) substrates at $T_g = 500$ °C. The data are shifted vertically for clarity. The film thicknesses were approximately 40 nm. See the text and Supplementary Fig. S1 for the phase identification of these films. (e) XRD patterns for 40-nm-thick Fe₀.₆₀Sn₀.₄₀ films on sapphire, glass and, PEN sheet substrates prepared at room temperature. (f) Cross-sectional high-resolution transmission electron microscopy image of a room-temperature sputtered Fe₀.₆₀Sn₀.₄₀ film on a sapphire substrate. The scale bar shows 5 nm. The inset shows a selected area electron diffraction pattern, revealing the presence of nanocrystalline domains.
of films with various \( x \) in the nanocrystalline film with \( x \) than quantifying physical properties of each phase. For phase analysis, we hereafter focus on composition dependent AHE characteristics in these mixed-phase films rather than quantifying physical properties of each phase.

For Fe composition dependences, we performed control experiments using high-temperature grown polycrystalline Fe\(_{0.62}\)Sn\(_{0.38}\) film (open green circles). For reference, the data of Fe\(_3\)Sn\(_2\) bulk in literature (ref.8) are also included (filled black squares). The results for polycrystalline Fe\(_{0.60}\)Sn\(_{0.40}\) film (filled green circles) and a polycrystalline Fe\(_{0.62}\)Sn\(_{0.38}\) film (open green circles). For reference, the data of Fe\(_3\)Sn\(_2\) bulk in literature (ref.8) are also included (filled black squares).

Comparison of anomalous Hall responses in nanocrystalline and polycrystalline films. Contrary to a naive expectation that the large AHE driven by band topology of the kagome lattice should smear out in such nanocrystalline films, we observed a clear AHE in the room-temperature grown nanocrystalline Fe\(_3\)Sn\(_2\)-like domains. The above XRD results are consistent with the bulk behavior. As shown in Fig. 1e, the formation of Fe\(_3\)Sn is found to be suppressed by room-temperature sputtering. Also, macroscopically, the room-temperature grown films do not have crystalline character. Cross-sectional transmission electron microscopy, however, reveals the presence of nanocrystalline domains with typical sizes of as small as a few nanometers, as displayed in Fig. 1f (scale bar, 5 nm). Although \( d \)-spacings values calculated from the selected-area electron diffraction pattern (Fig. 1f inset) are not uniquely indexed with one of the three compounds, the clearly visible layered lattice, together with the detailed characterizations (Fig. S2 in the Supplementary Information), suggests the existence of Fe\(_3\)Sn\(_2\)-like domains in the nanocrystalline film with \( x = 0.60\). Considering that further characterization is required for the comprehensive phase analysis, we hereafter focus on composition dependent AHE characteristics in these mixed-phase films rather than quantifying physical properties of each phase.

**Figure 2.** AHE in nanocrystalline Fe-Sn alloy films. (a,b) Hall resistivity \( \rho_{xy} \) (a) and out-of-plane magnetization \( M \) (b) at \( T = 300 \) K measured for room-temperature sputtered nanocrystalline Fe\(_x\)Sn\(_{1-x}\) films as a function of an out-of-plane magnetic field \( B \). The film magnetization was extracted by subtracting diamagnetic contributions from the data measured. (c,d) The results for polycrystalline Fe\(_x\)Sn\(_{1-x}\) films grown at \( T_g = 500 \) °C. (e) \( x \)-dependence of tangent of Hall angle, \( \rho_{xy}/\rho_{xx} \), at \( B = 2 \) T and \( T = 300 \) K for nanocrystalline (filled black circles) and polycrystalline Fe\(_x\)Sn\(_{1-x}\) films (open black circles). The inset shows \( \rho_{xx} \) (triangles) and \( \rho_{xy} \) (squares) for nanocrystalline Fe\(_x\)Sn\(_{1-x}\). (f) \( T \)-dependence of Hall conductivity \( \sigma_{xy} \) for a nanocrystalline Fe\(_{0.60}\)Sn\(_{0.40}\) film (filled green circles) and a polycrystalline Fe\(_{0.62}\)Sn\(_{0.38}\) film (open green circles). For reference, the data of Fe\(_3\)Sn\(_2\) bulk in literature (ref.8) are also included (filled black squares).
similar trend is also seen for the polycrystalline films that partly contain FeSn, Fe$_3$Sn$_2$, and Fe$_3$Sn (Fig. 1d). Note that such a composition dependence is not expected for mere Sn-rich $\alpha$-Fe.

In Fig. 2f, $T$ dependence of Hall conductivity $\sigma_{yx} = \rho_{yx}/(\rho_{xx}^2 + \rho_{yx}^2)^{1/2}$ is compared for nanocrystalline and polycrystalline films with $x \sim 0.6$ and also bulk Fe$_3$Sn$_2$ in literature. The occurrence of nearly $T$-independent $\sigma_{yx}$ in the polycrystalline films, which resembles the intrinsic behavior in Fe$_3$Sn$_2$ single crystals, but with a much smaller $\sigma_{yx}$ than the bulk value suggests a small fraction of Fe$_3$Sn$_2$ domains crystalized by high-temperature sputtering ($T_s = 500$ °C). Antiferromagnetic FeSn that persistently exists in the polycrystalline film, as found in the decreased $\sigma_{yx}$ at room temperature, does not give positive contributions to the AHE. In stark contrast, $\sigma_{xy}$ in the nanocrystalline film rivals the bulk data in the entire $T$ range. One obvious reason for this is the suppression of FeSn by room-temperature sputtering. Although it is not clear whether the mechanism of the large AHE discussed for Fe$_3$Sn$_2$ single crystals (ref.8) is valid for such nanocrystalline Fe-Sn films, these observations demonstrate that the AHE in the Fe-Sn alloy system is maximized at specific compositions. The large $\rho_{xx}$ and $\rho_{yx}/\rho_{xx}$ observed for $x = 0.77$ may suggest that ferromagnetic Fe$_3$Sn (refs9,10) is also a large AHE material, though the transport properties have not been clarified yet.

**Thermal stability of the AHE in room-temperature deposited films.** Having confirmed the large AHE in the nanocrystalline Fe$_{0.60}$Sn$_{0.40}$, we now turn to the characterization as a magnetic sensor element. Taking advantages of room-temperature sputtering, we extend our investigation to more commercially available substrates, glass and flexible polyethylene naphthalate (PEN) sheet (Fig. 3a, also see Fig. 1e for their XRD patterns). $T$-dependent AHE characteristics of Fe$_{0.60}$Sn$_{0.40}$ films with thickness $d = 40$ nm, displayed in Fig. 3b, are essentially similar on the three substrates, demonstrating that specific substrates are not required to achieve the large AHE. Magnetic-field sensing can be performed in the almost linear $\rho_{xx} - B$ region, and the differential coefficient, $\alpha = d\rho_{xx}/dB$, corresponds to the sensitivity for $B$ via $V_{yx}$. As displayed in Fig. 3c and inset, $\alpha$ is nearly constant to a large $B$ of approximately 0.5 T, and is rather insensitive to the $T$ variation (red colored regions in the inset). This is more clearly seen in the upper panel of Fig. 3d, where the temperature stability of $\alpha$ is tracked, defined as $\Delta\alpha = (\alpha (T) - \alpha (T = 300 K))/\alpha (T = 300 K)$. In a general operation range of $T = 200–400$ K, $\Delta\alpha$ is within a few percent, corresponding to approximately 0.02%/K. The small variation in $\rho_{xx}$, shown in the lower panel, gives an advantage over semiconductor devices that are restricted by inherent thermally activated transport.

**Characterization of Hall sensor responses and flexibility.** To enhance $V_{yx}$ further in view of the film thickness $d (V_{yx} = I \times \rho_{yx}/d)$, we examined the lower bound of $d$. Judging from the $d$ dependences of $\rho_{xx}$, $\rho_{yx}$, and $\rho_{yx}/\rho_{xx}$ at $B = 2$ T (Fig. 4a) and also $V_{yx}$ versus $B$ curves (Fig. 4b), we determine that the applicable large AHE persists down to $d = 4$ nm. The decrease to 2 nm is possible for $\rho_{xx}$, but is accompanied by a sharp rise in $\rho_{yx}$ and the drop of $\rho_{yx}/\rho_{xx}$. These increase power consumption when supplying a constant excitation current ($I$). In other
Figure 4. Magnetic sensor properties of nanocrystalline Fe$_{0.60}$Sn$_{0.40}$ films. (a) Thickness ($d$) dependences of $\rho_{xx}$, $\rho_{yx}$ and $\rho_{xy}/\rho_{xx}$ measured for a nanocrystalline Fe$_{0.60}$Sn$_{0.40}$ film on sapphire (0001) at $B = 2$ T. (b) $V_{yx}$ versus $B$ curves measured at $I = 0.1$ mA for $d = 40$ nm (black), 20 nm (blue), 10 nm (green), 4 nm (red), and 2 nm (brown). (c) $V_{yx}$ output characteristics as a function of $I$. (d) Out-of-plane magnetic field angle ($\theta$) dependence of $R_{yx}$ for a 4-nm-thick nanocrystalline Fe$_{0.60}$Sn$_{0.40}$ film on sapphire (0001). The measurement setup is shown schematically in the inset. The black dotted curve represents a relation $R_{yx} \propto \cos \theta$. (e) Anisotropic magnetoresistance measurement. Sheet resistance $\rho_{\text{sheet}}$ was measured in an in-plane $B$. The in-plane rotation angle ($\phi$) is defined in the inset. The dotted curve is a fitting result using a $\cos^2 \phi$ function. (f) Bending effects on transport properties of a 4-nm-thick Fe$_{0.60}$Sn$_{0.40}$ film on PEN. See Fig. 4g for the definition of x-bent and y-bent. The sample was first measured without bending (flat, black curves), and subsequently characterized under x-bent (red) and y-bent (blue) conditions. After these cycles, the device recovered back to the initial flat state (Fig. S5). (g) Photographs of the x-bent and y-bent devices. 4-nm-thick films on PEN sheet substrates were mounted on the surface of a 4.9-mm-radius semicircular jig in two different geometries.

words, at the fixed input voltage, $V_{yx}$ is reduced by the decreased $I$. The 1-nm-thick device was no longer conductive; $d$ of approximately 2 nm may be the critical thickness where island-like domains start to coalesce and form conduction paths. As presented in Fig. 4c, by injecting $I = 10$ mA into the 4-nm-thick device, a large $V_{yx}$ exceeding 0.1 V is generated from a magnetic field of $B = 0.5$ T.

We would like to here note some specific features, which are potentially utilized for three-dimensional magnetic-field sensing. Figure 4d shows out-of-plane magnetic field angle dependences of $R_{xx}$ under various $B$, measured for a 4-nm-thick Fe$_{0.60}$Sn$_{0.40}$ film on sapphire (0001) (the inset: the schematic measurement configuration). At $B = 9$ T, $R_{xx}$ obeys a $\cos \theta$ relation (black dotted curve) as expected from $M_{\text{eff}} \propto B_{\text{eff}} = B \cos \theta$ with $M_{\text{eff}}$ and $B_{\text{eff}}$ being the out-of-plane components of magnetization and magnetic field, respectively. As $B$ is decreased, because of the in-plane magnetic easy axis (see Fig. S3 in the Supplementary information), the actual direction of $M$ vector becomes not to fully follow that of $B$ vector, resulting in a deviation from the $\cos \theta$ relation. Also, the in-plane magnetoresistance $R_{\text{sheet}}$ vs $\phi$, shown in Fig. 4c, indicates an anisotropic magnetoresistance effect even at low $B = 0.25$ T (the inset: the schematic measurement configuration). By combining these anisotropic responses of $R_{xx}$ and $R_{\text{sheet}}$, the magnetic field vector $B(\theta, \phi)$ could be detected with a simple Hall-bar device.

Nanocrystalline Fe$_{0.60}$Sn$_{0.40}$, as demonstrated above, can be served as a Hall-type magnetic sensor. In particular, the capability of sensor integration onto a flexible substrate is appealing, potentially finding applications in flexible electronics. We examined the mechanical bending effect on a nanocrystalline Fe$_{0.60}$Sn$_{0.40}$ device using flexible PEN substrates. Figure 4f demonstrates that, even under severe bending conditions (see Fig. 4g for the definition of bending geometries), the nanocrystalline Fe$_{0.60}$Sn$_{0.40}$ device on PEN offers a reversible operation with an almost unchanged sensor performance (Fig. S5 in the Supplementary Information). Such new functionality enabled by nanocrystalline Fe$_{0.60}$Sn$_{0.40}$ films, in combination with its economically and environmentally friendly ingredients, would offer a new type of magnetic sensor design utilizing AHE.

Discussion and Conclusions

Large $\rho_{yx}$ at room temperature has also been obtained in ferromagnetic semiconductors and metal-insulator composites. According to the established classification of AHE origins, those highly resistive materials, however, are in the poorly conductive region ($\sigma_{xy} < 3 \times 10^7 \Omega^{-1}$ cm$^{-1}$). Our nanocrystalline Fe$_{0.60}$Sn$_{0.40}$ is essentially a metal with $\sigma_{xy}$ as high as $10^3$–$10^4$ $\Omega^{-1}$ cm$^{-1}$, being in a different category called the intrinsic region (Fig. S6 in the Supplementary Information). In fact, it is observed in nanocrystalline Fe$_{0.60}$Sn$_{0.40}$ films that $\sigma_{xy}$ is rather independent of $\sigma_{xx}$, as being consistent with intrinsic mechanisms. At present, the sensitivity of our device is about one order of magnitude lower than those of the state-of-the-art GaAs and Si Hall sensors. If the Berry curvature mechanism holds for the AHE in nanocrystalline Fe$_{0.60}$Sn$_{0.40}$ films, the device performance could be further improved by Fermi-level tuning into the gap at the Dirac point. Such an intrinsic approach, in addition
to its critical importance for the next-generation of Hall sensors, may also lead to devices that incorporate exotic quantum transport phenomena, e.g., quantized AHE. We believe that the thin-film structure would be the key enabler for exploration of new functionality that emerges on the kagome lattice.

Methods

**Thin-film growth.** Fe<sub>1−x</sub>Sn<sub>x</sub> alloy films were fabricated by RF magnetron sputtering. The RF power was 50 W, and Ar gas pressure was 0.5 Pa for x < 0.87 and 0.8 Pa for x = 0.87. The typical growth rate was approximately 4 nm/min as checked by X-ray reflectivity measurement and also with a surface profiler. For films with d < 4 nm, the surface was covered with a 15-nm-thick SiO<sub>2</sub> insulating layer to prevent oxidation. The SiO<sub>2</sub> layer was formed by RF magnetron sputtering using a SiO<sub>2</sub> target at an Ar gas pressure of 0.5 Pa. Compositional analysis of the films was performed with energy-dispersive X-ray spectroscopy and inductively coupled plasma atomic emission spectroscopy.

**AHE and magnetization measurements.** Electrical transport properties were measured with a VersaLab, a Physical Property Measurement System (Quantum Design) and a source-measure unit. Films were patterned into a Hall-bar structure (1b), and electrical contacts were made with an indium solder. The aspect ratio of electrode-electrode distance for V<sub>xx</sub> versus that for V<sub>xy</sub> was approximately unity. To remove thermoelectric and geometric effects, the measured data were symmetrized for V<sub>xx</sub> and anti-symmetrized for V<sub>xy</sub> against B as widely adopted to these measurements. Magnetization measurements were carried out using a vibrating sample magnetometry mode of VersaLab.

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Acknowledgements

The authors thank K. Nakahara, M. Kawasaki, H. Kato, N. Shibata, and H. Nishikawa for their helpful advice and K. Takanashi, S. Ito and F. Sakamoto for their assistance with experiments. This work was partly supported by JSPS KAKENHI (Grant Nos. 25000003 and JP15H05853) from the Japan Society for the Promotion of Science and Kumagai Foundation for Science and Technology.
Author Contributions
A.T. and K.F. designed the experiments. K.F. and Y.S. fabricated samples. Y.S., K.F. and J.S. performed electrical measurements. Y.S., J.S. and T.S. contributed to magnetization measurements. Y.S., K.F. and A.T. wrote the manuscript. All authors discussed the results.

Additional Information
Supplementary information accompanies this paper at https://doi.org/10.1038/s41598-019-39817-8.

Competing Interests: The authors declare no competing interests.

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