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Giant anomalous Hall effect in Fe-based microwires grown by focused-electron-beam-induced deposition

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Abstract
We report the temperature dependence of the resistivity, the anisotropic magnetoresistance and the Hall effect of iron microwires grown by focused-electron-beam-induced deposition. By modifying the growth conditions in a controllable way, we study wires with iron compositions varying from 45% to 70%, which present different electrical conduction mechanisms, with resistivity values differing over three orders of magnitude. The magnetoresistance depends highly on the composition, and it can be understood by a subtle interplay between the anisotropic magnetoresistance and intergrain magnetoresistance due to their complex microstructure, consisting of an iron–carbon–oxygen amorphous matrix. A giant value for the anomalous Hall effect is found, which we explain by a large contribution of the skew scattering mechanism. The present results emphasize the correlation between the exotic microstructure of the microwires, and their magnetotransport properties.

(Some figures may appear in colour only in the online journal)

1. Introduction
The growth of nanostructured materials in one single step on any targeted location of a substrate and with a remarkable control in the lateral width and thickness at the nanometre scale is the main advantage of the focused-electron/ion-beam-induced deposition techniques, FEBID and FIBID, respectively [1, 2]. Together with the conventional applications of these techniques in the semiconductor industry for mask repair and circuit editing, they also offer exciting perspectives for the growth of functional materials, such as the Fe-based microwires reported here.

The growth mechanism of FEBID/FIBID is considered to be a chemical vapour deposition (CVD) process assisted by a focused-electron-beam (FEB) or a focused-ion-beam (FIB). Most commonly, an organometallic compound is used as the precursor gas of the material to be grown. The decomposition of the precursor material is often not complete under standard working conditions; high vacuum (∼10⁻⁶ mbar), room temperature and a relatively low generation of secondary electrons (which are mainly responsible for the dissociation of the precursor bonds). Hence, the deposited material is usually composed of the desired (metallic) element plus carbon and oxygen originated from the incomplete dissociation of the molecules as well as the residual hydrocarbon species present in the vacuum chamber. Different strategies have recently been devised for the purification of some of these nanodeposits in order to improve their metal content and consequently their
physical properties [3,4]. However, as shown hereafter, the FEBID technique can produce Fe–C–O nanostructures in a wide range of compositions, and with a specific microstructure that may result in interesting magnetotransport physical properties which could find applications in magnetic sensitive sensors.

Nanofabrication of magnetic materials by FEBID has recently attracted significant attention due to their potential application in fields such as magnetic storage, sensing and logic. In particular, magnetic Co-based nanostructures fabricated by FEBID have been reported using Co2(CO)8 as the precursor material [5,6]. Tuning the growth parameters, pure Co wires (down to lateral size of 30 nm) have been created by means of FEBID, exhibiting similar physical properties to bulk polycrystalline cobalt [7–10]. Many studies based on Fe nanostructures fabricated by FEBID have been published using different precursor materials: Fe(CO)5, Fe2(CO)9 and Fe2(CO)12 [11–23]. Remarkably, when the growth is performed under ultra-high vacuum conditions, it is possible to achieve pure-Fe nanostructures [19–21,23]. There are, however, except for our previous work on this topic [22], hardly any reports on the transport properties of Fe nanostructures grown by FEBID.

In this paper, we present a detailed study of the magnetotransport properties of FEBID-grown Fe-based microwires with a variable Fe content and microstructure. A wide range of temperatures and applied magnetic fields has been explored in order to deeply investigate the magnetotransport mechanisms. Tuning the Fe content and microstructure of the microwires allowed us to investigate the change in the magnetotransport properties with different conduction mechanisms. In particular, a metal–insulator transition (MIT) has been observed as a function of Fe content [22], as commonly observed in disordered semiconductors [24,25]. We put forward [22] that the large change in Fe content results in a progressive evolution of the magnetoresistance (MR), evolving from anisotropic magnetoresistance (AMR) to intergranular tunnel magnetoresistance (ITMR). Moreover, Hall effect (HE) measurements unveil a giant anomalous Hall contribution (AHE), which recently regained interest, providing new insights into the physics of electron scattering in strongly correlated systems [26]. In our samples, the AHE is due to strong electron scattering produced by magnetic impurities, which are present as iron and iron-oxide species embedded in an amorphous Fe:O:C matrix [22].

### 2. Experimental details

Fe-based microwires were grown by FEBID as explained in [22]. In short, the atomic Fe content was varied between 45% ± 2% and 70% ± 2% by changing the residual water pressure in the vacuum chamber via a leak valve. Field-emission scanning electron microscopy (SEM) images were taken in order to obtain the lateral size of the microstructures. The typical dimensions of the microwires are 1 µm in width, 200 nm in thickness and 6 µm in length.

Atomic force microscopy (AFM) measurements were carried out in a commercial Veeco TMC Nanoscope V to investigate the thickness and uniformity of the deposits.

### 3. Results and discussion

#### 3.1. Electrical properties as a function of temperature

The composition and resistivity (ρ) of the fifteen Fe-based microwires studied in this paper are shown in table 1. The sample number increases with the room-temperature resistivity (ρ300K), which changes from 186 µΩ cm for S 1 up to 3.84 × 107 µΩ cm for S 15, and it approximately decreases with the Fe content, as was shown previously [22]. The dependence of ρ with the temperature is shown in figure 1(a).

As expected from a Fe:O:C matrix, the resistivity depends heavily on the Fe content and ranges from a semiconducting behaviour at low Fe content to the one typical of bad metals at high Fe content. In the case of S 15, the resistivity is 1.25 × 107 µΩ cm at 130 K, which corresponds to the maximum measurable resistance (∼7 × 106 Ω) in the PPMS. Let us first concentrate on the residual resistivity ratio (RRR, defined as ρ300K/ρ2K), as shown in figure 1(b). For S 14, with a low Fe content of 56%, ρ changes more than one order of magnitude from 300 to 2 K (RRR ∼ 0.05), whereas for S 1 to S 4, with an Fe content close to 70%, the resistivity hardly changes with temperature (RRR ∼ 0.93).

In figure 1(c), we plot ρ as a function of the Fe content at T = 150 K. We can clearly observe a slope change in the resistivity versus Fe content around 55% (note the semilogarithmic scale). This is very similar to previous results in Si1−xNix [27] and in Pt-based FIBID nanowires [28] where a MIT was observed as a function of the Pt–C ratio. The overall ρ ranges between values expected for microwires with a high Fe content ∼100 µΩ cm (pure Fe ∼ 10 µΩ cm) and

<table>
<thead>
<tr>
<th>Samples</th>
<th>Fe (at%±2%)</th>
<th>O (at%±2%)</th>
<th>C (at%±2%)</th>
<th>ρ300K (µΩ cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S 1</td>
<td>70</td>
<td>22</td>
<td>8</td>
<td>186</td>
</tr>
<tr>
<td>S 2</td>
<td>70</td>
<td>22</td>
<td>8</td>
<td>281</td>
</tr>
<tr>
<td>S 3</td>
<td>64</td>
<td>27</td>
<td>9</td>
<td>355</td>
</tr>
<tr>
<td>S 4</td>
<td>64</td>
<td>27</td>
<td>9</td>
<td>498</td>
</tr>
<tr>
<td>S 5</td>
<td>63</td>
<td>27</td>
<td>10</td>
<td>512</td>
</tr>
<tr>
<td>S 6</td>
<td>70</td>
<td>23</td>
<td>7</td>
<td>526</td>
</tr>
<tr>
<td>S 7</td>
<td>61</td>
<td>34</td>
<td>5</td>
<td>566</td>
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<tr>
<td>S 8</td>
<td>63</td>
<td>27</td>
<td>10</td>
<td>658</td>
</tr>
<tr>
<td>S 9</td>
<td>66</td>
<td>25</td>
<td>9</td>
<td>760</td>
</tr>
<tr>
<td>S 10</td>
<td>58</td>
<td>35</td>
<td>7</td>
<td>952</td>
</tr>
<tr>
<td>S 11</td>
<td>62</td>
<td>31</td>
<td>7</td>
<td>1373</td>
</tr>
<tr>
<td>S 12</td>
<td>58</td>
<td>36</td>
<td>6</td>
<td>2082</td>
</tr>
<tr>
<td>S 13</td>
<td>56</td>
<td>39</td>
<td>5</td>
<td>3344</td>
</tr>
<tr>
<td>S 14</td>
<td>56</td>
<td>38</td>
<td>6</td>
<td>7306</td>
</tr>
<tr>
<td>S 15</td>
<td>44</td>
<td>54</td>
<td>2</td>
<td>3.84 × 10^5</td>
</tr>
</tbody>
</table>
with very poorly defined Fe oxides $> 10^4 \, \mu\Omega \, \text{cm}$. The $\rho$ versus temperature (see figure 1(a)) of the microwires with highest resistivities (S 14 and S 15) follows a $\rho(T) \sim \rho_0 \exp(T_0/T)^{1/2}$ dependence, indicating that the conduction can be explained by a variable range hopping (VRH) mechanism of electrons between localized states. On the other hand, samples S 1 to S 6, with a Fe content in the range $\sim 71\% \pm 2\%$ to $\sim 64\% \pm 2\%$, and $\rho_{300K} = 186$ to $526 \, \mu\Omega \, \text{cm}$, show conduction properties expected for a bad metal. Finally, samples S 7 to S 13, with Fe content in the range $\sim 66\% \pm 2\%$ to $\sim 55\% \pm 2\%$, and $\rho_{300K} = 658$ to $2082 \, \mu\Omega \, \text{cm}$, have a combination of both metallic and hopping conduction.

These results demonstrate that the conductivity is progressively degraded as the Fe content decreases below 55%, approaching an insulating phase for lower Fe content. The different Fe/O ratios (the carbon content does not vary significantly) as well as the different microstructures found in Fe-based microwires results in a MIT with a dramatic change in the temperature dependence of the resistivity.

3.2. MR as a function of temperature

The MR was studied in the fifteen aforementioned microwires, with magnetic fields applied perpendicularly to the substrate-plane up to $\pm 9 \, \text{T}$, and temperatures between 10 and 300 K.

The MR is defined hereafter in the standard way:

$$\text{MR} \% = 100 \frac{R(H) - R(H = 0)}{R(H = 0)}$$

where $R(H)$ and $R(H = 0)$ are the resistances at a magnetic field $H$ and at zero field, respectively. Taking into account the microwire dimensions, a coherent rotation of the magnetization is expected for the reversal of the magnetization under a perpendicular magnetic field.

The MR of four representative samples is shown in figure 2. The AMR and ITMR were determined at an applied magnetic field of $\pm 9 \, \text{T}$. The AMR is originated from an anisotropic scattering of electrons due to the spin–orbit coupling, while the ITMR is caused from a spin-polarized tunnelling mechanism between grains.

In figure 2(a), the MR for S 1 ($\rho_{300K} = 186 \, \mu\Omega \, \text{cm}$) is shown. For this low $\rho$ sample, the observed MR is purely caused by the AMR, where the magnetization is completely saturated at $\pm 2 \, \text{T}$. At low field, the MR has in this case a quadratic dependence with the field, as expected for coherent rotation of the magnetization [29]. An increase in the negative AMR is observed when the temperature is decreased: $\text{AMR}(300K) = -0.59\%$ and $\text{AMR}(10K) = -0.91\%$. The presence of a pure AMR signal in the MR is strong evidence of the percolation of the Fe network in S 1. However, the value of AMR is high compared with the bulk Fe value, $-0.2\%$ [30]. This can be explained by taking into account the microstructure of these wires, composed by an amorphous matrix (Fe::O::C ) with small $\alpha$-Fe crystals (1 nm–2 nm in size) [22]. The Fe atoms, occupying many surface/interface positions, are less prone to quenching effects produced by the lattice crystal field and will likely show an enhanced spin–orbit coupling. This enhanced spin–orbit coupling would be at the base of the enhanced AMR. The strong decrease in the AMR value observed when increasing the temperature from 10 to 300 K also indicates that thermal fluctuation effects are large as expected in systems with enhanced interface or bond-defective magnetic atoms [31].

The MR of S 9, with $\rho_{300K}$ of 760 $\mu\Omega \, \text{cm}$, is shown in figure 2(b). The MR presents both AMR and ITMR

Figure 1. (a) Resistivity of Fe-based microwires as a function of the temperature. (b) RRR as a function of the resistivity at room temperature. (c) Resistivity of the microwires as a function of the Fe content at 150 K. The error bar in the Fe content is the standard deviation of the analysed spectra and the error bar in resistivity data was calculated assuming a 20% error of its value. Resistivity values of Fe oxides at room temperature are indicated.
Figure 2. Perpendicular MR as a function of the applied magnetic field at different temperatures. (a) S 1; (b) S 9; (c) S 12; (d) S 14. (e) MR of all the Fe-based microwires as a function of resistivity at room temperature, where the samples shown in (a)–(d) are marked with arrows. Note that S 1 and S 12 were measured exclusively for positives magnetic fields; a visual-guide line has been drawn to indicate the extrapolated MR at negative values. AMR and ITMR were determined at an applied magnetic field of ±9 T. (f) MR of the Fe-based microwires shown in (a)–(d) as a function of the temperature.

...The ITMR is caused by the spin-polarized tunnelling of electrons between metallic grains in granular ferromagnets, and is often difficult to saturate even under very high magnetic fields [32]. The MR (±9 T) is −0.37% at 300 K and −0.66% at 100 K, becoming temperature independent below 100 K. Thus, the MR is dominated here by the AMR at high temperatures, from 300 K down to 200 K, where the MR is completely saturated above 1.5 T. However, in the range 150 to 25 K,
both AMR and ITMR effects coexist. Below 150 K, the MR does not saturate at the maximum field of ±9 T, as observed in materials with MR dominated by the ITMR [32]. This is well correlated with the microstructure at that composition, formed by both Fe and Fe-oxide-based grains embedded in the Fe : O : C matrix [22].

Two examples of Fe-based microwires where the ITMR fully dominates the MR signal are shown in figures 2(c) and (d). The MR of S 12, ($\rho_{300K} = 2082 \mu \Omega \cdot \text{cm}$), is shown in figure 2(c). The MR does not saturate at any temperature even under application of ±9 T. An increase in the MR is observed as T is decreased from room temperature, MR(300 K) = −0.36% and MR(100 K) = −0.69%, but it remains independent of temperature below 100 K. The increasing dominance of the ITMR effect is explained by increasing amount of oxygen (Fe/O = 1.61), which suggests the presence of a significant amount of iron-oxide grains in the microwire. From the full dominance of the ITMR effect in this wire, as well as its resistivity value close to the metallic limit established by the Mott–Anderson theory for noncrystalline materials ($\rho = 3000 \mu \Omega \cdot \text{cm}$)[24], makes us conclude that S 12 does not present metallic conduction. A similar discussion can be made about S 14 ($\rho_{300K} = 7306 \mu \Omega \cdot \text{cm}$). For this microwire, the dominance of the ITMR effect is even more evident, with a MR value of −2.7% at ±9 T and 10 K.

In figure 2(e) we plot the MR at ±9 T versus $\rho_{300K}$ for all the microwires and we mark the four microwires shown in figures 2(a)–(d) by means of arrows. A clear trend in the MR is observed, evolving from MR mainly governed by the AMR for moderate resistivities to a high ITMR for high resistivity microwires.

Finally, figure 2(f) shows the MR at ±9 T of the four samples previously discussed as a function of temperature. Thermal effects are noticeably more important in samples where the MR is dominated by the ITMR effect. This is probably a direct consequence of the spin-polarized tunneling mechanism governing in the ITMR effect [32]. In the case of samples dominated by the AMR, the temperature influence is less decisive and can stem from changes in phonon–electron scattering, combined with thermal fluctuations acting on magnetic atoms at the interface with the matrix.

Summarizing this section, the Fe-based microwires present a MIT transition tuned by their Fe content and their microstructure. Consequently, the MR behaviour presents a gradual transition from AMR to ITMR as the Fe/O ratio decreases, which can be correlated with the microwire resistivity. The high value of the MR compared with bulk Fe films [30] indicates that the electron scattering is enhanced for FEBID-grown Fe microwires. As shown in the next section, this fact has a remarkable impact on the HE.

3.3. Giant HE as a function of temperature

Hall resistivity measurements in magnetic materials provide useful information on the electron scattering processes, the carrier concentration and the magnetization processes as a function of the magnetic field. The Hall resistivity is

$$\rho_H = \frac{V_H I}{t} = \rho_{\text{OHE}} + \rho_{\text{AHE}} = \mu_0 (R_0 H + R_S M)$$

where $V_H$ is the measured Hall voltage, transversal to the current ($I$) direction and $t$ is the microwire thickness. In magnetic materials, $\rho_{\text{OHE}}$ is given by the ordinary ($\rho_{\text{OHE}}$) and anomalous ($\rho_{\text{AHE}}$) contribution, where $R_0$ and $R_S$ are the ordinary and anomalous Hall coefficients, respectively. In our previous work, a few room-temperature Hall measurements in selected samples were presented in order to demonstrate the ferromagnetic character of the Fe microwires [22]. However, no quantitative analysis of the data was carried out, which has only been possible by systematic measurements in the whole temperature range and for all the microwires of this study.

The ordinary HE (OHE) contribution was determined by fitting the slope at high magnetic fields, beyond magnetic saturation. The AHE contribution was determined at low magnetic fields by extrapolating the slope at $\mu_0 H = 0$. From the coincidence point of the linear fits for the OHE and the AHE, the anisotropy field $N M_S$ was obtained, where $N$ is the out-of-plane demagnetization factor of the microwire and $M_S$ is the saturation magnetization [$7, 33$] (see figure 4(a)). Using this method, a clear correlation between the room-temperature $M_S$ and the longitudinal resistivity is found as shown in figure 3. As expected, the $M_S$ decreases as a function of the longitudinal resistivity, from $1371 \pm 16 \text{ emu cm}^{-3}$ down to $706 \pm 20 \text{ emu cm}^{-3}$; since this is directly correlated with the Fe content ($M_S$(pure Fe) = $1700 \text{ emu cm}^{-3}$, $M_S$(iron oxides) = $300–600 \text{ emu cm}^{-3}$) and thus with the intensity of the ferromagnetic interaction in the sample. However, the main information extracted from the Hall measurements comes from the analysis of the AHE in terms of the electron scattering processes.

In figures 4(a)–(c), the Hall resistivity as a function of magnetic field for three of the wires where the MR was discussed in section 3.2 (S 1, S 9, S 12) is plotted at different temperatures. The temperature dependence of the Hall resistivity at 2 T is plotted in figure 4(d). S 14 is not included here due to a noisy HE signal which hampers a meaningful analysis.
Figure 4. Hall resistivity as a function of the applied magnetic field at different temperatures. The $\rho_{\text{OHE}}$ is determined at high fields, whereas the $\rho_{\text{AHE}}$ is calculated by extrapolating the OHE slope at zero field. From the intersection of the linear fits the value $N M_S$ is obtained ($N$ demagnetizing factor, $M_S$ saturation magnetization), an example is shown in (a) for S 1 at 10 K [7]. (a) S 1; (b) S 9; (c) S 12; (d) Anomalous Hall resistivity at 2 T as a function of temperature for the microwires shown in (a)–(c).

The first remarkable result is the high absolute value of $\rho_H$ for the three microwires (in the range $10–150 \mu\Omega \text{cm}$) when compared with the values obtained in epitaxial Fe [33], Fe microwires [23] and oxidized-iron films [30]. In fact, even in epitaxial Fe films with enhanced surface scattering, the Hall resistivity is never higher than $5 \mu\Omega \text{cm}$ [33]. Similarly, oxidized-iron films show a Hall resistivity of the order of $1 \mu\Omega \text{cm}$ [30]. On the other hand, we note that very thin epitaxial Fe$_3$O$_4$ films ($<40$ nm) show large room-temperature AHE ($\rho_H \sim 10–40 \mu\Omega \text{cm}$), which was attributed to the enhanced electron scattering at antiphase-boundary defects formed during the growth of the films on MgO (0 0 1) substrates [34]. Thus, although the AHE in Fe microwires should be generally small ($<1 \mu\Omega \text{cm}$), additional electron scattering mechanisms could give rise to an enhanced AHE. This hypothesis is certainly interesting since appropriate tailoring of the microstructure and composition of the Fe microwires could result in a giant AHE. For the FEBID Fe-based microwires studied here, the giant AHE is due to the heterogeneous and disordered nature of the deposits [22]. First, this microstructure will likely enhance the electron scattering inside the amorphous matrix as the electron mean free path is strongly reduced in amorphous materials. Secondly, the presence of small iron and iron-oxide grains will be another source of enhanced electron scattering as frequently observed in granular ferromagnetic materials [35]. It is well known that the AHE value generally increases with the longitudinal resistivity provided that the degree of ferromagnetism is maintained [26]. Thus, we argue hereafter that the explanation for the giant AHE observed in our Fe microwires is caused by their microstructure, which produces an excess of electron scattering. Some of these electron scattering processes will be spin-dependent, contributing to the giant AHE.

The first clue on the origin of the observed giant AHE is its strong dependence on the longitudinal resistivity, as represented in figure 5(a), for the three samples here considered. As a first approximation, a linear dependence is noticed, suggesting the ‘skew’ scattering mechanism as
The three representative microwires selected here show different behaviour in this sense. S 12, with a high longitudinal resistance, will likely have larger contribution from $\rho_0$ than from $\rho_S$. When this occurs ($\rho_S \ll \rho_0$), formula (3) is reduced to $\rho_{AHE} \approx \gamma \rho_0 \rho_S \propto \rho_{xx}$. An approximately linear dependence is thus expected between the AHE resistivity and the longitudinal resistivity (mainly due to ballast centres), as observed in figure 5(a). Therefore, $\rho_S$ does not vary significantly with temperature and the temperature-dependent $\rho_0$ term governs the AHE dependence. However, the quadratic term of the expression (3) cannot be neglected in samples S 1 and S 9, where the longitudinal resistivity is significantly lower and the $\rho_S$ term is expected to contribute more significantly to the total value of $\rho_{xx}$. This is illustrated through the analysis of the results in S 1, where the lowest longitudinal resistivity is present. As shown in figure 5(b), the Hall resistivity in this sample displays a maximum around 125 K. This can be plausibly explained by the relative contribution of the linear and the quadratic terms in formula (3). $\rho_0$ is expected to increase with temperature due to the increasing electron–phonon scattering. This explains the initial increase of the anomalous Hall resistivity from 10 to 100 K, provided that $\rho_S$ does not change significantly in that temperature interval. However, $\rho_S$ is expected to decrease with an increasing temperature because the magnetization, the spin polarization and the corresponding magnetic interactions will decay with temperature. This temperature-dependent scenario for $\rho_S$ is supported by the AMR, also displayed in figure 5(b), which reflects the same decay of these parameters above 100 K. Thus, above 100 K, the quadratic term in formula (3) will decay significantly and can produce an overall decrease in the anomalous Hall resistivity. It was already noted in section 3.2 that the decrease in the AMR with temperature was large in comparison with the one expected for pure Fe. This proves that thermal fluctuations are strongly affecting the ferromagnetism in these FEBID Fe microwires. This is not rare taking into account the microstructure of the sample, which consists of an heterogeneous amorphous Fe : O : C matrix and dispersed Fe and Fe-oxide grains. The nanometric crystalline particles of $\alpha$-Fe and Fe-oxide observed by HRTEM [22] will be prone to superparamagnetism at high temperature [37], thus diminishing the skew scattering processes. The microstructure also suggests many interfacial and bond-defective magnetic atoms with suppressed magnetism and with high tendency to be affected by thermal fluctuations.

4. Conclusions

We have carried out a thorough investigation of the transport properties of Fe-based microwires grown by FEBID by means of resistivity, magnetoresistance and Hall effect measurements, from 300 K down to 10 K. The Fe content of the wires can be tuned in a wide range, which is well correlated with the resistivity value of the microwire as well as with its residual resistivity ratio. The observed magnetoresistance has two contributions, coming from the AMR effect and the ITMR. The first one is more important in microwires with high Fe content and the second one in microwires...
with low Fe content. The main result of the study is the giant AHE, far greater than in pure-Fe nanowires, which has been explained in terms of the large skew scattering produced by the complex heterogeneous microstructure of these microwires. In microwires with high Fe content, a subtle interplay between contributions from spin-dependent and spin-independent scattering is invoked to account for the temperature dependence of the Hall resistivity. This interplay is reinforced by the important role played by thermal fluctuations in this heterogeneous system. In conclusion, we have shown that the use of the FEBID technique permits the growth of Fe-based microwires with a microstructure totally different to that produced by other growth techniques, with tremendous impact on the magnetotransport properties, especially the giant anomalous Hall effect observed here with applications in magnetic sensing, logic and storage.

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