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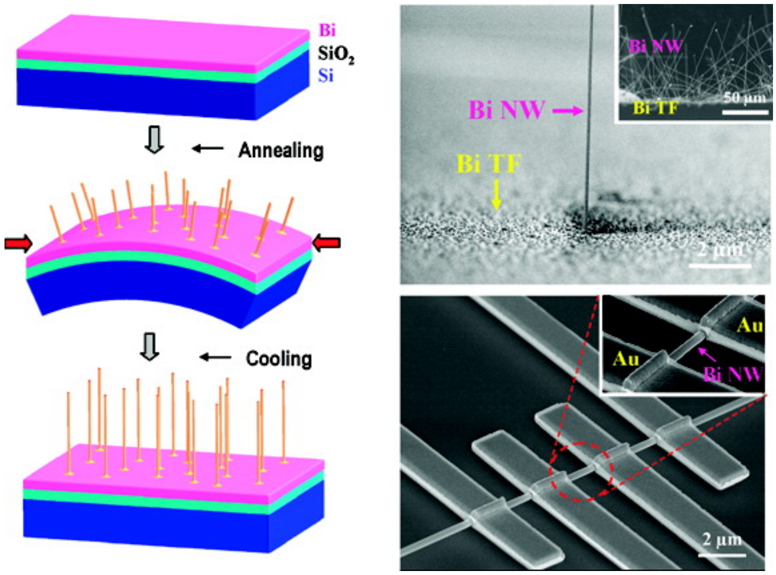

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On-Film Formation of Bi Nanowires with Extraordinary Electron Mobility

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ABSTRACT

A novel stress-induced method to grow semimetallic Bi nanowires along with an analysis of their transport properties is presented. Single crystalline Bi nanowires were found to grow on as-sputtered films after thermal annealing at 260−270 °C. This was facilitated by relaxation of stress between the film and the thermally oxidized Si substrate that originated from a mismatch of the thermal expansion. The diameter-tunable Bi nanowires can be produced by controlling the mean grain size of the film, which is dependent upon the thickness of the film. Four-terminal devices based on individual Bi nanowires were found to exhibit very large transverse and longitudinal ordinary magnetoresistance, indicating high-quality, single crystalline Bi nanowires. Unusual transport properties, including a mobility value of 76900 cm^2/(V s) and a mean free path of 1.35 μm in a 120 nm Bi nanowire, were observed at room temperature.

The search for new growth methods for one-dimensional (1D) structures[1] with nanometer diameters, such as nanowires, continues to be of central importance in nanoscience and nanotechnology, since the nanowires have great potential for testing and understanding their unusual quantum properties and for use in nanoscale devices.[2−4] In particular, bismuth (Bi) nanowires have been of great importance in nanophysics to both theorists and experimentalists, as Bi is known to be a group V semimetallic element that exhibits unusual transport properties due to its highly anisotropic Fermi surface.[5−9] Single-crystalline Bi nanowires have motivated many researchers to investigate novel quasi-one-dimensional phenomena such as the wire-boundary scattering effect[5,6] and quantum confinement effects[6] due to their electron effective mass (~0.001 m_e), which is the smallest of all known materials.[7] Moreover, single-crystalline Bi nanowires are expected to usher in new class of thermometric devices with high thermoelectric figure-of-merit (*ZT*) values.

Here, a novel stress-induced method for growing high-quality single-crystalline Bi nanowires is presented. With this method, the diameter, shape, and aspect ratio of Bi nanowires can be tuned by controlling the growth conditions. Furthermore, four-terminal devices based on individual Bi nanowires were successfully fabricated using a plasma etching technique to remove an oxide layer from the outer surface of the nanowires. Shubnikov−de Haas oscillations as well as the largest known values of ordinary magnetoresistance (OMR) in an individual Bi nanowire were observed, demonstrating that Bi nanowires grown by the proposed stress-induced method are high-quality single crystalline nanowires. It was also found that a Bi nanowire with a diameter of 120 nm exhibits an electron mobility of 76900 cm^2/(V s) and an extraordinary mean free path of 1.35 μm at room temperature, as measured by the electric field effects.

A number of growth methods for Bi nanowires[5,6,8−13] have been studied in an effort to understand their unique transport properties. Several template-based synthesis methods have been developed for the growth of Bi nanowires. These include electrochemical deposition[8,9] and pressure injection[5,6] using nanochannels of anodized aluminum oxide (AAO). Other methods requiring aqueous solution-phase[10−12] reagents and catalysts, e.g., Bi−CrN composites,[13] have also been reported as well. Although the investigations of magnetoresistance in arrays of Bi nanowires embedded in a porous alumina matrix have been reported,[4,6,8,9] to date, the transport properties in an individual single-crystalline Bi have not been studied, as there are a number of difficulties and limits in conventional methods with respect to device fabrication and obtaining high-quality single crystalline materials. In particular, electrical Ohmic contacts to the Bi nanowires are extremely difficult to produce due to a native Bi oxide layer

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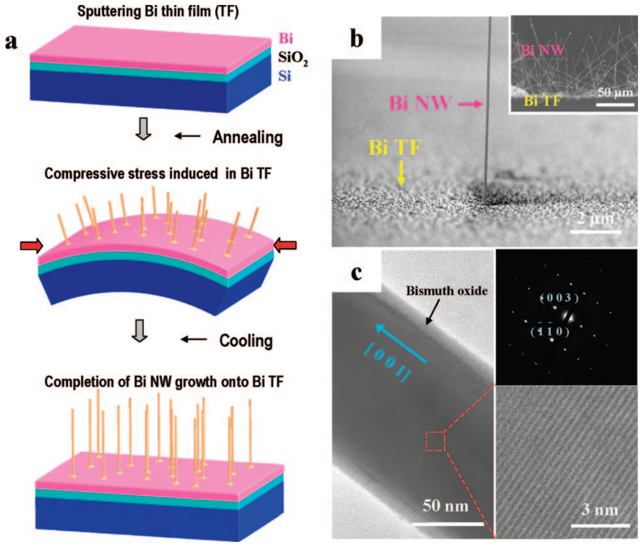

Figure 1. Growth mechanism and structural characteristic of the single-crystalline Bi nanowires. (a) A schematic representation of the growth of Bi nanowires by OFF-ON: (Step 1) Deposition of Bi thin films by an UHV rf-sputtering system. (Step 2) Initiating the thermal stress originating from a thermal expansion mismatch between the film and the substrate by annealing at 260−270 °C for 10 h. (Step 3) Completion of Bi nanowire growth by releasing the compressive stress after annealing. (b) A SEM image of a Bi nanowire grown on a Bi thin film: The inset clearly shows a side view of the Bi nanowires that extrude from the surface of the as-sputtered film. (c) A low-magnification TEM image of a Bi nanowire: the ED pattern (top right) of the nanowire along the $[\bar{1}10]$ zone axis indicates that the growth direction of the nanowire is [001], and a high-resolution TEM image (bottom right) of the Bi nanowire shows a perfect single-crystalline material without defects.

that forms on the outer surface of the nanowire and because of the very low melting point (271.3 °C) of the Bi.[14,15] Four-terminal devices based on individual single-crystalline Bi nanowires are of particular significance, as they provide an ideal system for investigating the unusual carrier transport of Bi nanowires and can be utilized with thermoelectricity.

A novel growth method for Bi nanowires is termed the on-film formation of nanowires (OFF-ON), as based on the observation of the spontaneous growth of Bi nanowires from Bi thin films without the use of conventional templates, catalysts, or starting materials. OFF-ON is a stress-induced method for the growth of Bi nanowires that has received little attention in the nanotechnology community.[16] Figure 1a schematically illustrates the OFF-ON process, showing the origin and driving force for the spontaneous growth of the Bi nanowires. A Bi thin film was initially deposited onto a thermally oxidized Si(100) substrate at a rate of 32.7 Å/s using an ultrahigh vacuum (UHV) radio frequency (rf) sputtering system. Heating the thin film to 270 °C initiates thermal stress, which originates from a mismatch of the thermal expansion between the film and the substrate. This mismatch is attributable to the large difference between thermal expansion coefficients of Bi (13.4×10^{-6}/°C) and SiO_2/Si ((0.5×10^{-6}/°C)/(2.4×10^{-6}/°C)); this difference acts as a thermodynamic driving force for spontaneous growth during the thermal annealing process. The Bi film expands while it is annealed in the temperature range 260 °C−270 °C, while the substrate restricts expansion, putting the Bi film under compressive stress. Given that the annealing temperature is close to the melting point of Bi (271.3 °C), substantial atomic diffusion takes place. Therefore, the spontaneous growth of the Bi nanowires becomes a means of releasing the compressive stress through atomic diffusion. In addition, we observed no appreciable tangential motion of the Bi film along the substrate to release the stress that can be caused by the incoherent interface between the film and substrate.

Figure 1b shows scanning electron microscopy (SEM) images of Bi nanowires grown by OFF-ON after annealing at 270 °C for 10 h. Uniform and straight Bi nanowires with high aspect ratios were found to be extruded from the surface of the as-sputtered films. Nanowires grown by OFF-ON grew to several hundred micrometers in length and down to a few tens of nanometers in diameter. High-resolution transmission electron microscopy (HR-TEM) was utilized to investigate the crystal structure of a Bi nanowire with $d = 100$ nm, as shown in Figure 1c. The nanowire was found to be uniform in diameter and to have formed a 10 nm thick Bi oxide layer on its outer surface. The electron diffraction (ED) pattern obtained in the direction perpendicular to the long axis of the nanowire was indexed to the hexagonal lattice of Bi ($a = 4.574$ Å, $c = 11.80$ Å) with the $[1\bar{1}0]$ zone axis, Figure 1c. High-resolution transmission electron microscopy (HR-TEM) studies revealed that its axis was oriented along the trigonal direction [001] and that the Bi nanowire was defect-free, high-quality single-crystalline. Very recently, we also observed various growth directions of Bi nanowires such as [110] and $[\bar{1}02]$, which leads us to conduct further studies on the relationship between orientations of grains in a film and growth direction of nanowires.

OFF-ON is similar to the formation of whiskers (or hillocks) in metal films in electrical packaging[17] due to the relaxation of stress. However, OFF-ON is wholly different from the hillock formation for several reasons. Many studies[18,19] concerning whiskers (or hillocks) have been conducted in an effort to find methods to prevent hillock formation, which is a detrimental to the reliability of electronic packaging systems. OFF-ON can be exploited to grow perfect single-crystalline Bi nanowires for fundamental transport studies and possibly for use in thermoelectric applications. Furthermore, the diameter and length of the Bi nanowires are tunable via OFF-ON, as described below.

Figure 2 presents SEM images of Bi nanowires. Here, the diameters were (a) $d = 1.2$ μm, (b) 450 nm, (c) 140 nm, and (d) 98 nm, and the wires were grown on Bi films with different thickness values ($t = 3.3$ μm, 830 nm, 83 nm, and 55 nm, respectively) after annealing at 270 °C for 10 h. It was found that the thicknesses of 3.3 μm, 830 nm, 83 nm, and 55 nm in the as-grown films correspond to the average grain sizes of 600, 260, 125, and 100 nm. Moreover, as the mean grain size of the as-grown film decreased, the diameter of the nanowires grown on the films decreased after annealing. Consequently, the average grain sizes of 600, 260, 125, and 100 nm in the as-grown films corresponded to Bi

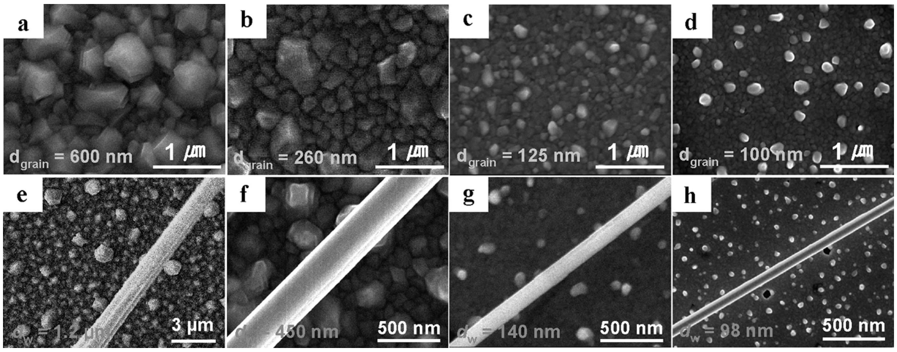

Figure 2. Control of the diameter of the Bi nanowires. SEM images of Bi thin films with thicknesses of 3.3 μm, 830 nm, 83 nm, and 55 nm with average grain sizes of (a) 600 nm, (b) 260 nm, (c) 125 nm, and (d) 100 nm, respectively. The average grain sizes of (a) 600 nm, (b) 260 nm, (c) 125 nm, and (d) 100 nm in the as-grown films correspond to nanowire diameters of (e) $d = 1.2$ μm, (f) 450 nm, (g) 140 nm, and (h) 98 nm, respectively.

wire diameters of 1.2 μm, 450 nm, 140 nm, and 98 nm, as shown in Figure 2, panels e−h.

We found that the correlations between the thickness and mean grain size of the Bi films and the diameter of the Bi nanowires show the diameters of the Bi nanowires decrease in proportion to the thickness and grain size of the films.[20] These results demonstrate that the diameters of Bi nanowires depend on the mean grain sizes in the as-grown films, which are determined by the thickness of the films. It is clearly observed that a Bi nanowire grows at the grain of a Bi thin film, indicating that the Bi nanowire grew from the bottom of the wire, where it was attached to the film, fed by Bi via grain boundary diffusion from the supporting Bi thin film.[21] This result indicates that the Bi nanowire diameter is strongly coupled to the grain size of a film. The discovery that the diameter of Bi nanowires grown by OFF-ON can be tuned by controlling the sputtering time is important, since it is one of the simplest known means of growing Bi nanowires. In the present work, Bi nanowires with a diameter as small as 30 nm were obtained.[20] Further discussion on the mechanism of OFF-ON will be presented in subsequent work.[21]

A nanodevice with four probes based on an individual Bi nanowire is of great significance for exploring unusual fundamental transport properties such as the classical[9] and quantum size effects.[5,6] In order to realize a Bi nanowire device, two crucial demands should be met. First, the Bi nanowire must be single crystalline; this demand is satisfied by OFF-ON in this work. Second, reliable electrical Ohmic contacts to the nanowires are essential. A plasma etching technique was employed to sputter away a Bi oxide layer that forms on the outer surface of the nanowire, and Au electrodes were deposited by sputtering, Figure 3a. Both procedures were done in situ without breaking a vacuum in order to prevent further formation of the oxide layer. A combination of electron-beam lithography and a lift-off process was utilized to fabricate an individual 400 nm diameter nanowire device, as seen in Figure 3b.

Panels c and d of Figure 3 show the variation of the transverse and longitudinal magnetoresistance (MR) of the 400 nm diameter Bi nanowire. MR is defined as

$$\Delta R(H)/R(0) = [\mathrm{R}(H) - R(0)]/R(0) \qquad (1)$$

where $R(0)$ is the zero-field resistance and $R(H)$ is the resistance at a given magnetic field H. MR results from the bending of the trajectories of the carriers, for which the fundamental quantity is $w_c\tau$, where $w_c = eH/m^*$ is the cyclotron frequency and m^* is the effective mass. In this work, the largest transverse MR (2496%) was observed at 110 K, as shown in Figure 3c. Such a large MR value originates from a long relaxation time (τ), indicating a long mean free path (l). The results of this study support the view that Bi nanowires grown using OFF-ON are the higher quality single-crystalline nanowire compared to Bi nanowires grown by other methods.[8,9]

The single-crystalline nature of the Bi nanowires is clear from the observation of the wire boundary scattering effect that arises from the reduction of the cyclotron radius, $r_c = (mv^2)/e(vH)$, which is caused by a high magnetic field parallel to the axes of cyclotron resonance.[5,6] In Figure 3d, the MR ratio was found to increase as H increased when $H < 9.6$ kOe at 2 K. However, it gradually flattened out, reaching a maximum MR at $H_m = 9.6$ kOe. It finally decreased as H increased when $H > 9.6$ kOe. In this work, the lowest H_m (9.6 kOe) and largest longitudinal MR (−38%) values at 2 K were observed as compared to any other values reported in the literature, indicating the strongest known wire boundary scattering effect in Bi nanowires reported in the literature.[5] Again, the results of this study demonstrate that the Bi nanowire has the longest known mean free path due to its high-quality, single-crystalline properties. In effect, the largest longitudinal MR of 22.7% at 5 T found in this study, which is an order of 2 increase compared to 90 nm single-crystalline Bi nanowires that show a longitudinal MR of 0.05% at 5 T,[5] suggest a very long mean free path for Bi nanowires grown by OFF-ON. For instance, a 120 nm thick Bi nanowire was found to exhibit a mean free path of 1.35 μm (vide infra). Interestingly, the Bi nanowire was found to exhibit pronounced Shubnikov−de Haas (SdH) oscillations with magnetic fields in both the transverse and longitudinal directions.[21] This provides further direct evidence of a high-quality single-crystalline Bi nanowire grown using OFF-ON. The SdH oscillations in both geometries will be described in detail in the literature.

The dependence of the conductance G on the gate voltage V_g was found in a field effect transistor (FET) based on an individual single-crystalline Bi nanowire with $d = 120$ nm, Figure 4. Three regions exist: (I) G decreases as V_g increases

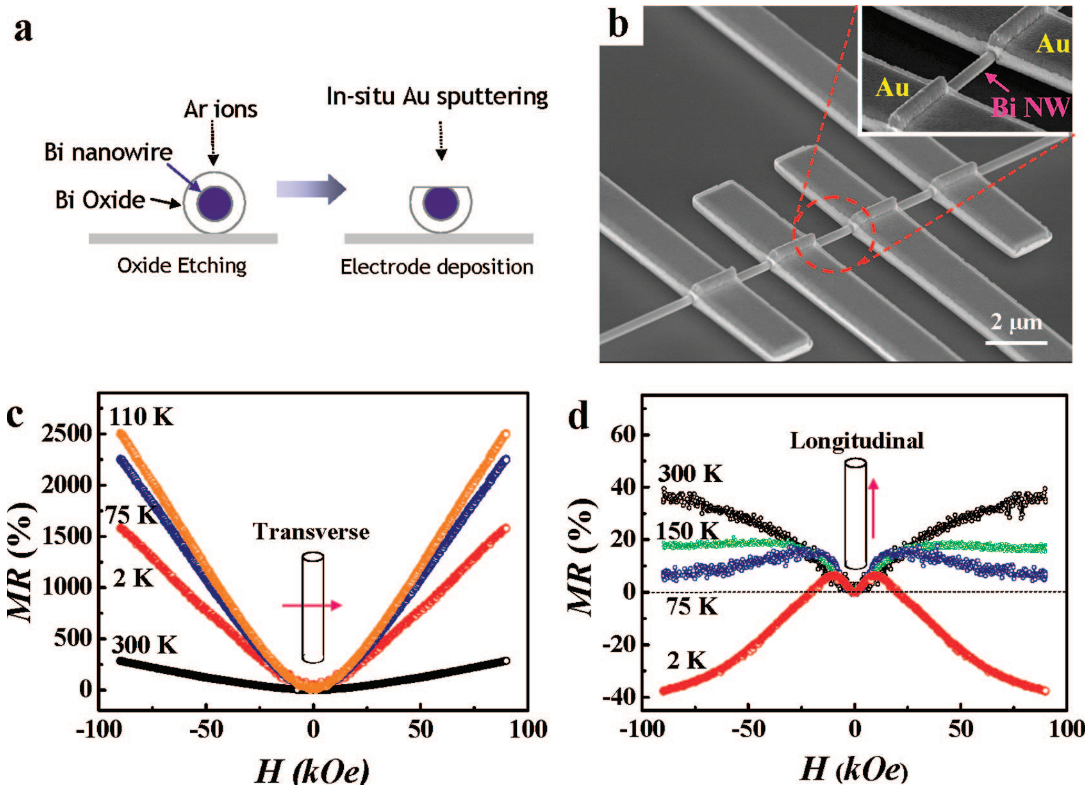

Figure 3. Device based on an individual Bi nanowire and its magnetotransprot properties. (a) Schematic diagrams of the plasma etching technique used to make electrical Ohmic contacts to an individual Bi nanowire. (b) A SEM image of a four-terminal device based on an individual Bi nanowire: The inset shows the Au contacts where the voltage difference was recorded. Also shown is the OMR of a 400 nm diameter Bi nanowire for the (c) transverse and (d) longitudinal geometries.

when $V_g < -35$ V, (II) G increases as V_g increases when $-35\ \text{V} < V_g < +35$ V, and (III) G rapidly increases as V_g increases when $+35\ \text{V} < V_g$, implying that the dependence of G on V_g is symmetric. This is attributable to the inherent characteristics of Bi, in which the number of electrons is equal to that of the holes without an applied electric field.[22] The slope dG/dV_g is related to the mobility of the nanowire by the relationship

$$\frac{dG}{dV_g} = \frac{1}{V_{sd}}\left(\frac{dI}{dV_g}\right) \propto \mu \qquad (2)$$

where V_{sd} is the voltage difference between the source and drain in the FET structure. The values of dG/dV_g for regions I, II, and III correspond to −0.0038, 0.0030, and 0.0065 S/V, respectively, indicating that the electrons have higher mobility than the holes. In other words, the pure holes (region I) and electrons (region III) are the only carriers, whereas both holes and electrons participate in the charge transport in region II. This is illustrated by the electronic structures and the Fermi levels in the inset of Figure 4. This can be quantitatively justified: 0.0030 $(\text{slope}_{\text{regionII}}) \approx -0.0038$ $(\text{slope}_{\text{regionI}}) + 0.0065$ $(\text{slope}_{\text{regionIII}})$.

The charge density n_d by the electric field effect (EFE) is characterized by a linear increase with the gate voltage, defined as $n_d = (\varepsilon_0\varepsilon_{SiO_2}/eh_{SiO_2}d_{Bi})V_g$, where ε_0 and ε_{SiO_2} are the permittivities of the free space and SiO_2, respectively, h_{SiO_2} is the thickness of the SiO_2 layer (300 nm), and d_{Bi} is the diameter of the Bi nanowire (120 nm). To ensure that the nanowire is under EFE conditions, the screening length of a Bi nanowire can be estimated as 40 nm.[20] Quantitatively, the carrier concentration of the Bi nanowire, which has an equal number of holes and electrons at $V_g = 0$, can be written as

$$n_e = n_h = n_d(V_g = \pm 35\ \text{V}) \qquad (3)$$

which is derived from the fact that holes are substituted with electrons as V_g increases. Using eq 3, the hole and electron concentrations of the present single-crystalline Bi nanowire were found to be $6.3 \times 10^{17}\ \text{cm}^{-3}$, which is lower than that in the bulk, at $2.5 \times 10^{18}\ \text{cm}^{-3}$ at 300 K. A lower carrier

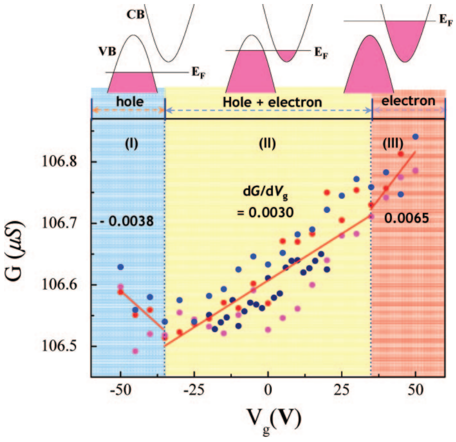

Figure 4. Conductance G vs back gate voltage V_g for a 120 nm diameter individual Bi nanowire at 300 K. The conductance (G) values were measured as an external gate voltage was swept from −50 V to +50 V and were collected separately during several different scans to avoid possible drift effects.

concentration of the Bi nanowire, relative to the bulk, is attributable to the lower Fermi wave vector k_F caused by the small diameter of the nanowire.[20]

The measured value for n_e reported herein, the electron mobility, and the mean free path at 300 K were found to be $\mu_e = (en\rho)^{-1} \approx 76900$ cm^2/(V s) with $\rho \approx 1.29 \times 10^{-4}$ Ω·cm and $l_e = (h/2e)(3n_e/\pi)^{1/3}\mu_e \approx 1.35$ μm. The results show that the mobility and mean free path in the single-crystalline Bi nanowire with $d = 120$ nm are 3 and 2 orders of magnitude, respectively, larger than that in a polycrystalline Bi nanowire.[23] Indeed, the mobility in the Bi nanowire is comparable to semiconducting carbon nanotubes (79000 cm^2/(V s) at 300 K).[24]

In summary, four-terminal and three-terminal devices based on individual Bi nanowires grown by OFF-ON showing unusual transport properties were investigated. One can use OFF-ON to grow perfect single-crystalline Bi nanowires with diameters that can be reduced to 30 nm. This technique is neither template- nor catalyst-assisted and is much simpler than existing methods. The transverse MR (2496% at 110 K) and longitudinal MR ratios (−38% at 2 K) for a 400 nm diameter Bi nanowire were found to be the largest known values reported in the literature, suggesting that the Bi nanowires grown using OFF-ON were perfectly single crystalline. These experiments demonstrate a field effect transistor based on a 120 nm diameter Bi nanowire exhibiting extraordinary electron mobility (76900 cm^2/(V s)) and a long mean free path (1.35 μm). Very recently, OFF-ON was used to grow high-quality single-crystalline Bi_2Te_3 nanowires on Bi and Te cosputtered thin films after annealing.[25] Importantly, the results of this study suggest the possibility of designing single-crystalline Bi and Bi_2Te_3 nanowires that can be used in thermoelectric devices with high thermoelectric figure-of-merit (*ZT*) values.

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Supporting Information Available: Description of growth of the single-crystalline Bi nanowires via OFF-ON and transport properties of individual single-crystalline Bi nanowires. This material is available free of charge via the Internet at http://pubs.acs.org.

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