Spin-filter effect at the interface of magnetic/non-magnetic homojunctions in Li doped ZnO nanostructures

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After more than a decade of extensive research on the magnetic order triggered by lattice defects in a wide range of nominally non-magnetic materials, we report its application in a spintronic device. This device is based on a spin-filter phenomenon we discovered at the interfaces between defect-induced magnetic and non-magnetic regions, produced at the surface of a Li doped ZnO microwire by low-energy proton implantation. Positive magnetoresistance is observed at 300 K and scales with the number of interfaces introduced along the wire.
INTRODUCTION

Since the discovery of the giant magnetoresistance (GMR) effect in magnetic/non-magnetic metal heterostructures nearly 30 years ago [1,2], researchers have focused on finding new magnetoresistive effects and improving their strength for applications in spintronics and sensor devices such as high-performance read heads, nonvolatile memories, and other state-of-the-art storage devices (see Ref. [3] and references therein).

In recent years, the advance in magnetic semiconductor material research has opened new ways to achieve GMR in semiconductor devices [4–6]. Since the prediction of ferromagnetism at room temperature in ZnO doped with magnetic transitional metal ions [7], it has become a promising candidate material for semiconductor oxide based spintronic applications, due to its large availability and low production cost.

Different approaches have been followed to induce GMR in ZnO based material systems, for example transition metal (e.g. Co or Mn) doping of ZnO thin films [8–10], magnetic/non-magnetic semiconductor oxide thin film heterostructures [11] and magnetic tunnel junctions between a Co and a ZnO:Co film [12]. All approaches showed high magnetoresistance (MR), but only at low temperatures (below 50 K).

We should note that after the first reports on magnetic order at room temperature appeared, doubts were quickly raised on the homogeneity of the ZnO-based samples as well as on the origin of the magnetism. In the last years, however, the possibility to produce magnetic order by introducing ~ 5 at% lattice defects (like Zn vacancies) in ZnO has been successfully shown in different laboratories [13–16] and by different characterization methods including X-ray magnetic circular dichroism (XMCD) [17]. This kind of magnetism, called defect induced magnetism (DIM) has been discovered in several other oxide and non-oxide materials [13–16]. We also note that despite the large amount of research on ZnO, no simple and feasible spintronic applications have been found yet at room temperature.

In this work we present the observation of GMR in a ZnO based device even at room temperature. The device consists of a Li-doped ZnO microwire, in which DIM is induced by low energy proton implantation [17]. Alternating magnetic and non-magnetic regions are produced in a ~10 nm thick surface region of the wire, effectively building magnetic/non-magnetic homojunctions with sharp interfaces. We identify a spin filter mechanism at the junction interfaces as the source of the magnetoresistance. In contrast to other known magnetoresistive effects, the effect we describe in this paper scales with the number of interfaces.

RESULTS AND DISCUSSION

Fig. 1 (a) depicts the process we used to create magnetic strips at the surface of ZnO:Li microwires. The magnetic regions were implanted with a proton dose of 10^{17} cm^{-2}, while the non-magnetic regions were implanted with a dose of 10^{15} cm^{-2}. For a detailed description of the process, we refer the reader to the methods section. Photoluminescence scans along the implanted wires clearly show the different regions (see Supplementary Figure 1).

Fig. 2(a) shows the field dependence of the MR at selected temperatures between 75 K and 300 K and at an applied bias voltage of 1 V for the sample S1. The MR was calculated as follows:

\[
\text{MR}(B, T) = \frac{R(B, T) - R(B = 0, T)}{R(B = 0, T)} \times 100, \tag{1}
\]

where \( R(B = 0, T) \) is the resistance at zero field and \( R(B, T) \) the resistance at a finite magnetic field \( B \).

Fig. 2(b) shows the MR as a function of temperature for temperatures between 5 and 300 K when a bias voltage of 1 V and a magnetic field of 7 T are applied. The sample shows MR up to 300% at 30 K and 7% at room temperature. Above 50 K, the MR decreases exponentially. Below 30 K, the MR drops within ~10 K to zero. This behaviour is completely unusual. In general, the MR increases while decreasing temperature. We will show in our simulations that this behaviour can be explained by a ferromagnetic exchange interaction.

As previously reported, the formation of Zn vacancies (V_{Zn}) during proton implantation is responsible for the magnetic order in ZnO:Li microwires [17]. The formation of such magnetic order is related to the density of V_{Zn} and their stabilization by Li dopants. A certain concentration of V_{Zn} is necessary to induce magnetic order. Our previous experiments show that a dose of 10^{17} cm^{-2} implanted protons induces a magnetic order, while doses of 10^{15} – 10^{16} cm^{-2} are not sufficient. Thus, after implanting a dose of 10^{17} cm^{-2} protons in the small regions along the wire, magnetic/non-magnetic (FM/NM) interfaces are produced along the wire’s main axis. Due to the short penetration depth of the H^+, the interfaces between magnetic and non-magnetic regions are sharply defined in space. In addition, H^+ implantation introduces shallow donor states [25], which once activated provide free electrons and decrease the resistivity of the wires in the implanted region and within the first 10 nm from the surface.
FIG. 1. **Sample preparation and characterisation.** (a) Schematic of the $H^+$ implantation process to produce defect-induced magnetic strips on the surface of a ZnO:Li microwire. The PMMA mask is produced by electron beam lithography to achieve structure sizes down to 100 nm. (b) SEM picture of a wire with a mask covering 100 nm long regions for the creation of alternating magnetic/non-magnetic stripes by proton implantation. (c) Sketch describing the spin filtering effect at the interfaces between magnetically ordered (FM) and non-magnetic (NM) regions of the wire. In the FM regions, the current is spin polarized due to the spin subband splitting. Spin-down electrons accumulate at the FM/NM interface, while spin-up electrons can more easily pass the potential barrier. In the NM regions, spin-up and spin-down electrons become indiscernible and the current becomes unpolarized. At the NM/FM interface, the probability for spin-down electrons to be extracted from the NM region is higher than that of spin-up electrons. Thus, the current density decreases along the wire.

Fig. 3 shows the IV characteristics of sample $S1$. The IV curves show a clear diode rectification behavior. When the wire is uniformly implanted with a dose of $10^{17}$ cm$^{-2}$ of $H^+$ along the whole wire, the IV curves become linear (see inset of Fig. 3) at all temperatures. This is an indication that the contacts are Ohmic. Thus, the non-linearity observed is related to a potential barrier produced between the highly donor doped ($n^+$) magnetic and the less donor doped ($n$) non-magnetic regions, due to the very different free electron concentrations. Note that the IV curves are asymmetric with respect to the current polarity. In the present configuration, one would expect a symmetric behaviour. This could be explained by an asymmetric termination of the alternating chain of magnetic/non-magnetic regions at the metal contacts.
As the IV characteristics of the sample are highly non-linear, we measured the MR at different applied bias voltages (see supplementary figure 2). Above 30 K, the MR is highest at small bias voltage and decreases with increasing bias voltage. Below 30 K, the behaviour is reversed. By applying a bias voltage, the potential barriers at the interfaces between the FM and NM regions are shifted, indicating that these interfaces play a role in the MR effect.

To further prove the effect of the interfaces, we increased the carrier concentration in the non-magnetic region of sample S1. For this purpose we implanted H⁺ in the whole wire. Note that the magnetic regions are already saturated with H⁺ and Vₓ̂Zn (from the previous implantation step) so that the additional implantation process does
FIG. 3. **IV characteristics** of the wire S1 prepared with 37 magnetic stripes, measured without applied magnetic field in a temperature range between 30 K and 300 K. The IV curves are non-linear and asymmetric due to the n/n+ junctions formed by the weakly doped NM regions and the highly doped FM regions. The inset compares the IV characteristic at 30 K of the wire S1 with 37 magnetic strips, before (blue) and after (red) a uniform implantation of $10^{17}$ cm$^{-2}$ H$^+$ along the whole wire. This last IV curve is linear, indicating that the nonlinearity indeed comes from the interfaces and not from the contacts.

not change the conductivity of these regions significantly. Fig. 4 (a) shows the magnetic field dependence of the MR for different implantation doses in the NM regions. The curves (1), (2), (3), (4) were measured after implanting doses of $2.5 \times 10^{15}$ cm$^{-2}$, $3.7 \times 10^{15}$ cm$^{-2}$, $7.5 \times 10^{15}$ cm$^{-2}$ and $3.7 \times 10^{16}$ cm$^{-2}$ respectively. As one would expect, the MR decreases with increasing implantation duration in the NM regions, as the carrier concentration levels up with that in the FM regions and the potential barriers shrink. After the implantation of $3.7 \times 10^{16}$ cm$^{-2}$ of protons in the NM regions, the MR becomes negative (4) and has a maximum value of $-1\%$ at 7 T. The potential barriers disappeared and with them the GMR effect. Note that the observed negative MR is only due to the FM regions, as already shown in a previous work [21].

This suggests that there are two different contributions, namely the negative MR contribution from the magnetic regions and a giant positive MR due to interfaces, in agreement with the variation of the MR with the applied bias voltage. In such a case a decrease of the number of interfaces along the wire would produce a reduction of the total MR. To demonstrate it, we performed similar experiments on Li doped ZnO wires on which different number of strips were produced. Fig. 4 (b) shows the MR as a function of an applied magnetic field at a constant temperature of 30 K for different samples having 36, 21, 17, 5, 1 and 0 magnetic strips. We can see that the MR increases with the number of interfaces. For 1 strip, the negative MR is observed, since the effect of a single interface is not strong enough to compensate the contribution of the magnetic region.

The presented measurements show several peculiar features of the effect at hand, namely: 1) The positive MR is linear in applied magnetic field. 2) The positive MR scales with the number of NM/FM interfaces. 3) The MR drops to zero at temperatures below 30 K. These observations suggest that the measured MR originates from a spin filter effect at the interfaces between the NM and FM regions, as we will show.

Fig. 4 (c) shows a sketch of the model we use to explain the observed effect. We first recognize that: 1) A potential barrier exists between the magnetic and non-magnetic regions due to the different carrier concentrations produced by the H$^+$ implantation. 2) In the magnetic (FM) strips, the spin degeneracy is lifted and the conduction band is split due to the exchange interaction ($E_{ex}$) and a large Zeeman splitting ($E_Z$). All this induces a spin polarization of the conduction electrons.

At each interface between the magnetic and non-magnetic regions, a potential barrier is built due to the different
FIG. 4. Interface effect. (a) Magnetoresistance measured at 30 K with a bias of 1 V as a function of applied magnetic field after an H$^+$ implantation dose of 2.5 × 10$^{15}$ cm$^{-2}$ (1), 3.7 × 10$^{15}$ cm$^{-2}$ (2), 7.5 × 10$^{15}$ cm$^{-2}$ (3) and 3.7 × 10$^{16}$ cm$^{-2}$ (4) in the non-magnetic regions of sample S1. With increasing dose, the potential barrier height between the magnetic and non-magnetic regions becomes smaller and finally disappears after implanting a dose of 3.7 × 10$^{16}$ cm$^{-2}$ (4), where the only remaining contribution to the MR comes from the magnetic regions. (b) MR at an applied field of 1 T, 2 T, 5 T and 7 T as a function of the number of magnetic stripes. The results indicate that the effect scales with the number of interfaces.
doping profiles, which results in a difference in chemical potential at both sides of the interface. The spins can cross the potential barrier by thermionic emission and undergo spin scattering processes in the non-magnetic part. As the spin diffusion length in ZnO is smaller than the length of the non-magnetic parts we created, the current remains less spin polarized when reaching the next NM/FM interface.

At the NM/FM interface, a spin filtering process takes place, as one spin orientation is energetically more favorable to cross the interface, while the electrons with the opposite spin orientation get accumulated at the interface due to the spin band splitting in the FM strip. By adding more interfaces in series, the effect can potentially be scaled indefinitely.

We can model the spin transport through these interfaces using a very simple approximation of the barrier, as sketched in Fig. 5(a). At thermal equilibrium, the conduction band (CB) is deformed in the magnetic region (FM) due to the different doping concentrations $n_0$ and $N_0$ in the NM and FM regions respectively and is shifted by $V_{bi} = \frac{k_B T}{e} \log \left( \frac{N_0}{n_0} \right)$ compared to the NM region. In the FM region, the spin-subbands are split by the energy $2E_S = 2(E_{ex} + g\mu_B B)$, which has a magnetic field dependent contribution from Zeeman interaction and a constant contribution $E_{ex}$, the exchange energy. At the FM/NM interface, a space-charge region of width $w$ is built out, in which spin-up and spin-down electrons have different transmission probabilities. The spin dependent current density through the interface can be expressed as

$$j_{\uparrow\downarrow} \propto \mu_{\uparrow\downarrow} T^2 \exp \left[ -\frac{e}{k_B T} \left( V_{bi} - V_n \pm \frac{E_S}{e} \right) \left[ \exp \left( \frac{eV_n}{k_B T} \right) - 1 \right] \right]$$

where $\mu_{\uparrow\downarrow}$ is the mobility of spin-up and spin-down electrons respectively, $V_n$ the depth of the donor states taken from the conduction band edge, $V_n$ the applied voltage, $e$ the electron charge, $k_B$ the Boltzmann constant and $T$ the temperature. The total charge current through the interface is then the sum of the two spin channel currents $j = j_{\uparrow} + j_{\downarrow}$. From this, we calculate the magnetoresistance:

$$MR(B, T) = \frac{\tau_B(B, T) \left[ \mu_{\uparrow} + \mu_{\downarrow} \tau_{ex}^4 \right]}{(1 + \tau_{ex}(T)^2) \left[ \mu_{\downarrow} + \mu_{\uparrow} \tau_{ex}(B, T) \tau_{ex}(T)^4 \right]} - 1$$

with $\tau_B = \exp \left( \frac{qB}{k_B T} \right)$ and $\tau_{ex} = \exp \left( \frac{E_{ex}}{k_B T} \right)$ the Boltzmann factors of the Zeeman and Exchange splitting respectively.

In this simple model, spin dependent scattering processes are encoded in the electron mobility $\mu_{\uparrow\downarrow}$, that is assumed to be different for the two spin orientations.

Fig. 5(b) shows the normalized MR as a function of temperature as calculated using Eq. (3), where we set the applied magnetic field to 7 T, the ratio of mobilities of spin-up and spin-down electrons to $\mu_{\uparrow}/\mu_{\downarrow} = 100$ and the exchange energy $E_{ex}$ ranging between 0 meV and 20 meV. By comparing the calculations to our measurements, we can estimate the exchange energy of the magnetic regions of our sample to $E_{ex} \sim 1$ meV. Taking into account the Curie temperature reported for defect induced magnetic ZnO (between 450 K and 700 K), this value for $E_{ex}$ fits very well with those reported for several transition metals, e.g. Fe ($T_C = 1041$ K, $E_{ex} = 7.6$ meV) or Co ($T_C = 1394$ K, $E_{ex} = 32.3$ meV).

These results suggest that the MR at room temperature could be strongly improved by using materials with stronger magnetic coupling $E_{ex}$ or by tweaking the concentration of defects inducing the magnetic coupling. Also, the exponential decay of the MR with temperature could be addressed by acceptor doping of the non-magnetic regions and thereby creating magnetic/non-magnetic p/n junctions. Although acceptor doping is challenging in ZnO, other semiconductor materials, e.g. GaN, exhibit defect induced magnetism [26] and could be used to this end.

**CONCLUSION**

More than ten years after the realization of defect induced magnetism, we present in this work its practical application in a spintronic device, which makes use of a spin filtering effect at the interface between defect-induced magnetic and non-magnetic regions at the surface of a ZnO microwire. This device exhibits giant magnetoresistance, which scales with the number of interfaces. The experimental method described in this work is simple and economical and can be applied to a large number of materials showing defect-induced magnetism.
FIG. 5. **Model of the spin filter effect.** (a) Sketch of the conduction band (CB) structure along the magnetic (FM) / non-magnetic (NM) junction. $V_{bi}$ is the built-in potential step, $E_F$ the Fermi level and $E_s$ the spin-band splitting energy. (b) The squares show the magnetoresistance at an applied magnetic field of 7 T and a bias voltage of 1 V measured as a function of temperature and normalized to its maximum value. The lines show the results of the numerical calculation of the temperature dependence of the MR due to a spin polarization (Eq. (3)) for values of the exchange energy $E_{ex}$ between 0 meV and 20 meV.

**METHODS**

Li-doped ZnO microwires of 100 µm to 300 µm length and 0.5 µm to 2 µm diameter were prepared by a carbothermal process [17, 18]. The Li concentration was chosen following reports in Ref. [19]. Single wires were selected using an optical microscope and fixed on a Si/Si$_3$N$_4$ substrate. A dose of $10^{17} \text{cm}^{-2}$ of H$^+$ was implanted in the extremeties
of the wires where gold contacts were made by e-beam lithography and DC sputtering. The $\text{H}^+$ implantation was realized using a remote hydrogen DC-plasma chamber \cite{14} with an implantation energy of 300 eV and a current of 60 $\mu$A. Assuming a displacement energy of 18.5 eV (Zn) and 40 eV (O), “Stopping and range of ions in matter” (SRIM) \cite{20} simulations indicate that $\text{H}^+$ is implanted in the top 10 nm of the wire surface and that Zn-vacancies ($\text{V}_{\text{Zn}}$) and O-vacancies ($\text{V}_{\text{O}}$) are created during the implantation. Previous studies indicate that after implanting a dose of $10^{17}$ cm$^{-2}$, the resistivity of the Li-doped ZnO material decreases drastically in the 10 nm subsurface region \cite{21}, allowing us the preparation of ohmic contacts for transport measurements.

In a second step, a PMMA mask consisting of small exposed regions was created on top of the wires between the contacts using e-beam lithography (see fig. 1(a)). A dose of $10^{17}$ cm$^{-2}$ of $\text{H}^+$ was then implanted in the exposed regions. Due to the creation of $\text{V}_{\text{Zn}}$, the exposed regions become magnetically ordered \cite{17}.

In a final step, a dose of $10^{15}$ cm$^{-2}$ of $\text{H}^+$ was implanted in the whole wires. This dose is not sufficient to induce magnetic order in the regions between the magnetic strips but reduces the resistivity to enable us to measure a current.

Different wires were prepared using different numbers of strips. Also, different lengths (parallel to wire axis) of strips were tested, showing comparable results. In the following, we describe samples from a batch produced with different numbers of strips of 400 nm length and 4 $\mu$m separation. For example, sample $S1$ was prepared with 37 strips and sample $S2$ with 17 strips.

Magnetotransport measurements were performed in a He-cryostat with a maximum magnetic field of 7 T at temperatures ranging between 5 K and 300 K. Resistance was measured using a Keithley 6517a Electrometer and IV characteristics were acquired using a Keithley 220 Current Source and a Keithley 182 Voltmeter.

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Supplementary Information

PHOTOLUMINESCENCE CHARACTERISATION

FIG. 6. Photoluminescence characterisation. (a) Photoluminescence spectra of ZnO:Li wires implanted with doses of $2.5 \times 10^{15}$ cm$^{-2}$, $5.0 \times 10^{16}$ cm$^{-2}$ and $1.0 \times 10^{17}$ cm$^{-2}$ of protons respectively. The ratio between the intensities of two main emission bands increases with the implantation dose. (b) Line scan along the wire S1 showing the ratio UV/VIS of the intensities of the emissions at 385nm and 525nm. The regions with high UV/VIS ratio were implanted with a dose of $1.0 \times 10^{17}$ cm$^{-2}$ of protons, the regions with low UV/VIS ratio with a dose of $2.5 \times 10^{15}$ cm$^{-2}$. (c) Photoluminescence line scan along the wire S1, showing the emission spectra in the range between 360nm and 610nm.

To characterize the magnetic/non-magnetic regions at the surface of ZnO:Li microwires, we performed microphotoluminescence measurements. For reference, we first measured the emission spectra of a wire after an implantation of $2.5 \times 10^{15}$ cm$^{-2}$, $5.0 \times 10^{16}$ cm$^{-2}$ and $1.0 \times 10^{17}$ cm$^{-2}$ of protons respectively, as shown in Fig. 1 (a). The wire mainly emits luminescence in two spectral ranges, namely the UV emission at $\sim 380$ nm attributed to the recombination of free excitons and their phonon replica [1, 2] and several bands of green luminescence at $450 - 575$ nm attributed to intra-bandgap defect levels, such as Zn vacancies [3]. The ratio UV/VIS between the emission intensities of the two spectral ranges significantly increases with the proton implantation dose. We used this fact to characterize our samples and the magnetic/non-magnetic regions produced along the wires by proton implantation. Fig. 6 (b) shows the UV/VIS-ratio along the wire S1. The 400nm long magnetic regions implanted with a dose of $1.0 \times 10^{17}$ cm$^{-2}$ show high contrast between the UV and green emission, whereas the $\sim 2 \mu$m long non-magnetic regions implanted with a dose of $2.5 \times 10^{15}$ cm$^{-2}$ show much less contrast.

EFFECT OF APPLIED BIAS VOLTAGE

In accordance with the IV characteristics shown in the main text, increasing the applied bias voltage decreases the total resistance of the wires at all temperatures (see Fig. 7 (a)). On the magnetoresistance however, increasing
the applied bias voltage shows different effects depending on the temperature, as shown in Fig. 7 (b). Below the point of maximal MR, around 30K, increasing the bias voltage increases the MR while above 30K, the MR decreases with increasing bias voltage. This behaviour indicates that the occupation of the spin-split bands changes at that temperature. Below 30K, the thermal energy is not sufficient for electrons to reach both spin-split conduction bands. At the critical point, the lower spin-band is occupied accounting for the measured current, but the upper spin-band is not and therefore no spin filtering takes place at the magnetic/non-magnetic junctions. The application of a bias voltage facilitates the filling of the upper spin band and enhances the spin filter effect. Above 30K, both spin bands are filled and the applied bias voltage reduces the spin filter effect.

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