IOSR Journal of Applied Physics (IOSR-JAP)
e-ISSN: 2278-4861.Volume 7, Issue 2 Ver. I (Mar. - Apr. 2015), PP 09-15
www.iosrjournals.org

Site-specific measurement of the giant magnetoresistance (GMR) of individual NiFe/Pt multilayers nanowires

Mohamed Elawayeb[1*], Kevin J. Briston[2], Yong Peng[3] and Beverley J. Inkson[2]
[1]*Biomedical Engineering/ College of medical Technology Misurata, Libya*
[2]*Department of Materials Science and Engineering, University of Sheffield, UK*
[3]*Key Lab for Magnetism and Magnetic Materials of the Ministry of Education, School of Physical and Technology, Lanzhou University 730000, China*

Abstract: *As devices shrink, there is a requirement to characterize the magnetic properties of individual inhomogeneous nanosized components. Here the site-specific giant magnetoresistance (GMR) of individual NiFe/Pt multilayer nanowires has been evaluated in the current perpendicular to plane (CPP) geometry using a new in-situ method directly inside a scanning electron microscope (SEM). In this method a controlled magnetic field is produced directly inside the SEM using a custom designed electromagnet. Two nanomanipulators equipped with sharp tips enable the site-specific measurement of the resistance of individual nanowires in the gap between the coils as the field is varied. The CPP-GMR measurement of ~70 nm diameter individual NiFe/Pt multilayer nanowires shows that, at 500 Oe, the nanowires have a magnetoresistance effect of up to 5.8 % dependant on the nanowire layer thicknesses and magnetic field orientation. This in-situ method of GMR measurement enables precise nanocharacterisation of magnetoresistance of nanowires as a function of local nanowire microstructure, geometry, length, and field orientation, whilst concurrently allowing the observation of any changes to the microstructure.*
Keywords: *Giant magnetoresistance (GMR), multilayer nanowires, in-situ SEM.*

I. Introduction

Nanostructured materials such as nanoparticles, nanorods and nanowires can exhibit novel and magnetoresistance properties and thus have important technological applications in nanosensors [1,2], information storage systems [3,4] and electronic devices [5]. Nanostructured magnetic materials can consist of one or more ferromagnetic elements such as Fe, Ni and Co [6] or a combination of magnetic and nonmagnetic elements such as Ni/Cu [7] and CoPt/Pt [8]. The magnetoresistance properties of the nanomaterials are affected by the nanoscale microstructure, geometry and defects of these elements.

In recent years the trend has been for smaller devices making use of inhomogeneous nanocomponents such as magnetic nanowires as opposed to, for example, thin films. It is important therefore to be able to characterize nanocomponents individually rather than in groups and with high spatial resolution along a given inhomogeneous component. Due to technology limitations, however, most magnetoresistance measurements made on multilayered nanowires over the last decade have been based on an average of multiple nanowires, contacted while still inside the template used in the manufacturing process, rather than the measurement of individual nanowires [9,10]. This is due to the difficulties of connecting probe electrodes to a single nanowire. Measurements are typically performed by making contact on either side of the template, and the numbers of contacted wires between the contact points are estimated [9,10]. Subsequently, some groups have improved these methods by reducing the number of wires embedded in a template that are contacted by using a low pore density template, for example a polymer template (< 10^9 pores cm^{-2}) [11], or using a single pore template [12]. Another improved method involves dissolving the template, dispersing the nanowires on a substrate and then contacting individual nanowires using fixed electrodes attached by e-beam lithography [13].

In this work, we report a new, flexible technique that enables site-specific GMR measurements to be made on individual nanowires with real-time imaging of the structures under test. This technique makes use of a novel in-situ electromagnet inside a scanning electron microscope (SEM), and movable electrodes attached to nanomanipulators. Site-specific GMR measurement has been applied to the characterization of magnetoresistance of individual NiFe/Pt nanowires. The in-situ method has the advantages that it allows the magnetoresistance properties of multilayer nanowires to be measured between chosen contact points, at variable lengths and at variable magnetic field. Measuring nanowires while they are in a template, on the other hand, means that the length and contact points are fixed. Also, any inhomogeneities in the wires being tested cannot be directly observed or avoided in the measurement process.

II. Experimental Procedure

The new technique for GMR measurement inside a SEM utilizes an electromagnet made from two small coils wound around 2 mm diameter pure iron (Fe) rods. Each coil is formed from 350 turns of 0.2 mm diameter insulated copper wire to create an electromagnet. The two magnetic coils, which act as a source of controlled magnetic field within the SEM chamber, are mounted in a 30 mm diameter Al base and are separated by a 2mm gap (Fig. 1(a)). The magnetic field of the coils was calibrated using a Hall probe before nanowires were positioned in the gap.

Magnetic multi-layered nanowires of different combinations of magnetic and nonmagnetic layer elements are ideal structures to study the GMR effect as the resistances of nanowires are much larger than the resistances of equivalent multi-layered thin films. Here, the GMR effect of the magnetic NiFe/Pt multilayer nanowires was measured in the current perpendicular to plane (CPP) geometry by placing the nanowires between the coils and connecting to them electrically using the tip electrodes (Fig. 1(b)). The measurements were made on individual nanowires in magnetic fields up to H = 500 Oe and at different orientations of the externally applied magnetic field with respect to wire axis ($\theta = 0°, 90°$).

Electrodeposition into anodic aluminium oxide (AAO) nanoporous templates has been utilized to produce the NiFe/Pt nanowires since nanowires with flexible geometry, chemistry and with high aspect ratio can be easily grown by this method [14,15]. Nanoporous AAO templates with ~ 70 nm diameter pores were prepared by a one-step anodization technique using 0.6 M oxalic acid solution and an applied voltage of 40 V for 180 min with continuous stirring. The multi-layered NiFe/Pt nanowires were fabricated by a three electrode electrolytic cell at room temperature using a single bath solution containing 90 g/l $NiSO_4$, 13.5 g/l $FeSO_4$, 5 g/l $PtCl_4$ and 30 g/l H_3BO_3. The electrodeposition process of the multilayer nanowires was carried out at a constant voltage of −1.4 V for NiFe layers and −0.4 V for Pt layers. The fabrication techniques of the AAO templates and NiFe/Pt nanowires are described in a previous paper [16].

The morphology and structure of individual NiFe/Pt nanowires were characterized at the nanoscale using SEM (JEOL JSM 6500F, Japan) and transmission electron microscopy (TEM) (JEOL JEM-2010F, Japan). The magnetoresistance of individual NiFe/Pt multilayer nanowires was measured in-situ in a SEM using the in-situ electromagnet and two Kleindiek MM3A nanomanipulators connected to a Keithley 6487 picoammeter. Two sharp conductive nichrome probe tips were used in the nanomanipulators as electrodes and were prepared by electrochemical etching in a solution of 1M NaCl [17].

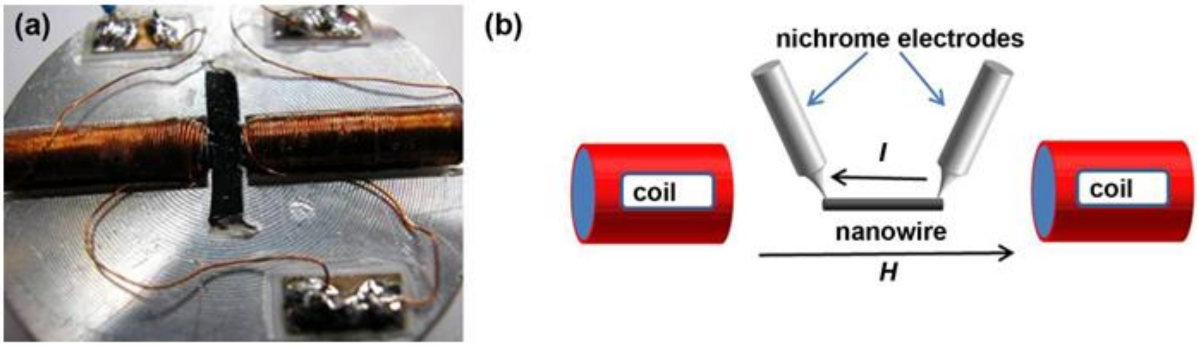


FIG. 1. (a) Image of the SEM magnetoresistance measurement system. (b) Schematic showing the setup for measuring the GMR of individual nanowires inside a SEM.

III. Results And Discussion

3.1 Nanowire structure and resistance measurement

The structures of NiFe/Pt multilayer nanowires have been analysed at the nanoscale using SEM and TEM [24]. Figure 2 shows a typical TEM image of the NiFe/Pt multilayer nanowires used in this work after having been completely dissolved out of the AAO template. The NiFe/Pt nanowires have uniform diameters of about 70 nm and an alternating NiFe and Pt layer structure. Group A nanowires were fabricated with layer thicknesses of ~35 nm NiFe/~8 nm Pt while Group B were made with ~5 nm NiFe/~15 nm Pt. The NiFe and Pt layers are polycrystalline with fcc lattice structure and the average grain size of both layers is 3–10 nm.

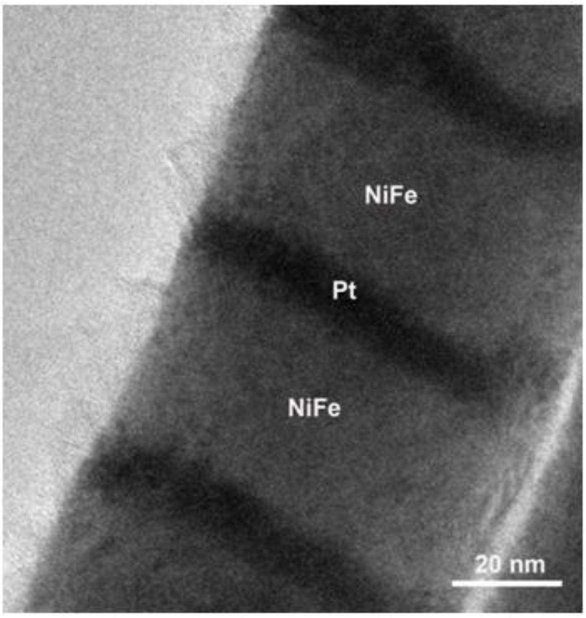


FIG. 2. Typical TEM image of an individual NiFe/Pt multilayer nanowire with alternating NiFe (grey) and Pt (dark) layers.

The site-specific electrical resistance of individual NiFe/Pt nanowires was measured directly using two manoeuvrable nichrome tip electrodes attached to in-situ SEM nanomanipulators [17,18]. The measured line resistance (R_L) of a typical circuit incorporating two nichrome electrodes connected directly to each other was ~133 Ω (Fig. 3(a)), which includes the resistances of the nichrome tips and wires. The probe electrodes attached to the nanomanipulators can move freely inside the SEM with <10nm resolution to create conductive contacts at chosen positions along an individual NiFe/Pt nanowire. Using this set up, the total electrical resistance (R_T) of measured circuits with no applied magnetic field could be determined. For example, a circuit incorporating a ~5 µm length of an individual NiFe(35 nm)/Pt(8 nm) multilayer nanowire was 2173 Ω (Fig. 3(b)). The current–voltage (I–V) curves of R_L and R_T circuits were linear (Fig. 3(c)).

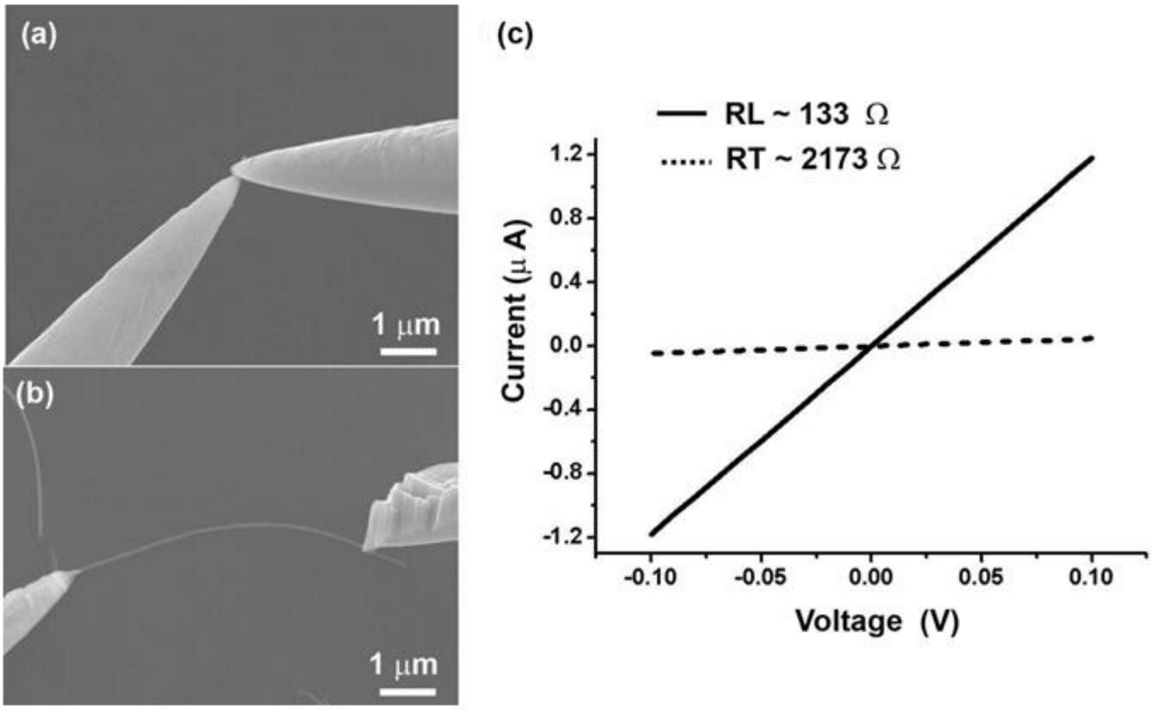


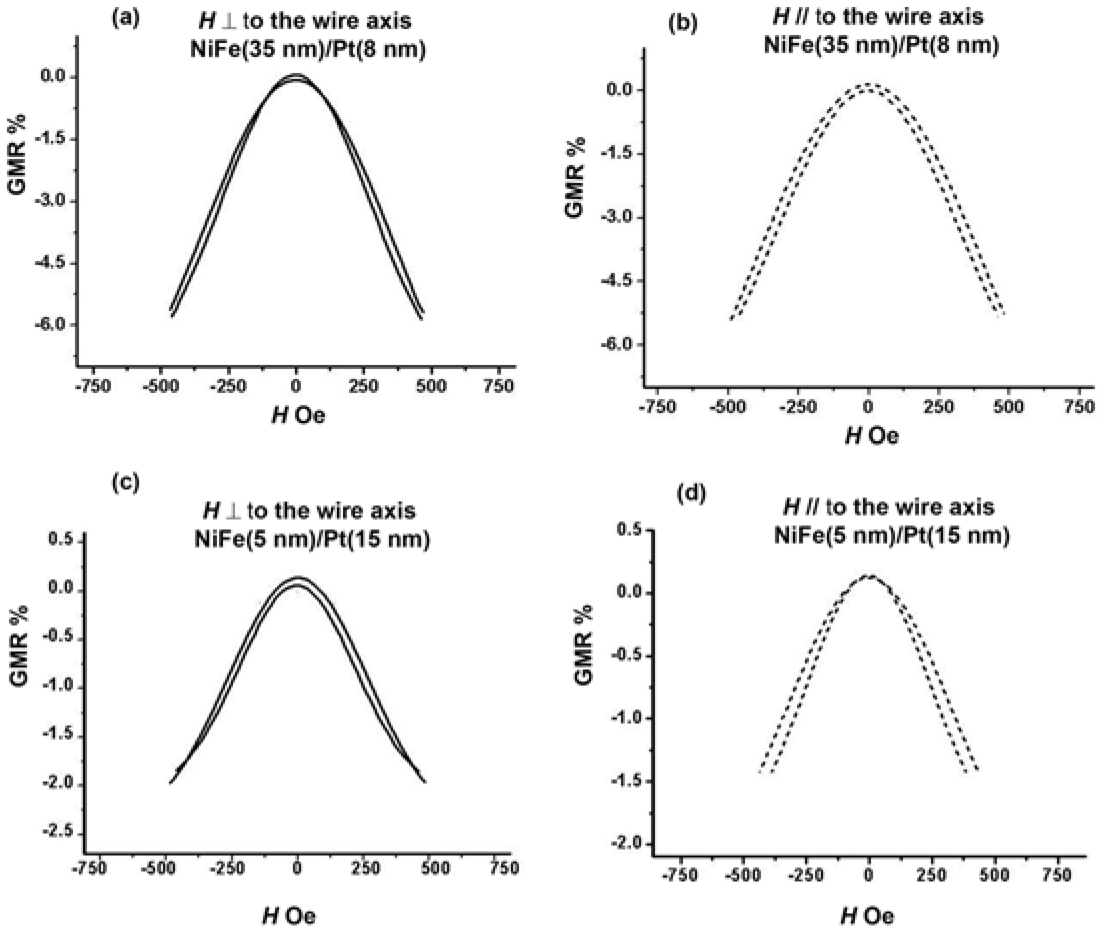
FIG. 3. Typical SEM images of the nanoscale electrical connection of (a) nichrome probe tips and (b) probe tips to an individual NiFe/Pt nanowire. (c) I–V behaviour of a typical tip–tip circuit (solid black line) and tips–NiFe/Pt nanowire circuit (dotted line).

1.2 Magnetoresistance effect of NiFe/Pt nanowires

The magnetoresistance of the nanowires, which is their change of electrical resistance when placed in an external magnetic field, is affected by a number of factors including materials, microstructure and geometry of the constituent layers, and strength and direction of applied magnetic field. Here the magnetoresistance effect of individual NiFe/Pt multilayer nanowires has been measured in the CPP geometry by passing current along the nanowire axes. The site-specific CPP-GMR measurements of individual NiFe(35 nm)/Pt(8 nm) and NiFe(5 nm)/Pt(15 nm) multilayered nanowires, and also single phase NiFe nanowires, were carried out with the magnetic field applied both perpendicular and parallel to the wire axis.

The CPP-GMR effect of the individual NiFe/Pt nanowires was calculated as a ratio using GMR% = $[(R_H - R_0) / R_0] \times 100$, where R_H is the electrical resistance of the nanowire in the applied field H and R_0 is the resistance of the nanowire at zero field. Several measurements of GMR for different wires of the same structure at room temperature and external magnetic fields H = 0–500 Oe have been made using the same technique. Fig. 4 shows GMR values for two similar wires at a given field H which were found to be consistent within < 0.3% (Fig. 4), indicating that the wire microstructures are repeatable from a given electrodeposition process.

The observed CPP-GMR effect in the presence of a magnetic field depends on the scattering of electrons within the layers and at interfaces between magnetic (NiFe) and nonmagnetic (Pt) layers [19,20,21]. There could also be boundary scattering of the electrons because the nanowire diameter used in this work is small (~70 nm) [22]. As expected, for a given nanowire microstructure the GMR% for magnetic field applied perpendicular to the wire axis is larger than the GMR% for the magnetic field applied parallel to the wire axis due to the contribution from anisotropic magnetoresistance (AMR) (Fig. 4) [10,23]. To verify this the magnetoresistance (MR%) of individual ~ 50 nm diameter, single phase NiFe nanowires were measured using the same technique with external magnetic field (H) applied perpendicular and parallel to the wire axis. The magnetoresistance was found to be ~ −0.19 % and ~ +0.05 % for H = 500 Oe applied perpendicular and parallel to the wire axis respectively (Fig. 4(e)), which is consistent with the MR% of permalloy nanowires reported by other research groups [24,25].



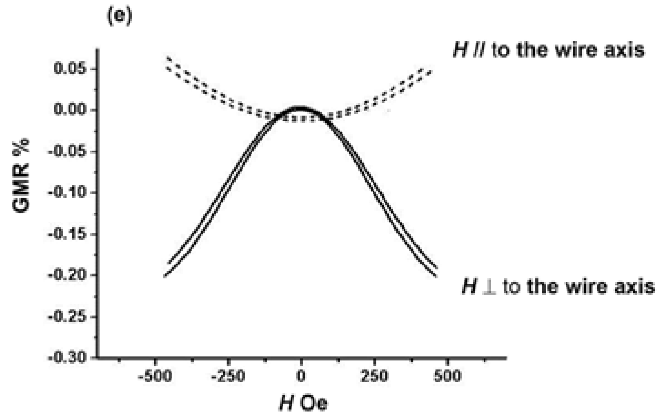


FIG. 4. Magnetoresistance effect of individual NiFe/Pt and single phase NiFe nanowires; resistance versus applied external field H. Measurements from two different nanowires are given in each case. (a and c) H perpendicular to the wire axis of individual NiFe(35 nm)/Pt(8 nm) and NiFe(5 nm)/Pt(15 nm) multilayer nanowires respectively (black line), (b and d) H parallel to the wire axis of NiFe(35 nm)/Pt(8 nm) and NiFe(5 nm)/Pt(15 nm) multilayer nanowires respectively (dashed line) and (e) MR% of individual NiFe nanowires with H perpendicular to the wire axis (black line) and H parallel to the wire axis (dashed line).

The optimal layer thickness of permalloy in NiFe/Cu multilayer nanowires was found by Dubois et al to be 12 nm (with a thickness of 4nm for the Cu layers), which produced a GMR of >70% at low temperature [23]. In the work presented here, the GMR value for a given H is strongly dependant on the layer thicknesses. For external field up to H = 500 Oe, the GMR% measured in magnetic field applied perpendicular ($\theta = 90°$) to the wire axis was found to be 5.8 % for NiFe(35 nm)/Pt(8 nm) but reduced to 2.0 % for NiFe(5 nm)/Pt(15 nm) (Fig. 4(a) and (c)). Similarly for the external magnetic field applied parallel ($\theta = 0°$) to the wire axis the GMR observed at 500 Oe was 5.3 % for NiFe(35 nm)/Pt(8 nm) but reduced to 1.6 % for the NiFe(5 nm)/Pt(15 nm) (Fig. 4(b) and (d)).

The GMR of NiFe/Pt nanowires is affected by the thickness of the nonmagnetic layers, decreasing as the non-magnetic layer thickness increases from 8nm to 15nm. This is because the dipole interaction field between adjacent magnetic layers (which tends to align the magnetizations of the successive magnetic layers antiparallel in the absence of an external magnetic field) reduces [9, 20,26], causing the magnetic layers to orientate more randomly [9,27,28]. The GMR can also reduce rapidly as the thickness of the non-magnetic layer exceeds the non-magnetic material's spin flip diffusion length since the electrons, due to the increasing number of spin flip events, no longer retain their polarization from the previous magnetic layer as they reach the next magnetic layer [22,29]. For the NiFe/Pt system measured here, the GMR% may also decrease with increasing Pt layer thickness due to the increasing resistance contribution of Pt (which has a higher resistivity than NiFe) to the overall wire resistance [18,27].

The applied maximum external magnetic field H for GMR measurements in this work was smaller than that required to achieve GMR saturation and there is limited detailed data in the literature on GMR in the low field 0–500 Oe regime. For multilayer nanowires with nonmagnetic layer thickness (< 10 nm), the averaged GMR% at 500 Oe, determined using $GMR\% = [(R_H - R_0) / R_0] \times 100$, is ~1.8% for FeNiCo/Cu [19], 12% for Co/Cu [30] and 2.2% for CoNiCu/Cu [31]. The GMR values measured here for the individual NiFe/Pt nanowires are of similar magnitude, but lower than the Co/Cu nanowires.

Previous works have concentrated on nanowires embedded in templates, reporting average GMR values calculated from estimated values of contact resistance of the wires [9,10,25,27]. The measurements performed with the new site-specific in-situ technique presented here, though, are directly made on individual nanowires and not based on an average, so the calculated GMR values are an accurate result for future applications. Furthermore, the measurements are performed in-situ in the SEM so the nanowires can be observed in real-time during testing. For these NiFe/Pt nanowires, under the chosen current and magnetic field conditions, no microstructural changes during the GMR measurements were seen but failure of similar NiFe/Pt multilayer nanowires at higher current densities has been observed in previous work [18].

IV. Conclusion

The magnetoresistance effect of ~70 nm diameter individual NiFe/Pt multilayered nanowires has been investigated using a new site-specific technique for in-situ GMR measurement. An accurate measurement of the GMR effect of the magnetic NiFe/Pt multilayer nanowires was carried out in the current perpendicular to plane (CPP) geometry. The CPP-GMR effect of the individual NiFe/Pt nanowires with two different layer thickness geometries was measured at variable magnetic field H up to 500 Oe and with different directions of applied magnetic field with respect to the nanowire axis. The CPP-GMR was dependent on both magnetic field orientation and layer thicknesses, being maximum for NiFe(35 nm)/Pt(8 nm) and H perpendicular to the wire axis. The GMR % of individual NiFe(35 nm)/Pt(8 nm) and NiFe(5 nm)/Pt(15 nm) nanowires measured with H = 500 Oe applied perpendicular to the wire axis was 5.8% and 2.0%; and for H = 500 Oe parallel to the wire axis was 5.3% and 1.6% respectively. For individual single phase NiFe nanowires, anisotropic magnetoresistance was measured as −0.19% for H = 500 Oe applied perpendicular to the wire axis and +0.05% for H = 500 Oe applied parallel to the wire axis. The ability to measure magnetic properties of individual multilayer nanowires and other nanocomponents at specific locations will be of significant benefit as nanowire systems become more diverse and inhomogeneous, and essential for industrial applications in nanodevices and GMR sensors..

References

[1]. J Sarkar, G Khan and A Basumallick Nanowires: properties, applications and synthesis via porous anodic aluminium oxide template, Bull. Mater. Sci. 30 271 (2007).

[2]. Y Cui, Q Wei, H park and C M Lieber, Nanowire nanosensors for highly sensitive and selective detection of biological and chemical species, J. Science 293 1289 (2001).

[3]. L Berger, Emission of spin waves by a magnetic multilayer traversed by a current, Phys. Rev. B 54 9353. (1996).

[4]. S Sun, C B Murray, D Weller, L Folks, and A Moser, Monodisperse FePt Nanoparticles and Ferromagnetic FePt Nanocrystal Superlattices, Science 287 1989 (2000).

[5]. Y Cui and CM Lieber, Functional Nanoscale Electronic Devices AssemUsing Silicon Nanowire Building Blocks, Science 291 851 (2001).

[6]. A Saedi, M Ghorbani, Electrodeposition of Ni–Fe–Co alloy nanowire modified AAO template, Materials Chemistry and Physics 91 417 (2005).

[7]. L Wang, K Yu-Zhang, A Metrot, P Bonhomme and M Troyon, TEM study of electrodeposited Ni/Cu multilayers in the form of nanowires, Thin Solid Films 288 86 (1996).

[8]. Y Peng, T Cullis, I Luxmoore and B Inkson, Electrical properties of individual CoPt/Pt multilayer nanowires characterized by in situ SEM nanomanipulators, Nanotechnology 22 245709 (2011).

[9]. X-T Tang, G-C Wang, and M Shima, Layer thickness dependence of CPP giant magnetoresistance in individual CoNi/Cu multilayer nanowires grown by electrodeposition, Phys. Rev. B 75 134404 (2007).

[10]. S Krimpalis, O Dragos, A Moga, N Lupu and H Chiriac, Magnetization process in electrodeposited NiFe/Cu multilayered nanowires, J. Mater. Res. 26 1081 (2011).

[11]. S Pignard, G Goglio, A Radulescu, L Piraux, J L Duvail, S Dubois and A Decl´emy, Study of the magnetization reversal in individual nickel nanowires, J. Appl. Phys. 87 824 (2000).

[12]. M E Molares, N Chtanko, T W Cornelius, D Dobrev, I Enculescu, R H Blick and R Neumann, Fabrication and contacting of single Bi nanowires, Nanotechnology 15 S201 (2004).

[13]. L Vila, L Piraux, J M George and G Faini, Multiprobe magnetoresistance measurements on isolated magnetic nanowires, Appl.Phys. Lett. 80 3805 (2002).

[14]. J I Martin, J Nogués, Kai Liu, J L Vicent and Ivan K Schuller, Ordered magnetic nanostructures: fabrication and properties, J. Magn. Magn. Mater. 256 449 (2003).

[15]. M S Sander, A L Prieto, R Gronsky, T Sands and A M Stacy, Fabrication of High-Density, High Aspect Ratio, Large-Area Bismuth Telluride Nanowire Arrays by Electrodeposition into Porous Anodic Alumina Templates, Adv. Mater. 14 665 (2002).

[16]. M Elawayeb, Y Peng and B Inkson, Nanostructure and chemical characterisation of individual NiFe/Pt multilayer nanowires, J. Nanosci. Nanotech. 11 1 (2011).

[17]. Y Peng, T Cullis, G Möbus, X Xu and B Inkson, Conductive nichrome probe tips: fabrication, characterization and application as nanotools, Nanotechnology 20 395708 (2009).

[18]. M Elawayeb, Y Peng, K J Briston and B Inkson, Electrical properties of individual NiFe/Pt multilayer nanowires measured in situ in ascanning electron microscope, J. Appl. Phys. 111 034306 (2012).

[19]. P Shakya, B Cox, D Davis, Giant Magnetoresistance and Coercivity of electrodeposited multilayered FeCoNi/Cu and CrFeCoNi/Cu, J. Magn. Magn. Mater. 324 453 (2012).

[20]. S Krimpalis, O G Dragos, M Grigoras, N Lupu and H Chiriac, Magetoresistance and spin transfer torque in electrodeposited NiFe/ Cu multilayered nanowires, J. Adv. Res. Phys. 1 021005 (2010).

[21]. R H Yu, J Zhu, X Zhang and J Tejada, Temperature dependence of the magnetic and transport properties of Co15Cu85 magnetic granular alloys, J. Appl. Phys. 83 3134 (1998)

[22]. K Lin, K Nagodawithana, P C Searson and C. Chien, Perpendicular giant magnetoresistance of multilayered Co/Cu nanowires, Phys. Rev. B 51 7381 (1995).

[23]. S Dubois, C Marchal, J M Beuken, L Piraux, J L Duvail, A Fert, J M George and J L Maurice, Perpendicular giant magnetoresistance of NiFe/Cu multilayered nanowires, Appl. Phys. Lett. **70** 396 (1997).

[24]. Y Rheem, B-Y Yoo, B Koo, W Beyermann and N Myung, Synthesis and magnetotransport studies of single nickel-rich NiFe nanowire, J. Phys. D. Appl. Phys. 40 7267 (2007).

[25]. L Piraux, S Dubois, J Duvail, K Ounadjela and A Fert, Arrays of nanowires of magnetic metals and multilayers: Perpendicular GMR and magnetic properties, J. Magn. Magn. Mater. 175 127 (1997).

[26]. S Dubois, L Piraux, J M George, K Ounadjela, J L Duvail, and A Fert, Evidence for a short spin diffusion length in permalloy from the giant magnetoresistance of multilayered nanowires, Phys. Rev. B **60** 477 (1999).

[27]. X Tang, G Wang and M Shima, Perpendicular giant magnetoresistance of electrodeposited Co/Cumultilayered nanowires in porous alumina templates, J. Appl. Phys. 99 033906 (2006).

[28]. Nicola Spaldin Magnetic Materials: Fundamentals and Device Application (Second edition, Cambridge University Press, UK 2010).

[29]. A Fert, T Valet, and J Barnas, Perpendicular magnetoresistance in magnetic multilayers: Theoretical model and discussion, J. Appl. Phys. **75** 6693 (1994).

[30]. [30]. Z Song, Y Xie, S Yao, H Wang, W Zhang and Z Tang, Microstructure and magnetic properties of electrodeposited Co/Cu multilayer nanowire arrays, Mater. Lett. 65 1562 (2011).
[31]. G Heydon, S Hoon, A Farley, S Tomlinson, M Valera, K Attenborough and W Schwarzacher, Magnetic properties of electrodeposited nanowires, J. Phys. D: Appl. Phys. 30 1088 (1997).