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Tailoring interfacial exchange coupling with low-energy ion beam bombardment: Tuning the interface roughness

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By ascertaining NiO surface roughness in a Ni80Fe20/NiO film system, we were able to correlate the effects of altered interface roughness from low-energy ion-beam bombardment of the NiO layer and the different thermal instabilities in the NiO nanocrystallites. From experiment and by modelling the temperature dependence of the exchange bias field and coercivity, we have found that reducing the interface roughness and changing the interface texture from an irregular to striped conformation enhanced the exchange coupling strength. Our results were in good agreement with recent simulations using the domain state model that incorporated interface mixing.

Crystallite sizes in antiferromagnetic/ferromagnetic-based thin films and interface roughness between layers are considered to be the two properties that dominate interlayer exchange coupling.1,2 Roughness at the interface results typically in a topologically disordered spin configuration that can alter the antiferromagnetic domain arrangement, so that the exchange coupling strength increases with roughness, typically. Crystallite sizes can affect the magnetism of antiferromagnet and ferromagnet since even if the ordering temperatures may be higher than the temperatures where exchange coupling is present, small (usually nanoscaled) crystallite sizes suffer thermal instability where the effective energy barrier again thermally activated moment reversals is surpassed.

To help answer the question whether crystallite size or interface roughness is a dominate factor in the strength of the exchange bias field since both influence the interface spin configuration and number of uncompensated spins, we have studied how both parameters alter the exchange bias magnetism in a series of Ni80Fe20/NiO nanocrystallite-based thin films. The crystallite size and NiO surface roughness was altered by ion-beam bombardment before being capped with a Ni80Fe20 layer. NiO is a nonstoichiometric oxide3 and has a face-centered cubic structure with \( a = 4.195 \) Å and a Neél temperature of \( \approx 520 \) K (Ref. 4) with magnetocrystalline anisotropy \( K = 5 \times 10^8 \) erg/cm\(^3\). Exchange coupled thin films with this antiferromagnetic transition metal oxide have presented the complete range of exchange biased behavior, from thermally activated self-alignment of exchange coupling5 that was dominated by the NiO crystallite size to a distribution of exchange-coupled paths at the interface from both nanoscale NiO crystallite size and different interfacial roughnesses.1,6

The Ni80Fe20/NiO films’ temperature dependent exchange coupled magnetism exhibited the effects of different NiO crystallite size and interface roughness. By mapping NiO surface roughness using atomic force microscopy, we were able to correlate the interface roughness to the temperatures where NiO nanocrystallites underwent thermally activated fluctuations when the NiO film was coupled to Ni80Fe20 and the temperature dependence of the exchange bias fields and coercivities. By accounting for the energetics responsible for exchange coupling, we found that interfacial roughness dominated the exchange coupling magnetism and that the exchange bias field and coercivity tracked in a non-monotonic fashion with temperature and interface roughness. This was unlike the typically observed monotonic decrease of the exchange coupling strength with interface roughness1,2 that may result from not accounting for the effects of different thermal instabilities in the antiferromagnetic nanocrystallites (grains) occurring with altered interface roughness. Our results are in agreement with recent simulations using the domain state model incorporating interface mixing.7 In addition, we have found that reducing the interface roughness and changing the interface texture from a random to striped texture enhances the exchange coupling strength considerably.

A dual-ion-beam deposition technique was used to prepare the Ni80Fe20/NiO bilayers.8 A Kaufman source was used to focus an argon ion beam onto Ni80Fe20 (at. %) or Ni target surfaces, while an End-Hall source was used to clean the substrate or in situ bombard during the deposition of the bottom NiO layer with a fixed end-hall voltage \( V_{EH} \) of 70 V that ensured a constant ion-beam bombardment energy. The O2/Ar ratio in the end-hall ion source was kept at 16% to ensure NiO formation. No external field was applied during deposition. After each NiO deposition, the surface of the film was Ar-ion bombarded for 5 min using \( V_{EH} = 0 \), 70, 100, 130, and 150 V to alter the surface microstructure (roughness). A set of NiO film layers was made without a Ni80Fe20 layer to permit microstructural characterization of what became the interface layer.

A JEOL JEM-2010 transmission electron microscope (TEM) operating at 200 kV was used for microstructural analysis and a NT-MDT Solver Pro-M atomic force microscope (AFM) was used to characterize the NiO surface. X-ray diffraction (XRD) patterns were collected using a

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\( a_1 \)
Brüker D8 Discover. The results of these measurements are summarized in Fig. 1. The NiO film layers had crystallite sizes between 5 and 20 nm in diameter that penetrated the 16 nm film thickness (Fig. 1(c)) and had a rock-salt structures with \( a = 4.21 \text{Å} \) as determined from the electron diffraction patterns (e.g., (111) and (200) reflections). Figs. 1(a) and 1(b) show the reduction in crystallite size with increasing ion bombardment energy, and this trend was also described by the results of Scherrer analysis of the NiO XRD patterns (Fig. 1(d)). The Ni\(_{80}\)Fe\(_{20}\) microstructure remained unchanged by the results of Scherrer analysis of the NiO XRD patterns (e.g., (111) and (200) reflections). Figs. 1(a) and 1(b) show the reduction in crystallite size with increasing ion bombardment energy. The microstructural results indicated that while the interface texture would change with ion bombardment temperature AFM measurements (Fig. 1(b)) present how the interface texture would change with ion bombardment energy. The microstructural results indicated that while the average NiO crystallite size changed from 13 nm to 11 nm when altering the surface with ion-beam bombardment, increasing bombardment energy (\( V_{\text{EH}} = 70–150 \text{ V} \)) did not change the average NiO crystallite size. However, AFM measurements revealed a clear evolution of the surface roughness, \( \sigma \) with \( V_{\text{EH}} \), where \( \sigma = 0.61 \pm 0.1 \text{ nm} \) for \( V_{\text{EH}} = 0 \) down to \( \sigma = 0.42 \pm 0.2 \text{ nm} \) for \( V_{\text{EH}} = 130 \text{ V} \), in agreement with similar films.\(^7\) With the most energetic surface bombardment, \( V_{\text{EH}} = 150 \text{ V} \), a further reduction of surface roughness was achieved (\( \sigma = 0.37 \pm 0.2 \text{ nm} \)) and a change in texture from the previously irregular texture to a stripe-like roughness (Fig. 1(b)) occurred.

The exchange coupling between the 16 nm thick NiO and the 9 nm thick Ni\(_{80}\)Fe\(_{20}\) layer (Fig. 1(c)) was evident in the temperature dependence of the zero field-cooled (ZFC) and field-cooled (FC) DC susceptibility (\( M \) vs \( T \)) scans measured with a Quantum Design MPMS magnetometer. While the 9 nm thick Ni\(_{80}\)Fe\(_{20}\) film had identical ZFC and FC \( M \) vs \( T \) with 100 Oe (i.e., \( \Delta M_{\text{FC–ZFC}}(T) \) = 0, not shown), coupling the Ni\(_{80}\)Fe\(_{20}\) resulted in a clear divergence between \( M_{\text{ZFC}}(T) \) and \( M_{\text{FC}}(T) \) as demonstrated by \( \Delta M_{\text{FC–ZFC}}(T) \) in Fig. 2. For example, the Ni\(_{80}\)Fe\(_{20}/\)NiO \( V_{\text{EH}} = 0 \) film that had the largest NiO crystallites (\(~13 \text{ nm}\) ) showed a divergence between the ZFC and FC \( M \) vs \( T \) scans, with a gradual, monotonic increase of \( \Delta M_{\text{FC–ZFC}}(T) \) from 10 to 400 K, followed by a constant \( M_{\text{FC}}(T) \). Since the main part of the NiO layer crystallites were composed of ferromagnetic sheets with opposing magnetization directions\(^6\) so that only NiO near the Ni\(_{80}\)Fe\(_{20}\) layer should have been coupled (with the NiO layer likely aligned with the Ni\(_{80}\)Fe\(_{20}\) layer\(^1\)). \( \Delta M_{\text{FC–ZFC}}(T) \) = 0 above 360 K identified when the exchange coupling to the NiO was no longer present (the remaining magnetization above that temperature was from the Ni\(_{80}\)Fe\(_{20}\) layer), establishing the exchange-bias blocking temperature \( T_B = 360 \pm 5 \text{ K} \) (in agreement with field-cooled hysteresis loop measurements described below). When the NiO interface was ion-beam bombarded with \( V_{\text{EH}} = 70 \text{ V} \), we found a small change in \( T_B \) that was the result of the decreased NiO crystallite size and small decrease in interfacial roughness, \( \sigma \) from 0.61 \pm 0.03 nm to 0.56 \pm 0.02 nm for \( V_{\text{EH}} = 0 \) to 70 V, respectively. However, with the continued decrease in interfacial roughness while NiO crystallite size remained effectively unchanged, \( T_B \) continued to decrease with decreasing \( \sigma \), as shown by the impact of increasing \( V_{\text{EH}} \) upon \( T_B \) determined from \( \Delta M_{\text{FC–ZFC}}(T_B) \) in Fig. 2(a). The linear relationship...
of the domains in the NiO, we fit the ferromagnetic/antiferromagnetic coupling. Assuming the films, indicated that thermal fluctuations were dominant and the exchange bias loop shift, \( H_{\text{ex}} \) (Fig. 2(b)). The exchange coupled NiO layer permitted an effective interfacial magnetization that in combination with the nanoscaled crystallines formed AF domains, that is, an effective domain state configuration. The hysteresis loops at 10 K (where the effective exchange energy changes between films were weakest at \( T \ll T_B \) and \( H_{\text{ex}} \neq 0 \)) shown in Fig. 2(b) exhibited the effects of interfacial roughness on coupling. In agreement with simulations of the effects of interfacial roughness on a hysteresis loop, with decreasing \( \sigma \) the loops narrowed and became squarer as the distribution of switching fields around the interface narrowed with interactions likely shifting from antiferromagnetically to ferromagnetically dominated. That is, the inherently random mixing of ferromagnetic and antiferromagnetic spins at the Ni_{80}Fe_{20}/NiO interface resulted in distributions of magnetized areas (domains) whose directions were either with or against the direction of the applied field, resulting in nucleation centres or pinning sites that provided the distribution of switching fields.

The temperature dependence of the coercivity, \( H_c(T) \), and the exchange bias loop shift, \( H_{\text{ex}}(T) \), for the films is presented in Fig. 3. The temperatures where \( H_c(T) \) and \( H_{\text{ex}}(T) \) decreased to zero were in good agreement with the fit results for \( T_B \) of Fig. 2(a). The straggling thermal aftereffect fluctuation model using similar parameters to those in Ref. 13, assuming that this coupling was driven by the temperature dependence of the domains in the NiO, we fit \( H_c(T) \) and \( H_{\text{ex}}(T) \) using the thermal aftereffect fluctuation model using similar parameters to those in Ref. 14. The fits (solid lines in Fig. 3) used \( M_s(0) = 930 \text{emu/cm}^3 \) to describe the Ni_{80}Fe_{20} layer, freezing temperatures (\( T_f \equiv T_B \)) were set from the ZFC/FC \( M \) vs \( T \) scans described above. The nanoscale sizes of the Ni_{80}Fe_{20} and NiO crystallites, the temperature dependence of the ZFC/FC low field magnetization, and by inspection of Fig. 3, we see that \( H_c(T) \propto \sqrt{T} \) for all the films, indicated that thermal fluctuations were dominantly the ferromagnetic/antiferromagnetic coupling. Assuming that this coupling was driven by the temperature dependence of the domains in the NiO, we fit \( H_c(T) \) and \( H_{\text{ex}}(T) \) using the thermal aftereffect fluctuation model using similar parameters to those in Ref. 14. The fits (solid lines in Fig. 3) used \( M_s(0) = 930 \text{emu/cm}^3 \) to describe the Ni_{80}Fe_{20} layer, freezing temperatures (\( T_f \equiv T_B \)) were set from the ZFC/FC \( M \) vs \( T \) scans, \( K_{\text{NiO}} = 5 \times 10^5 \text{erg/cm}^3 \), and a Gaussian distribution of crystallites with average sizes set by the TEM and XRD results (Figs. 1(a) and 1(d)). The crystallite distribution’s standard deviation was fitted and remained essentially unchanged for all films (0.34 ± 0.05). A measure of the interface coupling energy, \( J_{\text{int}} \), was provided by the fits, and its dependence on the interface roughness, \( \sigma \), is presented in the top panel of Fig. 4, with values in agreement with previous results on NiO-based exchange biased systems.

As the interface becomes smoother, we observed that the interfacial coupling energy became smaller, behaviour that was mirrored by \( T_B \) decrease with smaller \( \sigma \) (Fig. 2) combined with \( H_{\text{ex}}(T) \) for the different \( V_{\text{EH}} \) (or \( \sigma \)) films shown in Fig. 3. The fit results for \( J_{\text{int}} \) were consistent with enhanced antiferromagnetic–ferromagnetic coupling with increasing roughness. That is, it seemed that as the number of isolated, interfacial Ni or Fe spins that were positively exchange coupled which were surrounded by superexchange, negatively coupled Ni from the NiO increased with roughness, and clusters of like spins began to form that could no longer remain pinned, were less frustrated, and began to rotate with the applied field and fluctuate at lower temperatures (e.g., shown by \( T_B \) vs \( \sigma \)). The intermixing of positive and negative exchange coupling between Ni_{80}Fe_{20} and NiO around the interface could be the other way around since there was no observable vertical shift of the hysteresis loops (i.e., \( \Delta M_s = 0 \)) that would help identify the ferromagnetic (positive) or antiferromagnetic (negative) nature of the interfacial exchange.16,17 We believe that the subsequent increase of \( J_{\text{int}} \) at the smallest \( \sigma \approx 0.37 \text{nm} \) is likely due to the change in interface structure from being effectively isotropic and random to stripe-like (Fig. 1). Recent Monte Carlo simulations have shown similar changes of \( J_{\text{int}} \) with interface configuration.18

Since it was \( J_{\text{int}} \) that set essentially the \( H_{\text{ex}} \) thermodynamics, examining the variation of \( H_{\text{ex}} \) scaled by \( J_{\text{int}} \) with \( \sigma \) permitted the effects of interface mixing to be tracked, presented in the bottom panel of Fig. 4. In agreement with the domain-state description of exchange bias, we found a maximum in \( H_{\text{ex}} \) with respect to interface roughness (or mixing of ferromagnetic and antiferromagnetic spins), behaviour that remained robust across all the measured temperatures where exchange coupling was present (i.e., scaling with \( \sigma = H_{\text{ex}}(T)/H_{\text{ex}}(10K) \)). With increased \( \sigma \) from \( \approx 0.37 \text{nm} \), the low values of interface roughness that permitted an optimization of the exchange bias with enhanced coupling as the interface layer spins became more concentrated with increasing roughness, enhancement was weakened (apparently only peripherally affected by interface confirmation or

![Graph showing temperature dependence of coercivity and exchange bias.](https://example.com/fig3.png)

**Fig. 3.** Temperature dependence of the coercivity, \( H_c(T) \), and exchange bias loop shift, \( H_{\text{ex}}(T) \), for the Ni_{80}Fe_{20}/NiO films with different interface roughness (or \( V_{\text{EH}} \)). The solid lines are to fits described.
crystallite size) as groups of ferromagnetically coupled spins unpinned since they were no longer being frozen via the exchange coupling to the antiferromagnetic spins. This would have been complimented by collections of antiferromagnetic spins no longer canting with the applied field and decoupling from the hysteresis processes (e.g., domain wall pinning, reversal and wall motion). In addition, we have found that reducing the interface roughness and changing the interface texture from an irregular to striped configuration enhanced the exchange coupling.

In summary, we have found that for the a Ni$_{80}$Fe$_{20}$/NiO film system interfacial roughness dominated the exchange coupling magnetism and that the exchange bias field and coercivity tracked in a non-monotonic fashion. By accounting for the effects of different thermal instabilities in the antiferromagnetic nanocrystallites and changes in the interface roughness, our results are in agreement with recent simulations using the domain state model incorporating interface mixing. In addition, we have found that reducing the interface roughness and changing the interface texture from an irregular to striped configuration enhanced the exchange coupling.

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