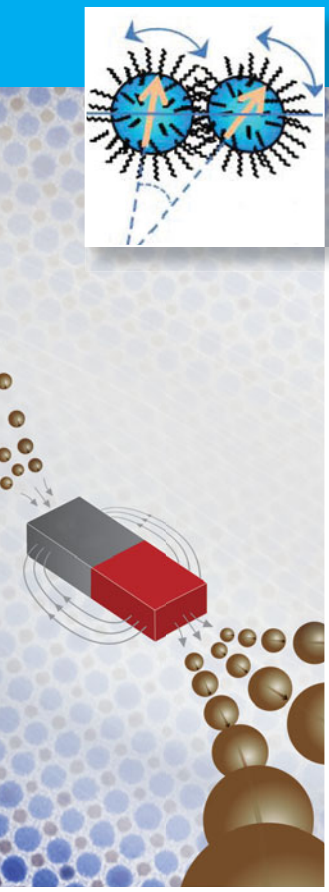


Magneto-transport and magnetization dynamics in magnetic nanoparticle assemblies

Gil Markovich

When colloidal nanoparticles made of soft magnetic materials have strong interparticle interactions, new magnetic phases may be formed, such as super-ferromagnets or super-spin glasses, with different local temperature and field-dependent magnetization dynamics. Magneto-transport experiments on nanoscale tunnel junctions formed in magnetic nanoparticle arrays, defined either by scanning probe tips or patterned nanoscale electrodes, can be used as probes of the magnetization dynamics.

Introduction

The magnetostatic interactions of neighboring magnetic nanoparticles can be tuned by their separation distance and magnetic moment. Since each ferro- or ferrimagnetic particle has some degree of spin polarization, interesting magnetic field and temperature-dependent electrical resistance effects may be observed if there is electron transport between particles.[1]

Recent achievements in tuning interparticle separations with short ligand molecules have led to improved control over interparticle tunnel resistance,[2] which may enable the preparation of sensitive magnetoresistive sensors made of colloidal magnetic nanoparticle films. With the fast development of printed electronics, these advances in nanoparticle-based magnetoresistive devices might lead to simple preparation methods of printable magneto-electronic devices, such as magnetic sensors,[3] as alternatives to magneto-resistive or Hall effect devices prepared by expensive vacuum deposition techniques.[4,5]

Until 15 years ago, studies of the physical properties of colloidal single domain magnetic particles were limited to measurements of magnetization or magnetic susceptibility of nanoparticle ensembles. Fundamental questions regarding equilibrium versus non-equilibrium (spin-glass-like) dynamics, including aging and memory effects, attracted much scientific interest for a general understanding of non-equilibrium systems with slow dynamics,[6–9] but those studies were limited by broad particle size and interparticle separation distributions.

This article describes magneto-transport experiments carried out on magnetic nanoparticle assemblies and shows how nanoscale electron transport studies in close-packed magnetite nanoparticle arrays may reveal temperature and magnetic field dependent single- or few-particle magnetization dynamics in such arrays. The discussion here is limited to colloidal ferro-/ferrimagnetic nanoparticles coated with organic surfactant molecules that control the interparticle spacing in close-packed arrays. These are the only systems in which various parameters, such as particle diameter, interparticle separation, and number of particle layers, can be well controlled separately. The finite and insulating separation between the nanoparticles makes the short-range (exponentially decaying) electronic exchange interactions between neighboring nanoparticles insignificant. The exchange interaction is the driving force for nearest-neighbor magnetic spin-spin alignment in ferromagnetic materials.

Ensemble measurements of magnetic nanoparticle magnetization dynamics

For close-packed nanoparticles consisting of low-anisotropy phases such as Fe_3O_4 and ε-Co, the dipole-dipole magnetostatic interaction energy may exceed the individual particle anisotropy energy barrier by up to an order of magnitude, and significant differences in magnetic properties would be found between close-packed arrays, where interparticle

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separation is much smaller than particle size, and isolated particle measurements. For example, in ~8 nm magnetite nanoparticles, the blocking temperature (T_b, also see the Introductory article in this issue) shifts from ~50 K for isolated particles to ~150–200 K for a close-packed array.[10]

Upon cooling from a superparamagnetic (SPM) state at high temperature, strongly interacting magnetic nanoparticle arrays may assume new magnetic states, such as super-ferromagnetic (SFM), where the magnetic moments of the particles form ordered domains and develop hysteresis, or superspin glass (SSG), where glass-like properties, such as spin frustration, slow dynamics, aging, and memory effects can be observed (see **Figure 1**).[11] The dimensionality of such arrays may also affect the magnetic properties.[12,13,16] The tentative phase diagram in Figure 1 shows interacting magnetic nanoparticles as a function of their interaction strength. Magnetic nanoparticle arrays were studied by a variety of techniques such as magnetometry; neutron scattering;[14] special electron microscopy techniques such as Lorentz microscopy[15] and electron holography;[16] and other magnetic imaging techniques[17] as a function of temperature, magnetic field, or structural order. Observations of SSG behavior have been claimed for nanoparticle systems with an intermediate strength of dipolar interactions. The particles' magnetic moments freeze collectively into a spin glass phase below a glass transition temperature, T_g. On increasing the particle density and, hence, the interaction between the particles, collective ferromagnetic-like correlations lead to a SFM phase with presumably different properties from those of a SSG.

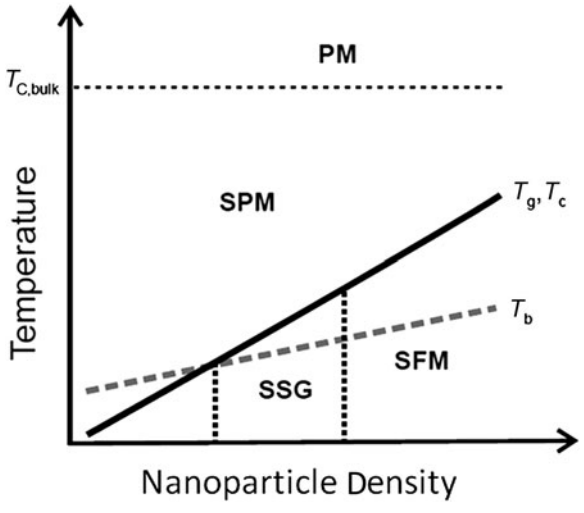


Figure 1. Schematic phase diagram, transition temperature versus nanoparticle array density, with paramagnetic (PM), superparamagnetic (SPM), superspin glass (SSG), and superferromagnetic (SFM) phases. Relevant lines are the blocking temperature of the individual particles, T_b, and the collective transition line (i.e., the glass transition, T_g) or SFM transition temperature, T_c. $T_{C,bulk}$ is the bulk ferromagnet Curie temperature. Adapted with permission from Reference 11. © 2009 IOP Publishing.

In SPM systems, the exponential dependence of magnetization relaxation time on particle volume causes small-sized polydispersity to significantly smear T_b and makes the temperature dependence of magnetization relaxation times look like a spin-glass (superposition of magnetization decays with a large spread of relaxation times) even when it is not glassy. Hence, the distinction between a glass-like state and a dipolar interaction governed ferromagnetic-like state is not simple, and often, magnetization and susceptibility data are not enough to conclusively determine which phase is obtained, in particular with respect to magnetization dynamics. These ambiguities call for experiments to obtain more information on the magnetization behavior of strongly interacting magnetic nanocrystal arrays, preferably with single nanoparticle resolution.

Magnetic field-dependent electron transport in magnetic nanocrystal arrays

The first step toward the measurement of local magnetization dynamics in magnetic nanoparticle arrays is establishing spin-polarized electron transport at such arrays; in other words, demonstrating magnetoresistance (MR) in the nanoparticle arrays. In particular, magnetite has been discussed as a half-metal and is supposed to be a highly spin-polarized conductor.[18] This property, together with its low magneto-crystalline anisotropy, makes it ideal for studying the dipolar SSG and SFM phases through magneto-transport measurements in magnetite nanocrystal arrays with particle sizes on the order of 10 nm or less.

To study localized magneto-transport properties over a small number of nanocrystals, nanoscale electrodes with small gaps need to be used, as depicted in **Figure 2**a. Such nanoscale magneto-transport experiments in close-packed arrays of colloidal magnetic nanoparticles began with the work of Black et al. on ε-Co nanoparticle arrays.[1] To achieve measurable tunnel currents at low temperatures, the separation between the nanoparticles must be reduced. The electrically insulating oleic acid coating of any conducting nanoparticle produces separation distances of ~2–4 nm, which are too large to obtain measurable currents. Reducing this separation by various thermal or chemical treatments to <1 nm allows for electron transport measurements in such arrays. The exponential sensitivity of the tunnel rate to interparticle separation and barrier height causes thermally activated transport to occur over a small subset of percolating pathways through the arrays, as portrayed by the blue nanoparticle path in Figure 2a. Moreover, along the percolating path, the various tunnel junction resistances connected in series are also non-uniform and may have a wide distribution of values. Thus, there is a high probability that in a nanoscale section of a magnetic nanoparticle array, such as the one sketched in Figure 2a, there will be only one (or very few) junction(s) that dominate the overall resistance.

The maximal MR achievable in magnetic nanoparticle arrays should obey predictions for tunnel MR (TMR) between pairs of ferromagnetic electrodes or nanoparticles. Julliere's

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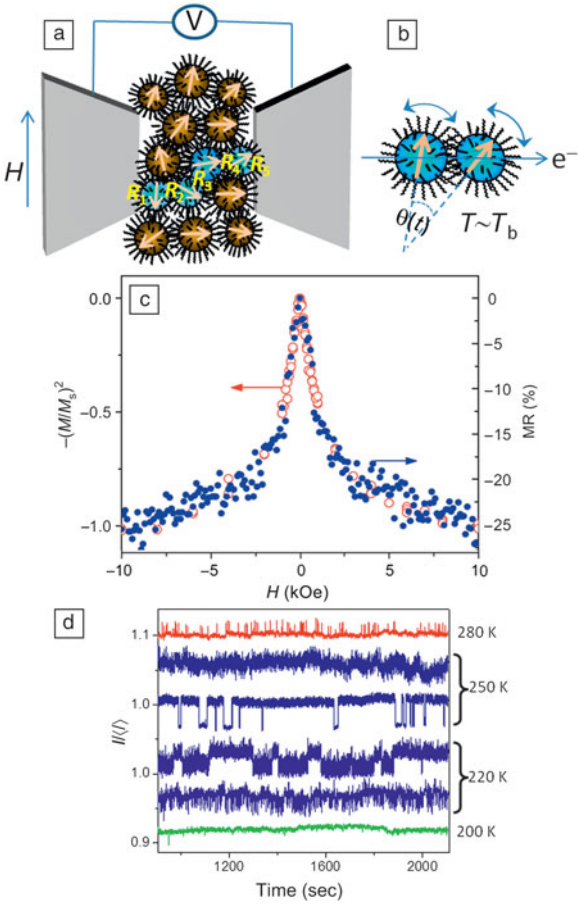


Figure 2. (a) Schematic nanoscale tunnel junction where a small finite number of close-packed colloidal magnetic nanoparticles coated with insulating organic surfactant contribute to the conduction between the electrodes. The blue particles are connected in a percolating electron transport path. The values of interparticle resistances R_1–R_5 may vary over many orders of magnitude. The brown particles are characterized by interparticle resistances that are larger than those indicated and thus not participating in the conducting network. (b) In a particular interparticle junction, when the temperature (T) is tuned near the blocking temperature (T_b), the individual particle magnetizations may fluctuate at different rates, and the relative angle between the orientations (θ) would fluctuate with time (t), changing the interparticle tunnel rate. (c) Magnetoresistance (MR, filled circles) and normalized squared magnetization value (M^2, open circles) as a function of magnetic field (H) near T_b (220 K) for a nanoscale junction patterned in a magnetite nanocrystal array. The fit between MR and M^2 is predicted by the simple tunnel magnetoresistance model of Reference 40. Reproduced with permission from Reference 10. © 2009 Elsevier. (d) Current (I) versus time traces measured at such a junction at several temperatures under a zero external magnetic field normalized to the average current ($\langle I \rangle$) and shifted for clarity. Reproduced with permission from Reference 32. © 2010 IOP Publishing. Note: M_s, saturation magnetization.

model describes an idealized case of tunneling between two ferromagnetic electrodes (or nanoparticles) with spin-state conservation of the tunneling electrons[19] in a uniaxial system (magnetization aligned along a single axis):

$$TMR = \frac{G_{AP} - G_P}{G_{AP}} = \frac{2P^2}{1-P^2}, \quad (1)$$

where G_{AP} and G_P are the tunnel conductances when the two electrodes (particles) have anti-parallel and parallel magnetizations, respectively, and P is the spin-polarization level of the material. In the case of magnetic nanoparticle arrays, it is clear that this is only a limiting case where the parallel configuration can be achieved with a saturating field, and anti-parallel alignment would be favored in zero field. Since a major part of the electron transport is thermally activated,[20] the TMR may be less than the Julliere model prediction.

Several groups have observed MR effects in close-packed Fe_3O_4 and Fe nanoparticle arrays,[20–25] but most found P values that were significantly lower than that for an ideal half-metal (P = 100%). In some cases, annealing brought the particles into contact and drastically changed the electron transport mode. Taub et al. obtained a field dependence of MR, which follows the square of the magnetization ($MR(H) \propto M(H)^2$, where M is the magnetization, and H is the magnetic field (see Figure 2c),[20] as expected from the TMR model of Inoue and Maekawa for relatively low P values (<50%), which allows for non-collinear magnetization orientations of the electrodes (particles).[26] This model uses a Langevin function to describe moment alignment of the SPM nanoparticles as a function of field and temperature. An additional interesting aspect of nanoscale magneto-transport measurements of Fe_3O_4 nanocrystals is the observation of the Verwey metal-insulator transition, which is a combined electronic-structural transition, and its mechanism is still under debate after nearly 80 years.[27] Poddar et al.[28] and other research groups[29,30] have observed this transition through electron transport measurements in magnetite nanostructures.

Magneto-transport experiments in close-packed magnetic nanoparticle arrays have thus shown that with sufficiently narrow tunnel barriers between nanoparticles, the MR behavior is that of model TMR between partially spin-polarized SPM electrodes.

Temporal fluctuations of MR in nanoparticle-based tunnel junctions

In cases where a single nanoparticle pair dominates the TMR behavior, the resistance of the device will depend on the local characteristics of the junction, such as the size of the particles and the local fields acting on each particle by the dipoles of the neighboring particles, as well as the temperature and external field. Near T_b the magnetic moment directions of the nanoparticles in the dominating junction become unstable over the experimental time scale, and θ (the angle between particle moments, see Figure 2b) may vary over time. Within a certain temperature range, magnetization jumps may occur between two energy minima, which will cause the MR and the current to fluctuate between two levels within this array domain. The frequency range in such experiments can be quite broad, from a high frequency limit dependent on the junction

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RC (R = resistance and C = capacitance) characteristics (typically between kHz and MHz) down to mHz and below. Frequency analysis of a telegraphic current noise pattern should have a Lorentzian line shape in the frequency domain, which is characteristic of the dynamics of transitions between two discrete energy levels. Lorentzian noise was observed in TMR junctions made of bulk electrodes,[30] as well as cobalt nanoparticle arrays.[31]

Telegraphic current noise was observed in magnetite nanocrystal arrays cooled to around T_b (Figure 2d).[32] At temperatures far from T_b (~230 K), the current noise (at constant bias voltage) looked random, and its amplitude diminished relative to the current fluctuations around T_b. Around T_b, the noise pattern was often discrete and telegraphic (two level). In such cases, the frequency response fitted a Lorentzian line shape. Application of a moderate magnetic field diminishes the telegraphic noise, similar to the effect of reducing the temperature. In isolated uniaxial magnetic particles,[33] the potential well asymmetry across the anisotropy axis increases with applied magnetic field, and the switching rate is reduced.

Current fluctuations in variable temperature scanning probe tunnel junctions

The best resolution possible for local electron transport probing of nanostructures is by scanning probe techniques. Spin-polarized scanning tunneling microscopy (SP STM) is the magnetic microscopy technique with the highest spatial resolution (single atoms) and has been widely applied to epitaxial atomic layers of ferromagnetic materials.[34] It relies on a ferromagnetic (or antiferromagnetic) tip emitting spin-polarized electrons, which have different tunneling probabilities to regions with different magnetization orientations. In these regions, the spin-dependent density of states has spatial variation. This is expressed as small height differences between domains of different magnetization orientations. SP STM has provided magnetization maps with atomic resolution for atomic layer islands of ferromagnetic[35,36] and antiferromagnetic materials.[37] But, it does not work for colloidal magnetic nanoparticle films deposited over conducting substrates due to the large variations in the tunnel resistance (R_S) between the particles and the substrate that mask the tunneling rate differences arising from varying particle moment orientations.

However, non-SP STM can also probe local magnetization dynamics in close-packed arrays of magnetic nanoparticles if the tip emits unpolarized electrons, but their paths cross at least two magnetic nanocrystals, as shown in **Figure 3**a. When the temperature is tuned to ~T_b, where magnetization switching slows down to frequencies suitable for standard STM electronics (up to ~100 kHz), and if at least one of the particles switches magnetization with respect to the other particles along the electron path, then changes in the inter-particle tunnel rate within the experimental time scale should occur. The spin-dependent tunnel resistance fluctuations must be significant relative to the total tunnel resistance determining the STM current. This dictates that the tip must be very close to the top particle (tip-nanoparticle tunneling resistance, R_{gap}, is minimal, typically <1 Å) and the nanoparticle-substrate resistance (R_S) is relatively small (ideally not more than several MΩ). Alternatively, a conductive atomic force microscope (AFM) tip may be used as the top contact. This was recently done in a related experiment, where spin-torque transfer in small

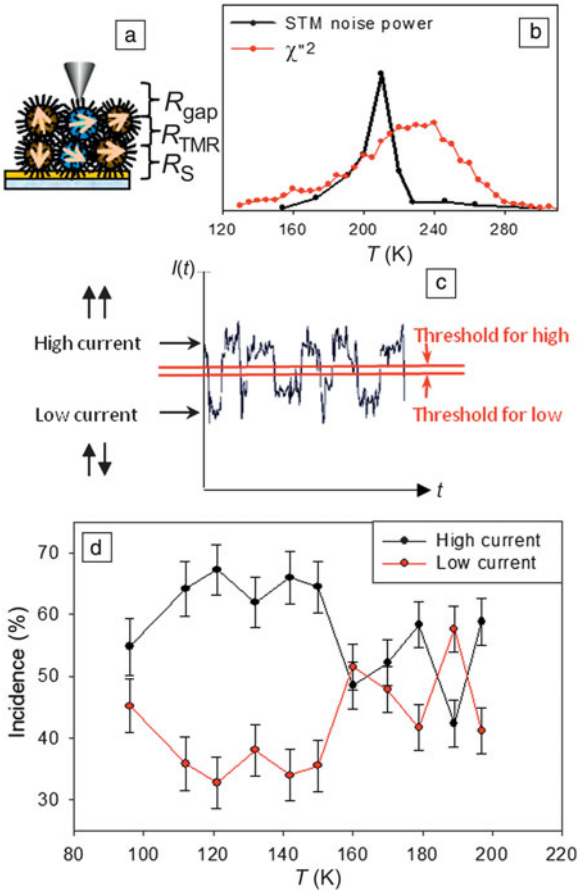


Figure 3. (a) The (non-superparamagnetic) scanning tunneling microscopy (STM) setup that enables local measurements of magnetization dynamics around the blocking temperature, T_b, where R_{TMR}, inter-nanoparticle tunneling resistance, is a significant part of the total tunnel resistance (R_{gap}+R_{TMR}+R_S), where R_{gap} is the tip-nanoparticle tunneling resistance, and R_S is nanoparticle-substrate tunneling resistance. (b) A comparison between the square of the AC magnetic susceptibility data (χ''^2) to the STM current power noise spectral density plotted as a function of temperature (T). Both measured on the same close-packed magnetite nanocrystal array at 100 Hz. (c) A sample of a current versus time ($I(t)$) curve measured near T_b over the magnetite nanocrystal array. The plot displays telegraph noise together with thresholds set for data binarization. (d) The temperature-dependent relative incidence of high- and low-current states measured on the magnetite nanoparticle array. Each data point is the average of hundreds of curves. The experimental data presented demonstrate that the temperature-dependent magnetization dynamics of single (or few) magnetic nanoparticles can be measured using regular variable temperature STM, and new physical information could be deduced from the analysis of the telegraph noise patterns. Reproduced with permission from Reference 41. © 2009 American Physical Society.

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magnetic multilayer pillars was induced by passing current from an AFM tip.[38] Current induced magnetization switching in atomic monolayer islands of Fe atoms was also demonstrated using SP STM,[39] as well as thermal switching of such non-interacting islands.[40]

Once the above resistance conditions are met, the tunnel current fluctuations can reflect magnetization switching events at the location where the STM tip lands. Indeed, recent experiments with magnetite nanoparticle films have shown that when 2–3 nanoparticle thick layers are measured close to T_b, local tunnel current fluctuations larger than the baseline current noise can be observed, and this noise peaks very close to the AC susceptibility peak for the whole nanoparticle film (Figure 3b).[41] This noise in the tunnel current often appears as a discrete bi- (telegraphic) or multi-level current pattern (Figure 3c), which can be analyzed using various statistical methods, such as finding up- and down-level lifetime distributions or calculating their time autocorrelation function.[41,42] Where the $I(t)$ curves exhibited two-level noise patterns, an algorithm was developed to identify the two levels and count the total time spent by the system at the low and high current levels. A highly simplified view may regard the high current level as the particular switching particle being at relatively parallel magnetization orientation with respect to the neighboring particle(s) and the low level as being at a relatively anti-parallel orientation. The plot of the relative times spent at low and high current levels as a function of temperature (Figure 3d) can be roughly divided into two regions with a "critical" temperature, T_c' which seems to occur about 50–60 K below the T_b, identified from the peak at the susceptibility measurements. Above this T_c', high and low current levels occur at roughly equal probabilities, while below this temperature, the high current level occurs with significantly higher probability (60–70%). At the low temperature range, there is also a finite remanent magnetization. Those two effects may signify the formation of an SFM state.

Summary and outlook

The preliminary results on nanoscale current fluctuations observed in close-packed magnetite nanoparticle arrays presented previously demonstrate the large potential of temperature and field-dependent local magneto-transport measurements as a tool to study the magnetization dynamics for small clusters of magnetic nanoparticles with strong dipolar interactions. Higher signal-to-noise ratios and a broader frequency range would enable deeper statistical analysis of the current fluctuations and the time scales of the magnetization fluctuations. This could lead to unambiguous identification of phases such as super-ferromagnetic or super-spin-glass and perhaps to the formulation of more accurate functional forms describing the dependence of the magnetization relaxation times on temperature near the superparamagnet-to-super-spin-glass or the superparamagnet-to-superferromagnet transitions.

Recent advances in synthesis technology to produce uniform close-packed arrays of colloidal nanocrystals of different types and, in particular, making use of new methods developed to enhance interparticle electronic coupling in semiconductor quantum dot arrays, may lead to enhanced magnetoresistance values in magnetic nanoparticle arrays, also at room temperature. This may open new horizons for application of these systems in printed electronics, such as printed magnetoresistive sensors.

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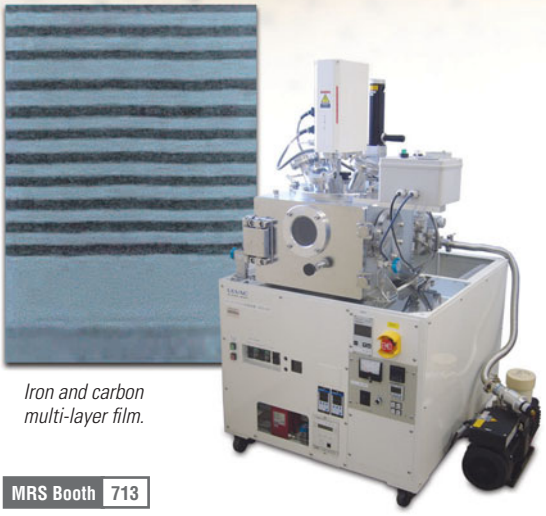
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During your year as a Fellow you will:

- be placed in a specific congressional office to serve as an advisor on science-related issues
- contribute widely to the effective use of materials science knowledge in government
- broaden awareness about the value of scientist- and engineer-government interaction among society members and within government
- network with more than 250 other participants of science and technology fellowship programs
- have significant freedom to follow specific topics and issues that interest you

The MRS Congressional Science and Engineering Fellowship Program is an invaluable experience, but don't just take our word for it. Our past Congressional Fellows explain it best!

"Academia taught me how to think, but the MRS Congressional Fellowship taught me how to get things done. Never have I had such leverage, such opportunities to comingle with dignitaries, to structure agreements and broker deals, as I did in that year. I learned how to navigate past armies of secretaries shielding a VIP, enlist military support for a project, take a rough idea and make it law, to fashion an event into a sound bite and then watch it propagate across the news. I learned to take data and present it in such a way that it gravitated, almost of its own accord, all the way up to the Vice President of the United States. These are skills anyone, who is going anywhere, can use."

Merrilea Mayo Founder, Mayo Enterprises, LLC.
MRS Congressional Fellow 1998-1999
Office of Senator Lieberman

"At the end of the fellowship year I found that I was enjoying "doing" science policy more than just teaching about it, and I ended up staying on in Rep. Honda's office as a member of the staff where I have remained for over a decade. I would not have had that opportunity without the Congressional Fellowship. I encourage anyone who wonders about how federal policies are developed or wants to have a greater role in that process to apply to be a Congressional Fellow."

Eric Werwa Legislative Director, Congressman Mike Honda
MRS Congressional Fellow 2001-2002
Office of Congressman Mike Honda

MRS To learn more about the MRS Congressional Science and Engineering Fellowship Program and how you can apply, visit **www.mrs.org/congressional-fellows**.

Applications for the 2014-2015 MRS Congressional Science and Engineering Fellowship Program are now being accepted. **Deadline for submission is January 3, 2014.**

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