Imaging the fine structure of a magnetic domain wall in a Ni nano-cylinder.

Supporting Information

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Methods

Sample preparation

Magnetic nickel nanowires are prepared by electrodeposition in commercial polycarbonate (PC) nanoporous membranes (Poretics PO 30 and PO 10). In order to deposit the Nickel a thin gold layer of thickness 100 nm is deposited on one side of the membrane. Then it is immersed into an aqueous electrolytic solution. For this, 65.5g of NiSO4 ;6H20 and 15g of boric acid H3BO4 are dissolved in 500mL deionized water (pH = 6.2). The electric potential between the golden surface of the membrane (working electrode) and an Ag/AgCl reference electrode is set to -1 volts. The electrodeposition is stopped when Ni has grown on top of the PC.

To get the best resolution possible a crucial point is the dissolution and the cleaning of the membrane. Indeed, the remaining piece of the insulating polycarbonate membrane can induce artifact in the phase image due to uncompensated correction of the electrostatic phase shift component associated to charge effect in the polymer. To collect the nanowires, the membrane is dissolved several times in CH_2Cl_2. Nanowires and remaining pieces of membrane are sonicated for 2 mn and separated both by centrifugal force (1000 rpm) and using a small magnet to collect the magnetic nanowires. These steps are repeated at least 5 times. Finally, nanowires are immersed in ethanol. When deposited on the carbon grid for electron microscopy, nanowires are often clustered together (Figure S1) but we can always find isolated µm long wires for domain wall imaging.

Electron holography experiments

The phase shift in a direction r in the plane of the sample can be expressed as:

$$\phi(\boldsymbol{r}) = C_E \int V(\boldsymbol{r}, z)dz + -\frac{e}{\hbar} \iint B_\perp(\boldsymbol{r}, z).dr.dz \qquad (1)$$

where z is the incident electron beam direction, C_E is a constant dependent on the accelerating voltage of the microscope, V is the electrostatic potential of the sample, and $B_\perp$ is the component of the magnetic induction (both inside the sample and in the surrounding leakage fields) perpendicular to both $\boldsymbol{r}$ and z. The magnetic and electrostatic contributions can be separated by acquiring two holograms for which the sample has been switched upside down and subtracting their phase image before dividing by 2. Indeed, when reversing the sample, the electrostatic contribution remains constant but the magnetic contribution changes in sign.

Micromagnetic simulations

In order to be as close as possible to the magnetic structure we studied, we have implemented a script in the 3D package of the OOMMF software in order to reproduce the polycrystalline structure of the wire. For this we have divided the nanowire into Voronoi polyhedra. In each polyhedron the magnetization is set to be uniform and the crystalline anisotropy axis is randomly distributed over the different polyhedra. This allows mimicking the random orientation of the grain inside the wire. As example, we show in Figure S2 an example of the simulated grains in a nano-cylinder of 85 nm diameter. Once the remanent magnetic configuration is obtained, we calculated the magnetic induction $\mathbf{B} = \mu_0(\mathbf{H_d} + \mathbf{M})$ in all the simulated universe. The phase shift is then calculated by summing the B_x and B_y components at each cells over the entire thickness Z. Moreover we apply the same mathematical filters to the calculated phase shift as for the experiment images.

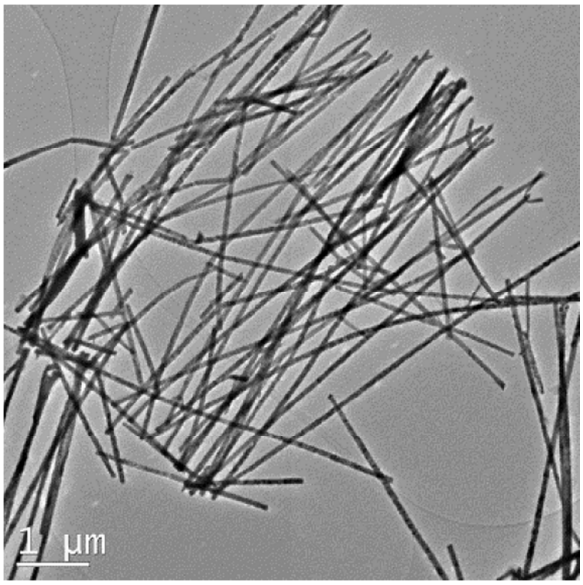

Figure S1. Example of µm long clustered Ni nanowires deposited on a TEM grid.

Figure S2. Simulation of the grains in a 85 nm diameter and 3.6 µm long Ni nanowire used to calculate the remanent magnetic state. Each part with different colors corresponds to different orientation of the crystalline anisotropy.

Magnetic phase shift at different angles

In order to avoid any ambiguity about the fine structure of a particular domain wall, it would be of particular interest if one could record electron magnetic holograms at different tilt angle around the wire axis. In the case of a TW for example, one should observe then a shift of the central part of the hologram accompanied by a variation of the symmetry of the stray field outside the wire that will confirm the model. Unfortunately the tilting of the wire around its axis is not so easy as its axis has to coincide with the sample holder tilt axis which is usually not the case. In addition performing several electron holography experiments keeping the same imaging conditions is very difficult. The main reason relies on the fact that to image

the magnetic configuration, we need to separate the magnetic and electrostatic contributions to the electron phase shift and then to remove the mean inner potential part. As explained above this is achieved subtracting two holograms obtained after the sample has been flipped upside-down and bring back to the exact reversed imaging conditions. Performing such an experiment would imply that the two electron holograms used to separate the magnetic and electrostatic contributions are taken at various tilts but in exact similar conditions when it is flipped upside-down. This is extremely difficult.

We however, have performed numbers of electron holography experiments on several nanowires and then observed the transverse wall along different observation directions in various wires which all drive to the same result. As an example, we show in the figure S3 the magnetic phase shift in a wire of 55 nm in diameter in which the spins within the transverse walls points almost in the X direction or at least with a high angle in the (Z, X) plane. As mentioned above we can see both the shift of the hologram in the X direction and additionally we clearly observe the asymmetry of the stray field outside (S3b.). When comparing with the simulation of a transverse wall pointing in the +X direction we can see the good agreement (S3c.). This observation is in perfect agreement with the configuration of the TW we report in the paper.

Let's note that in these images, the signal to noise ratio is strongly decreased due to the diffraction contrasts which appear on several grains (some are pointed in white circles in the hologram in S3a.). These induce sharp and strong variation of the electron phase shift locally (pointed in white circles in S3b.) and avoid observing clearly the magnetic field lines in all the wire. We remind here that we are studying Ni nanowire which has a small value of the magnetization $\mu_0 M_s$ (about 0.5 T) and that the magnetic phase shift is then proportional to Ms.

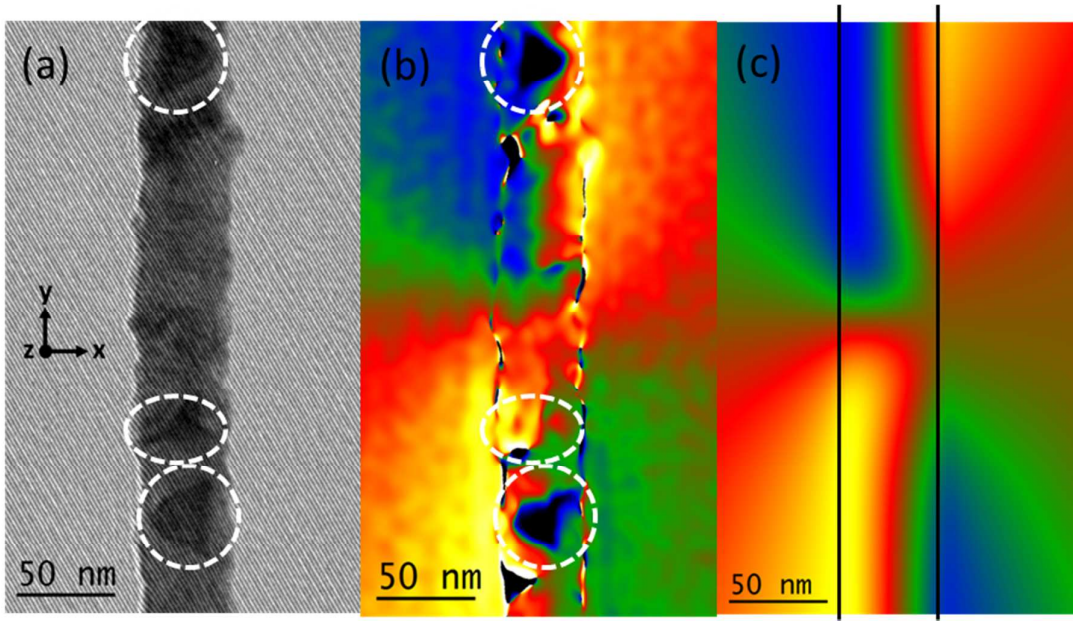

Figure S3. (a) Hologram of a 55 nm diameter Ni nanowire. The white circle shows the diffraction contrast on several grains. Experimental (b) and simulated (c) magnetic phase shift showing the tilt of the central part of the hologram and the asymmetry of the stray field as compared to the figure 3 presented in the main text.