

Artifacts in Ballistic Magnetoresistance Measurements

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Abstract

We have carried out an extensive search for credible evidence to support the existence of a ballistic magnetoresistance (BMR) effect in magnetic nanocontacts. We have investigated both thin-film and thin-wire geometries for both mechanically-formed and electrodeposited nanocontacts. We find no systematic differences between mechanically-formed and electrodeposited nanocontacts. The samples we have investigated include mechanical contacts between ferromagnetic wires, electrodeposited nanocontacts between ferromagnetic wires, ferromagnetic nanocontacts electrodeposited on Cu wires, nanocontacts electrodeposited between ferromagnetic films anchored on wafers, ferromagnetic nanocontacts electrodeposited on Cu films anchored on wafers, nanocontacts between two ferromagnetic films connected by a pinhole through an insulating film, and nanocontacts formed by focused ion-beam etching. In none of these

samples did we find credible evidence for a BMR effect. However, we did find a number of artifacts due to magnetostrictive, magnetostatic, and magnetomechanical effects that can mimic BMR.

Introduction

Over the past several years reports have been published of extremely large values for the magnetoresistance in ferromagnetic nanocontacts when a magnetic domain wall is presumed to lie in the nanocontact.¹⁻¹³ The key idea is that if the spatial extent of the domain wall is as less than the spin-flip mean-free-path of electrons, the electrons trying to cross the wall would have a high reflection probability due to poor matching of the Fermi surfaces of spin-up and spin-down electrons. This reflection would be manifest as a higher electrical resistance than when both sides of the nanocontact were magnetized in parallel.

The ballistic magnetoresistance values that have been reported are far beyond those of the giant magnetoresistance (GMR) effect. The implication was that GMR might soon be supplanted in device applications by a vastly larger BMR effect. The results were also of great interest to theorists since a satisfactory theory to explain the extremely large values was not in hand.

Experimental

The wires used in this work had a purity of 99.9% or better and were mounted on glass slides with epoxy. Except where otherwise noted, all electrodeposition of Ni and Fe nanocontacts was carried out between -1.0 V and -1.5 volts versus a standard calomel electrode. A detailed account of the electrodeposition may be found in Refs. 15 and 16.

Resistivity measurements were made either by 2-point or where possible by 4-point techniques with an estimated accuracy of $\pm 0.01\%$ of the measured value.

All films were deposited on Si(100) with 250 nm of thermal oxide. Wafers were cleaved into ≈ 2 cm x 2 cm pieces, cleaned ultrasonically in a detergent solution, rinsed in distilled water, blown dry, and installed in the deposition chamber. After bakeout, the deposition chamber has a base pressure of 3×10^{-8} Pa (2×10^{-10} Torr), of which 90% is H₂. The metal films were deposited at room temperature by dc-magnetron sputtering in 0.3 Pa (2 mTorr) Ar at a typical rate of ≈ 0.05 nm/s.

Results and Discussion

The original report of the BMR effect and a number of subsequent reports were based on the geometry illustrated in Fig. 1. Coils were wound around the Ni wires and current passed through them in an attempt to alternately magnetize the Ni wires in parallel and antiparallel states. In such experiments, if the switching is by domain wall motion and if the wires are axially magnetized during the electrical resistance measurements, magnetostriction should not be a problem. (Magnetostriction can, however, be a problem when the two states being compared are axially saturated and remanent.)

Unfortunately, it appears that in the geometry of Fig. 1 the additional factor of the magnetostatic force between the two wires is very important. A magnetostatic attractive force between the ends of magnetized wires with radius a can be obtained by considering the field energy in the gap between the ends. For flat wire ends,

the field energy in the gap is $E = \mu_0 M_s^2 \pi a^2 d / 2$, where d is the distance between the wires, $d \ll a$. The force on the wire is then

$$F = \beta^{-1} \mu_0 M_s^2 \pi a^2$$

where $\beta = 2$ for flat ends. A lower bound on the force for hemispherical ends can be obtained by considering the force between two magnetized spheres with dipole moments

$$\Delta l = \frac{\mu_0 M_s^2}{\beta Y} l$$

$m = 4\pi M_s a^3 / 3$ separated by a distance $2a$. In this case a similar expression for the force is obtained with $\beta = 6$. For 0.5 mm dia Ni wires with rounded ends this is a force of approximately 0.01 N. These magnetostatic forces will produce an elongation Δl in a wire of length l where Y is the modulus of elasticity. Using a typical wire length of 4 mm and $Y = 2 \times 10^{11}$ Pa, a lengthening of 1 nm is predicted for hemispherical ends. For flat ends the predicted lengthening is 6 nm. In general, our wires are intermediate between hemispheres and flat. When the wires are magnetized antiparallel there is an equal contraction in each wire that will occur due to the repulsive magnetostatic force. Thus, between the parallel and antiparallel states a change in separation between the ends of the wires between 4 nm and 24 nm will occur. Note that this result is independent of the wire diameter.

The physical dimensions of BMR nanocontacts are generally thought to be on the order of ~ 1 nm to ~ 10 nm. It would certainly be expected that a displacement of between 4 nm and 24 nm would have a profound effect on the structure of such nanocontacts and likewise on their resistivity.

An additional factor to be considered is how well the Ni wires are anchored at their ends. If they are anchored by epoxy, the displacement may be much greater since epoxy typically has a modulus of elasticity a factor of 100 smaller than Ni.

In some cases, the Ni wires are entirely immersed in epoxy. However, with a factor of 100 difference in modulus of elasticity the epoxy will largely deform to meet the demands of the Ni.

Note that any such stretching or contracting of the wires will mimic BMR. In the parallel alignment state the two wires will push together making a more intimate contact and lowering the resistance just as BMR would predict. Thus, Fig. 1 illustrates a geometry tailor-made for artifacts in the data. The conclusion to be drawn is that artifacts are very likely in any data recorded using the geometry of Fig. 1.

Figure 2 illustrates another common geometry for BMR measurements that is likewise prone to serious artifacts. In this geometry, at zero field the Ni wires will break up into domains. When an axial applied field magnetizes the axial wire, magnetostriction will make the axial wire shorter and the resulting force will pull on the nanocontact, making it less intimate. Not surprisingly in this geometry, the data frequently appears like the inset in Fig. 2, with low resistance at low field and a constant higher resistance at high field. We have found this type of data in both electrodeposited and mechanical nanocontacts using the geometry of Fig. 2. The constant resistance at high field is a characteristic of a magnetoresistance-induced effect because after the axial wire is magnetized axially, increasing the field produces no additional shortening. For a length of Ni wire of 4 mm the contraction due to magnetostriction is 136 nm or far more than is needed to deform a nanocontact.

Figure 3a presents data we have obtained using the geometry of Fig. 2. According to current publishing standards, the data of Fig. 3a would entitle us to claim the discovery of an infinite BMR effect. However, it is clear that all that has happened is that magnetostriction caused the nanocontact to break. This breakage is not surprising in light of the 136 nm displacement. The conclusion to be drawn is that the geometry of Fig. 2 is so vulnerable to artifacts that it cannot be used to establish the validity of BMR.

Moreover, there are additional factors at work to cause additional artifacts in experiments using the geometry of Fig. 2. Fig. 3b and 3c illustrate results obtained with permalloy wire in the geometry of Fig. 2. Permalloy has almost no magnetostriction, and using it we can largely eliminate artifacts due to magnetostriction. However, as Fig. 3b and 3c indicate we can still get infinite MR. It is not difficult to identify some of the forces at work here, although we should be careful to point out that there may well be others we have not identified yet. The heavy arrows in Fig. 4a indicate a magnetostatic force that will be applied to the transverse wire if it is mounted in such a way that it can experience the field gradient of the magnet. Such a force would tend to break the contact. It appears that this effect may be contributing to the data in Fig. 3b. As the field increases the transverse wire experiences an attraction towards the pole face that could break the nanocontact. Then, at a higher field the contact is reestablished. The magnetostatic attraction illustrated in Fig. 5 may be responsible, although we have not worked out the details of the magnitudes of the forces involved. Note here that the magnetostatic attraction described in Fig. 1 is also at work in the geometry of Fig. 2. As illustrated in Fig 5, there will be an attractive force between the two wires as they approach parallel magnetization. The force will be equivalent to the force of gravity on a mass of 1 gram

(as in Fig. 1). Such a force on the transverse wire has the potential to deflect the transverse wire a far greater distance than the stretching described in Fig. 1.

To test these ideas, measurements were made on samples using the geometry of Fig. 4b. In this case, the transverse wire is mounted in the region of homogeneous field to eliminate the attraction to the pole face. The data is presented in Fig. 3c. For a sample initially having infinite resistance the attraction illustrated in Fig. 5 brings the wires into contact, and there is no longer any evidence of the pole face attracting the transverse wire as seemed to be the case in Fig. 3b. Thus, simple consideration of elementary magnetostatic and magnetomechanical effects may explain the data without any need to resort to BMR.

It should be pointed out that the underlying assumption here is an infinite MR effect must be due to breaking the nanocontact. Theory predicts an infinite BMR effect only when a single channel of quantized conductance is present, and the resistivities of our samples show that hundreds or thousands of channels are present.¹⁴

Figure 6 presents a set of data on different samples using the geometry of Fig. 2. These data represent three common generic shapes that are the analogs of the three special results described in Fig. 3. In Fig. 6 the MR is large but not infinite. Given the qualitative similarity with the data in Fig. 3 it seems that the same forces are at work in both data sets. The principle difference is that in the samples of Fig. 6 the forces were not sufficient to break the nanocontact only to deform it. It should be emphasized that the data of Fig. 6 represents some of the most clear cut cases. Much of the data is more complicated, but it often seems to be combinations of the three generic types shown in Fig. 6. It is easy to imagine that subtle differences in sample geometries could produce a

wide range of mixtures of the artifacts that produce the three generic curves of Fig. 6. The conclusion to be drawn from the qualitative similarity of Figs. 3 and 6 is that forces are at work in the geometry of Fig. 2 that can easily mask any true BMR effect, making this geometry incapable of providing credible evidence for a real BMR effect.

In seeking to design artifact-free experiments that can identify a real BMR effect, we have explored many options for suppressing the physical displacement of wires due to the forces described above. These include: ferromagnetic nanocontacts electrodeposited on Cu wires, nanocontacts electrodeposited between ferromagnetic films anchored on wafers, ferromagnetic nanocontacts electrodeposited on Cu films anchored on wafers, nanocontacts between two ferromagnetic films connected by a pinhole through an insulating film, and nanocontacts formed by focused ion-beam etching. However, in none of these samples did we find credible evidence for a BMR effect. Other published work along these lines has also failed to find credible evidence for a real BMR effect.¹⁷⁻²⁵ These results raise the possibility that there may be no real BMR effect of any significant magnitude.

In the thin-film work we sometimes found that films would become detached from the thermal-oxide substrate and become subject to the artifacts of wires. We found an effective way to prevent detachment is to deposit 1 nm of Ta on the thermal oxide. It forms a strong bond to both the thermal-oxide substrate and to any metal film subsequently deposited. In this way films may be anchored to the substrate.

We have found one additional artifact that can produce very large MR in contacts electrodeposited on thin films anchored to wafers. If an unusually high voltage is used for electrodeposition, the deposit may not be a continuous film but a granular assembly of

particles. Figure 7 presents an example. The heavy lines in Fig. 7 show the shape of the Cu films underlying a granular deposit of Fe produced with a deposition voltage of -3.8V. Mossbauer spectroscopy shows that the Fe particles are metallic and ferromagnetic. The data from such samples is qualitatively similar to Fig. 6c. However, upon observing these deposits under an optical microscope it was noted that the Fe particles move as a field is applied. To immobilize the particles in one sample we used a drop of nail polish (a varnish). After the nail polish hardened, the MR in this sample dropped from 70% to 0.7%. The artifact at work here is illustrated in Fig. 8. When the field is applied and the Fe particles are magnetized in the same direction they clump together like simple bar magnets to create new conducting paths and lower the resistance.

When the electrodeposition is carried out at more normal potentials such as -1.0 to -1.5 V a continuous metallic film is produced. However, in such samples there is never any indication of BMR, and the observed MR is typically less than 0.4%.

Conclusions

- 1) Many previous attempts to observe a BMR effect have been subject to serious artifacts that can mimic BMR.
- 2) Experiments carefully designed to avoid these artifacts do not provide evidence for a real BMR effect.
- 3) It is entirely possible that there is no real BMR effect of any significant magnitude.

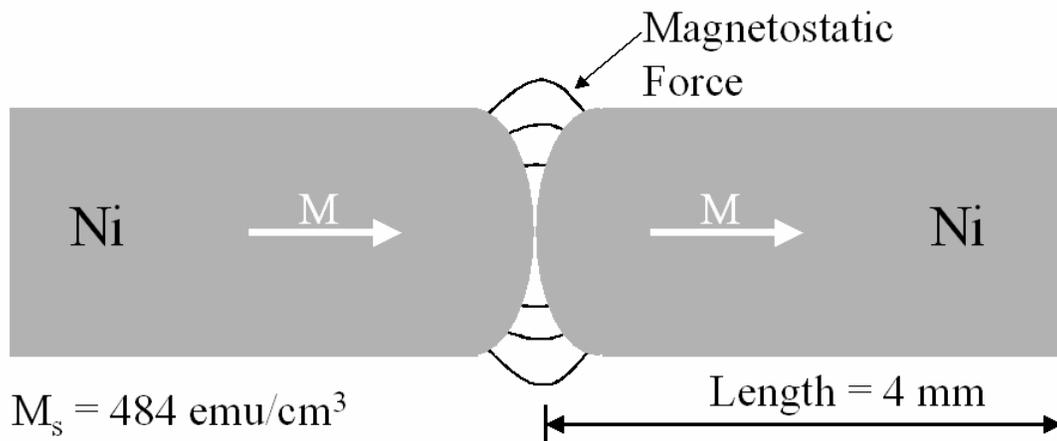
References

- 1) N. GarcPa, M. MuZoz, and Y.-W. Zhao, Phys. Rev. Lett. 82, 2923 (1999).
- 2) N. GarcPa, H. Rohrer, I. G. Savelief, and Y.-W. Zhou, Phys. Rev. Lett. 85, 3053 (2000).
- 3) N. GarcPa, M. MuZoz, G. G. Quan, H. Rohrer, I. G. Savelief, and Y.-W. Zhou, Appl. Phys. Lett. 79, 4550 (2001)
- 4) N. GarcPa, M. MuZoz, and Y.-W. Zhao, Appl. Phys. Lett. 76, 2586 (2002).
- 5) N. GarcPa, G. G. Qian, and I. G. Savelief, Appl. Phys. Lett. 80, 1785 (2002).
- 6) N. GarcPa, H. Wang, H. Cheng, and N. D. Nikolic, IEEE Trans. Mag. 39, 2776 (2003).
- 7) Harsh Deep Chopra and Susan Z. Hua, Phys. Rev. B 66, art. no. 020403 (2002).
- 8) Susan Z. Hua and Harsh Deep Chopra, Phys. Rev. B 67, art. no. 060401 (2003).
- 9) J. J. Versluijs, M. A. Bari, and J. M. D. Coey, Phys. Rev. Lett. 87, art. no. 0266011 (2001).
- 10) J. M. D. Coey, J. J. Versluijs, M. Venkatesan, J. Phys. D 35, 2457 (2002).
- 11) M. Viret, S. Berger, M. Gabureac, F. Ott, D. Olligs, I. Petej, J. F. Gregg, C. Fermon, G. Francinet, and G. Le Goff, Phys. Rev. B 66 art. no. 220401 (2002).
- 12) J. Baszynski, T. Tolinski, W. Kowalski, A. Kowalczyk, Czech. J. Phys. 52, A13, Suppl. A (2002).
- 13) J.-E. Wegrowe, T. Wade, X. Hoffer, L. Gravier, J.-M. Bonard, and J.-Ph. Ansermet, Phys. Rev. B 67, art. no. 104418 (2003).
- 14) L. R. Tagirov, B. P. Vodopyanov, and K. B. Efetov, Phys. Rev. B 65 art. no. 214419 (2002).

- 15) J. J. Mallett, E. B. Svedberg, H. Ettetdgui, T. P. Moffat and W. F. Egelhoff, Jr., Appl. Phys. Lett., in press.
- 16) E. B. Svedberg, J. J. Mallett, H. Ettetdgui, L. Gan, P. J. Chen, A. J. Shapiro, T. P. Moffat, and W. F. Egelhoff, Jr., Appl. Phys. Lett., in press.
- 17) A. D. Kent, U. Ruediger, J. Yu, S. Zhang, P. M. Levy, Y. Zhong, S. S. P. Parkin, IEEE Trans. Mag. 34, 900 (1998).
- 18) S. J. C. H. Theeuwen, J. Caro, K. I. Schreurs, R. P. van Gorkom, K. P. Wellock, N. N. Gribov, S. Radelaar, R. M. Jungblut, W. Oepts, R. Coehoorn, and V. I. Kozub, J. Appl. Phys. 89, 4442 (2001).
- 20) I. V. Roshchin, J. Yu, A. D. Kent, G. W. Stupian, and S. Leung, IEEE Trans. Mag. 37, 2101 (2001).
- 21) K. Mibu, K. Shigeto, K. Miyake, T. Okuno, T. Ono, T. Shinjo, Phys. Stat. Sol. (a) 2, 567 (2002).
- 22) C. C9spedes, M. A. Bari, C. Dennis, J. J. Versluijs, G. Jan, J. O'Sullivan, J. F. Gregg, and J. M. D. Coey, J. Mag. Mat. 242-245, 492 (2002).
- 23) K. Miyake, K. Shigeto, K. Mibu, and T. Shinjo, Appl. Phys. Lett. 91, 3468 (2002).
- 24) C.-S. Yang, J. Thiltges, B. Doudin and Mark Johnson, In-situ monitoring of quantum conductance in electrodeposited magnetic point contacts, J. Phys.: Condens. Matter **14**, L765 (2002).
- 25) E. Elhoussine, S. Matefe-Tempfil, A. Encinas, and L. Piraux, Appl. Phys. Lett. 81, 1681 (2002).

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Modulus of elasticity = 2×10^{11} Pa for Ni

= 3×10^9 Pa for epoxy

Each wire stretches ≥ 1 nm!

Figure 1 An illustration of a common geometry for BMR measurements and how it is subject to the artifact that the wires stretch when magnetized in parallel. When magnetized antiparallel they will retract an equal amount. If the nanocontact between them is becomes smaller upon stretching and larger upon compressing the resistivity change will mimic BMR. M_s is the saturation magnetization of Ni.

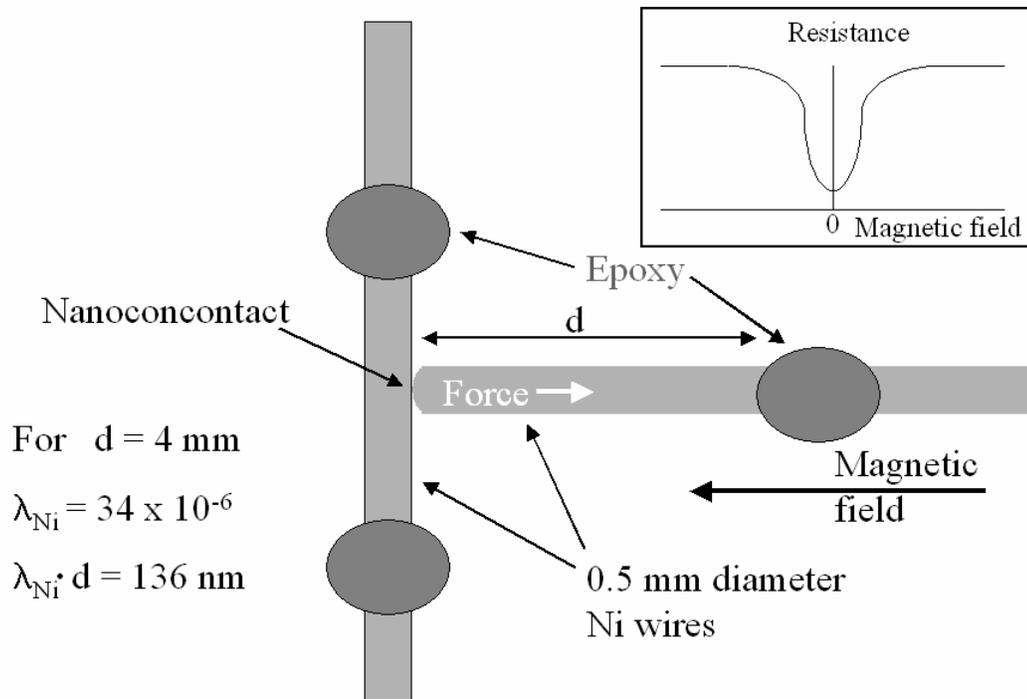


Figure 2 An illustration of another common geometry for BMR measurements and how it is subject to the artifact that magnetostriction will shorten the axial wire in an applied field. A resulting force will tend to stretch the nanocontact and if upon stretching it becomes smaller the resistance change will have the general features of the inset. The symbol λ_{Ni} stand for the magnetostriction constant for Ni.

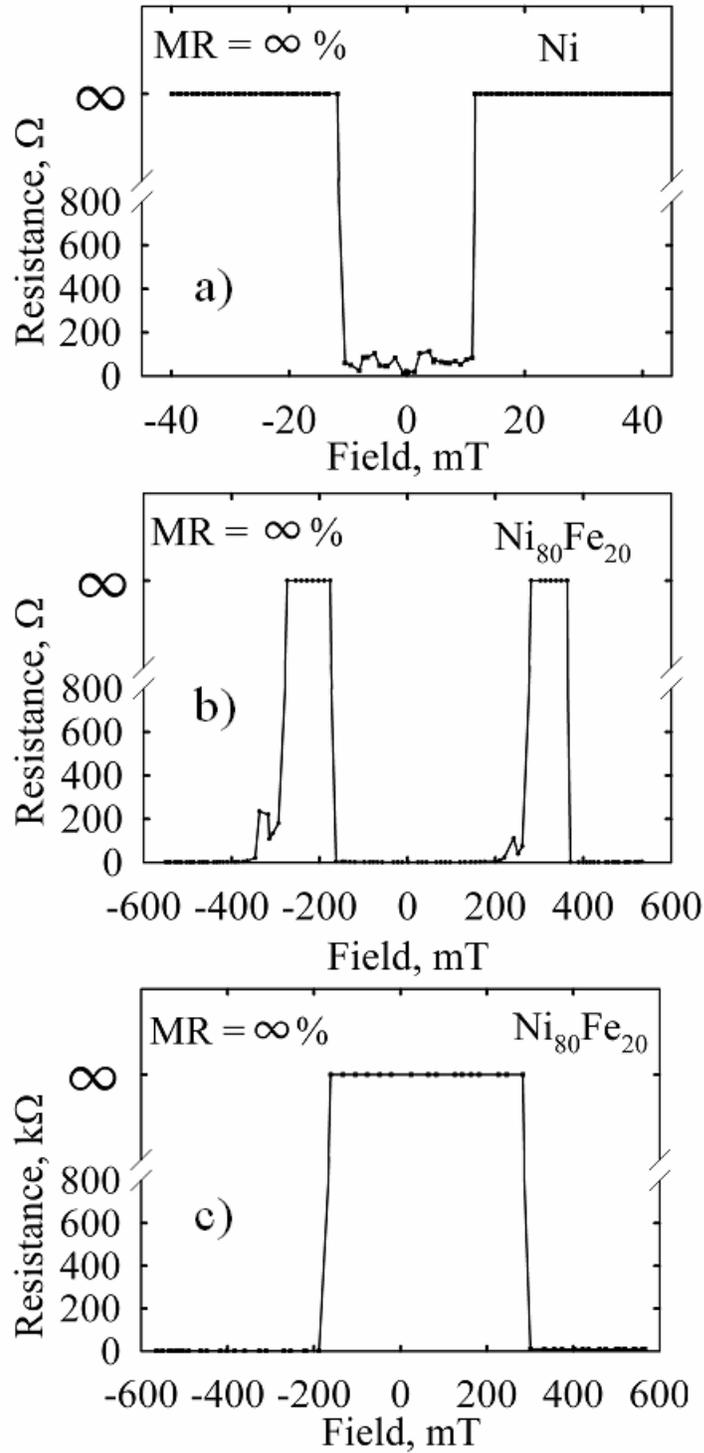


Figure 3 Magnetoresistance data consisting of a single sweep for a) Ni wires in the geometry of Fig. 2, b) permalloy wires in the geometry of Fig. 4a, and c) permalloy wire in geometry of Fig. 4b.

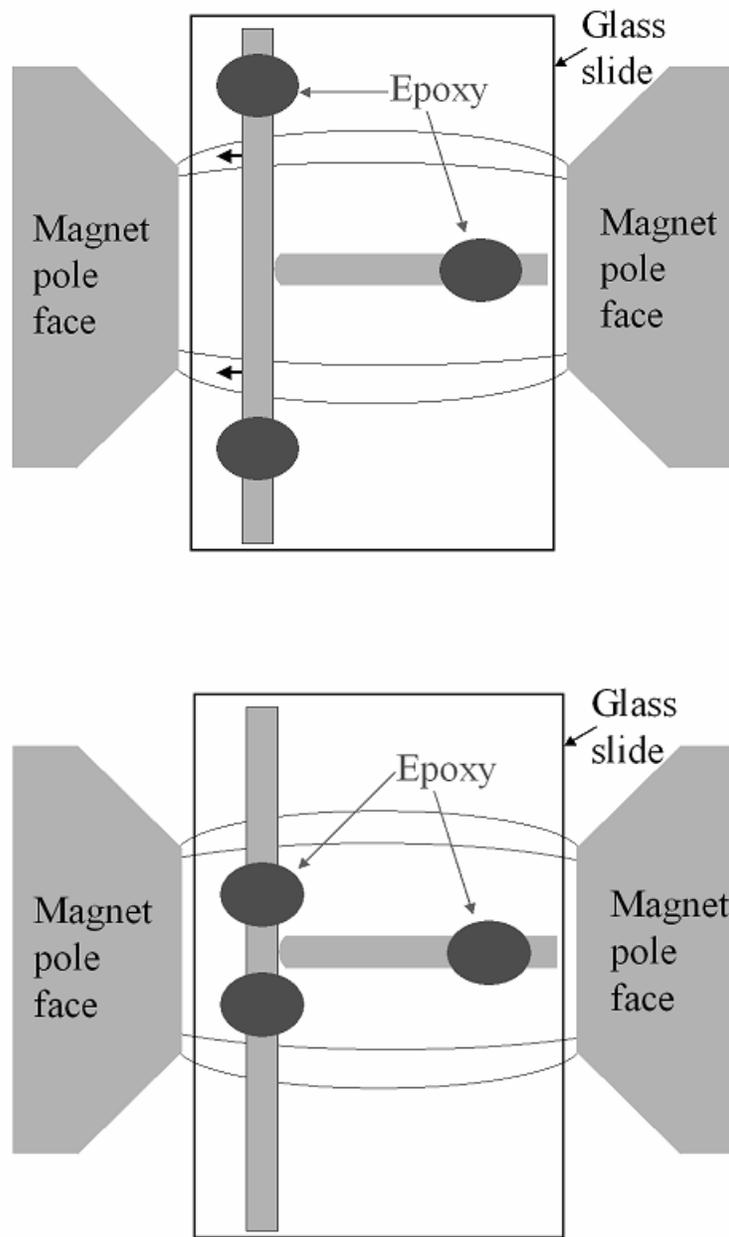


Figure 4 An illustration of different geometries that can give differently shaped MR plots due to different artifacts being present.

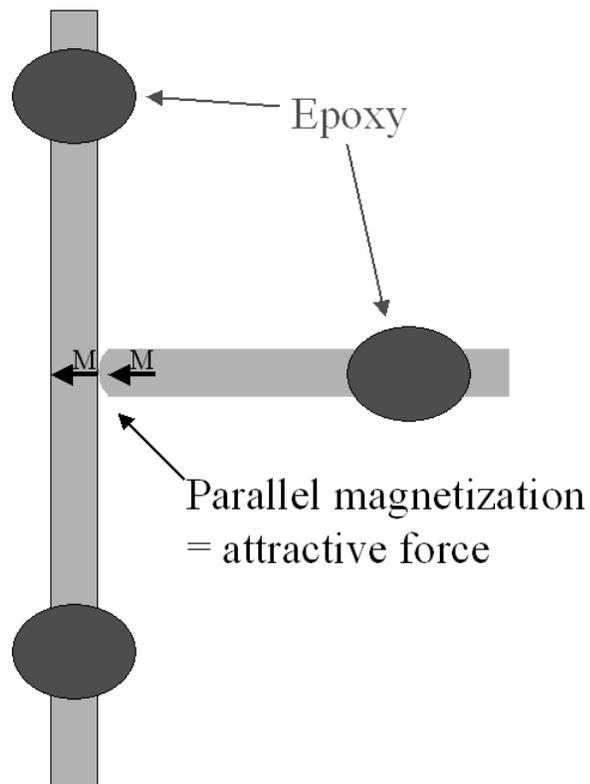


Figure 5 An illustration of how the artifact described in Fig. 1 can be present in the geometry of Fig. 2 but with a much greater effect since a slight deformation of the epoxy will allow the transverse wire to be easily deflected towards the axial wire.

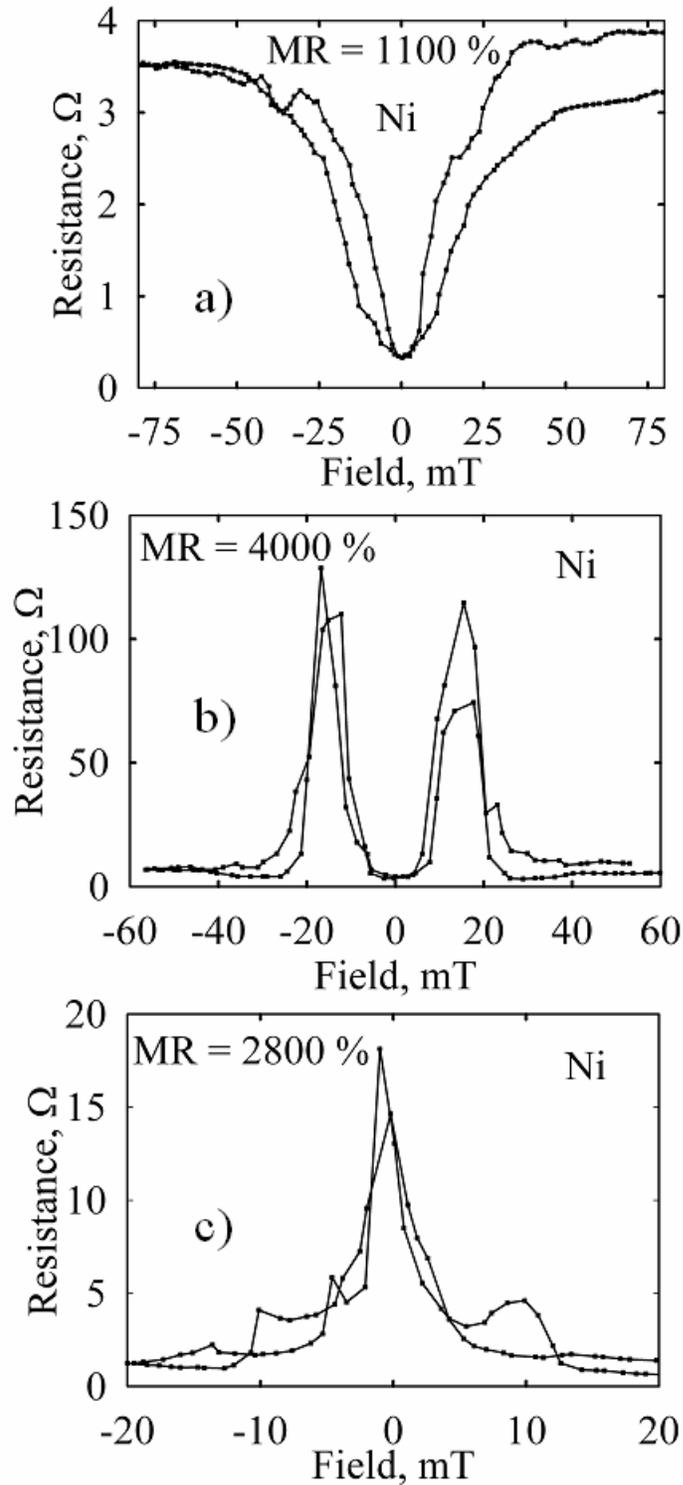


Figure 6 Three different generic types of data obtained on Ni samples in the geometry of Fig. 2, illustrating how inadvertent differences in sample mounting can lead to quite different artifacts dominating the data.

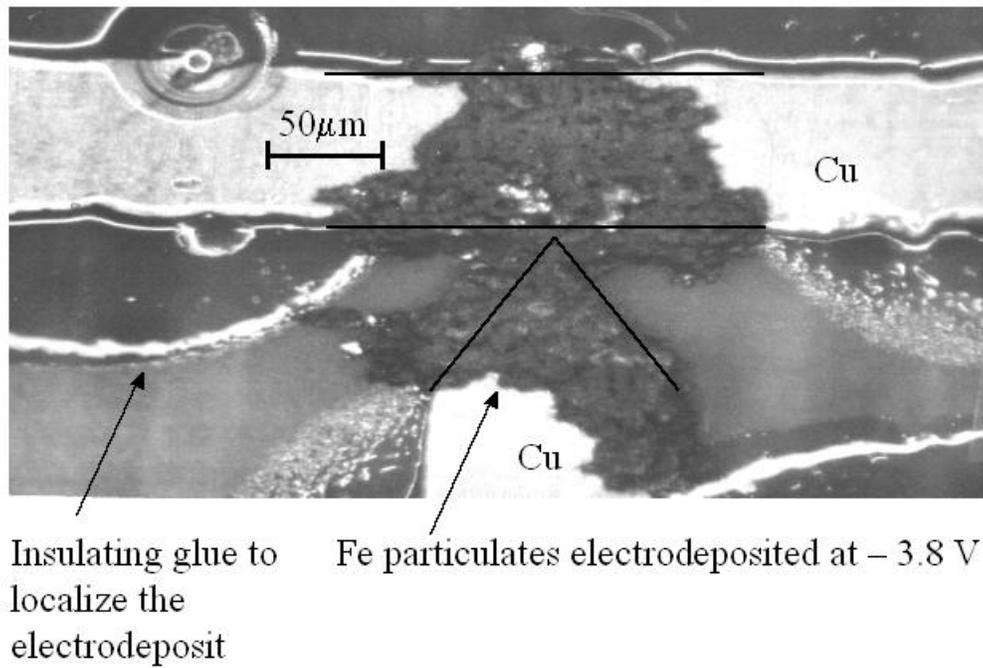


Figure 7 An optical image of the Fe particulate deposit that is found when Fe is electrodeposited at the unusually high potential of -3.8 V on a gap between Cu films.

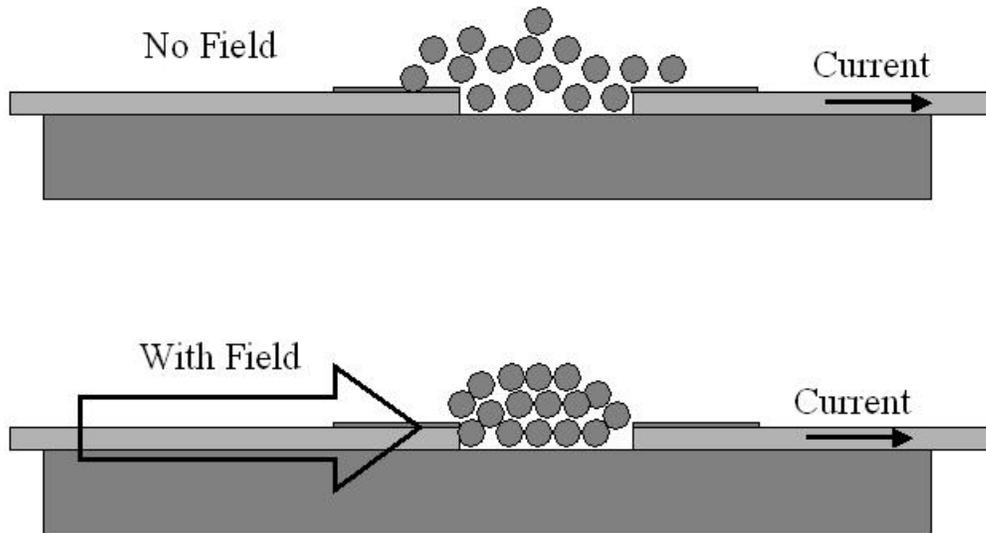


Figure 8 An illustration of the artifact manifest in data on samples like the one in Fig. 7 showing how magnetostatic forces cause Fe particles to clump together creating new conducting paths, lowering the resistance, and producing data qualitatively similar to that in Fig. 6c.