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Epitaxial spin-valve structures for ultra-low-field detection

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A new epitaxial "spin-valve"-type system for low-field magnetoresistive detection is described. This system is based on Fe/Pd epitaxial multilayers grown on (100)MgO by MBE. These films show a very abrupt transition from positive to negative magnetization as the reverse field is applied during hysteresis measurements. We have used these sensitive magnetic properties to fabricate epitaxial spin-valve structures by epitaxial growth of Fe/Ag, Co/Ag, or Co/Cu bilayers on top of a Fe/Pd bilayer. Hysteresis loops and magnetoresistance curves clearly indicate a significant field range with antiparallel alignment of the two components. Magnetoresistive sensitivities of up to 0.3% per Oe at low temperatures have been observed in these structures. The efficiency of the spin-dependent scattering has subsequently been improved either through the addition of planar Co impurities, in both the soft and hard magnetic layer, or by increasing the number of active Fe/Pd interfaces. This approach leads to a drastic improvement of the sensitivity, up to 1.5% per Oersted at room temperature.

Sensitive magnetic field sensors, based on the magnetoresistive effect, require a large magnetoresistivity at low fields. The discovery of giant magnetoresistance (GMR) in antiferromagnetically exchange-coupled multilayers has opened new avenues for these applications.[1] The principal problem with coupled GMR systems is related to the magnetic field range of the effect, which is usually of 1 kOe or more. To decrease the field scale of GMR, the most promising approach is based on achieving antiparallel alignment *without* antiferromagnetic exchange coupling. This can be obtained by selective rotation of the magnetic layers, using various types of layers with different magnetic properties. For example, a magnetic multilayer can be grown using alternatively magnetically hard and magnetically soft layers.[2] Another method is to block one of the magnetic layers through an exchange coupling with an antiferromagnetic material.[3] In practice, permalloy ($Ni_{79}Fe_{21}$) is often used as the soft material, and the growth of such materials is usually done by sputtering and leads to a polycrystalline structure. These systems, often referred to as a "spin-valve" structures, have led to sensitivities higher than 1% per Oersted[4] at room temperature. Here we propose an alternative system, which makes use of the sensitive magnetic properties of epitaxial Fe/Pd bilayers grown by molecular beam epitaxy (MBE). In epitaxial Fe/Pd superlattices, complete rotation of the magnetization of Fe occurs on very small magnetic field scales ($\approx$1 Oe). By combining a Fe/Pd bilayer with other epitaxial bilayers such as Fe/Ag or Co/Cu in a "spin-valve" structure, we have obtained highly field-sensitive epitaxial multilayers.

The present samples were grown in the same manner as the (100)Fe/Pd epitaxial multilayers studied previously.[5–7] Briefly, we used 1 cm×1 cm (100)MgO substrates, annealed in UHV for 30 min at 450 °C immediately prior to deposition. Fe grows very well on (100)MgO, without any kind of buffer layer. A substrate temperature of $T_S \approx 80$ °C was chosen to yield the best structural properties, as determined by low- and high-angle x-ray diffraction and cross-sectional transmission electron microscopy.[7] Measurements of the magnetic properties of Fe/Pd superlattices show narrow hysteresis loops at all temperatures.[5,6] Although the value of the coercive field H_c depends on the growth parameters such as temperature and deposition rate, and also on the thicknesses of the individual layers, it never exceeds 20 Oe at 15 K, and 5 Oe at 300 K. The Fe layers are ferromagnetically coupled to each other[6] for Pd thicknesses as high as 40 Å. Most importantly, we observe a very abrupt transition from positive to negative magnetization as the reverse field is applied, with a transition width between 1 and 3 Oe. Measurements of the absolute magnetization using wedge-shaped Pd layers have provided evidence of a strong polarization of the Fe/Pd interface,[7] and we have determined that the presence of this polarized interface is directly responsible for the low coercive fields.[8]

Following these results, we have used epitaxial Fe/Pd bilayers to develop new types of epitaxial "spin-valve" systems.[8] For example, we have deposited by MBE an Ag/Fe/Ag trilayer ($Hc \approx 100$ Oe) on top of a Fe/Pd bilayer ($Hc \approx 20$ Oe), resulting in a compound structure of $Fe_{(20\ Å)}/Pd_{(30\ Å)}/Ag_{(30\ Å)}/Fe_{(20\ Å)}/Ag_{(30\ Å)}$. RHEED patterns obtained during deposition indicate a fully epitaxial growth for the entire structure. The hysteresis loop of this structure [Fig. 1(b)] is essentially the sum of the two hysteresis loops obtained for separate $Fe_{(20\ Å)}/Pd_{(30\ Å)}$ and $Fe_{(20\ Å)}/Ag_{(30\ Å)}$ bilayers [Fig. 1(a)]. Thus, the two Fe layers in the structure are uncoupled, structurally as well as magnetically, by the $Pd_{(30\ Å)}/Ag_{(30\ Å)}$ interlayer. Antiparallel alignment of the two Fe layers is therefore achieved after reversal of the first Fe layer. Similar results are obtained as we replace Ag by Cu, and/or the second Fe layer by Co, which is a magnetically harder material. It is worth noting that although the growth proceeds epitaxially throughout the structure, RHEED patterns indicate a degradation of the layer flatness after the second bilayer system (i.e., Fe/Ag or Co/Cu) has been deposited. Therefore, it is not possible to repeat the layer sequence to form a thicker multilayer, since the subsequent

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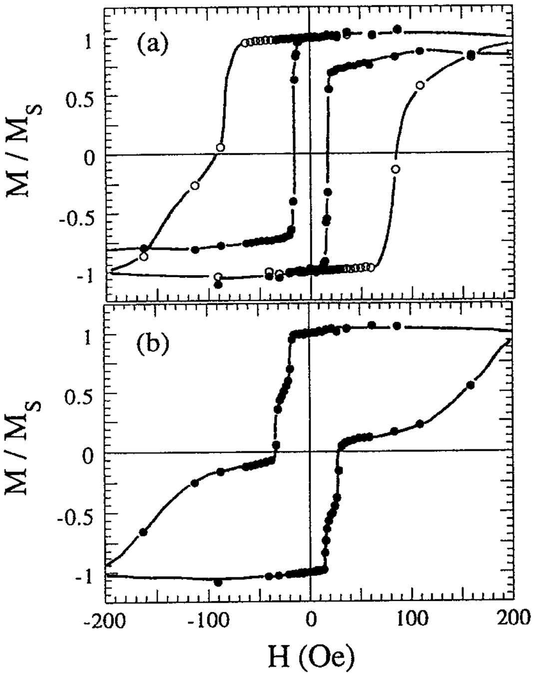

FIG. 1. Magnetic hysteresis loops at $T=15$ K of (a) a $Fe_{(20\ Å)}/Pd_{(30\ Å)}$ bilayer (filled circles) and a $Fe_{(20\ Å)}/Ag_{(30\ Å)}$ bilayer (open circles), and (b) a $Fe_{(20\ Å)}/Pd_{(30\ Å)}/Ag_{(30\ Å)}/Fe_{(20\ Å)}/Ag_{(30\ Å)}$ structure.

Fe/Pd bilayers would not retain the required soft magnetic properties.

The magnetoresistive (MR) response is shown in Fig. 2 for the Fe/Pd/Ag/Fe/Ag structure. The rotation of the magnetization in the first Fe layer, at low fields, induces an abrupt increase of the resistivity when antiparallel alignment is achieved, as is seen in many other multilayer-based GMR systems. The maximum MR response at 4.2 K is 0.8% for Fe/Ag bilayers, 0.5% for Co/Ag bilayers, and 1.5% for Co/Cu bilayers (also shown in Fig. 2), each epitaxially grown on a Fe/Pd bilayer. The magnitude of the MR effect originates in the strength of the spin-dependent impurity scattering that occurs near the magnetic interfaces.[1] Although

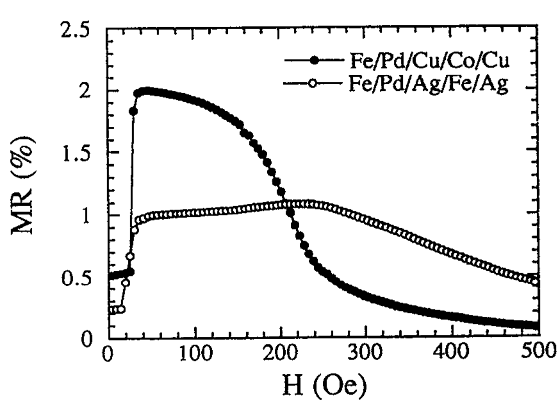

FIG. 2. Magnetoresistance as a function of applied magnetic field at $T=4.2$ K for $Fe_{(20\ Å)}/Pd_{(30\ Å)}/Ag_{(30\ Å)}/Fe_{(20\ Å)}/Ag_{(30\ Å)}$ and $Fe_{(20\ Å)}/Pd_{(30\ Å)}/Cu_{(30\ Å)}/Cu_{(30\ Å)}/Co_{(20\ Å)}/Cu_{(30\ Å)}$ epitaxial structures.

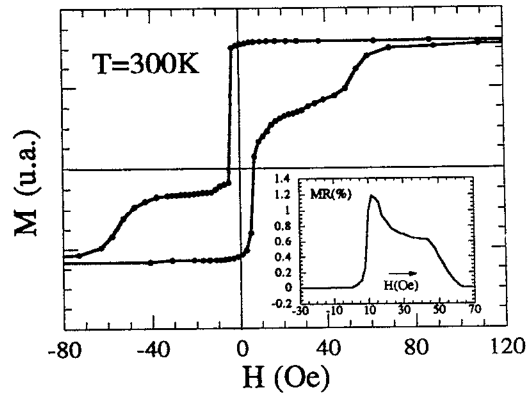

FIG. 3. Magnetic hysteresis loop at $T=300$ K of a $[Fe_{(20\ Å)}/Co_{(4\ Å)}/Pd_{(30\ Å)}]/[Cu_{(30\ Å)}/Co_{(20\ Å)}/Cu_{(30\ Å)}]$ structure. Inset: corresponding magnetoresistive response (shown for an increasing magnetic field).

small total magnetoresistance changes are observed, our interest lies in the small field scales (a few Oe) over which the MR increase is observed (up to 0.3% per Oe). At 300 K, the magnetoresistive effect is strongly decreased. For example, a value between 0.2% and 0.3% is observed for the Fe/Pd/Cu/Co/Cu structure above, depending on the precise value of individual layer thicknesses. Several factors contribute in limiting the maximum MR effect observed. First, the total thickness of the structure is on the order of the electronic mean-free path, therefore the efficiency of scattering within the structure is reduced. Second, unlike the case of exchange-coupled MMM's, "spin-valve" structures rely on spin-dependent scattering from different systems. Hence, in our multilayers, strong spin-dependent scattering must occur *both* within the Fe/Pd layers and within the other magnetic layers in order to achieve the maximum MR effect. Unlike the Co/Cu interface, which is well known to exhibit strongly spin-dependent electron scattering ($\alpha=\rho\uparrow/\rho\downarrow\ll1$), the Fe/Pd interface is not expected to be particularly favorable. Nonetheless, because the field scales over which this MR is observed is actually smaller at room temperature than at 4.2 K, sensitivities close to 0.15% per Oe have still been observed at 300 K.

We have checked several ways to increase the magnetoresistive response of these structures. In particular, we have tried to increase the spin-dependent scattering within the Fe/Pd layers. In a first approach, we have introduced ultrathin layers of impurities (Cr, Cu, or Co) in the Fe layer. Whereas 3 Å of Cr in the Fe layer drastically increases the coercive field, no such adverse effect on the magnetic properties was observed with the use of Cu or Co impurity layers, while the magnetoresistive effect is significantly increased. For example, the introduction of a 4 Å-thick layer of Co in a $[Fe_{(20\ Å)}/Pd_{(30\ Å)}]/[Cu_{(30\ Å)}/Co_{(20\ Å)}/Cu_{(30\ Å)}]$ structure leads to $\Delta R/R$ between 0.7% and 0.8% (a factor of 2 increase). Surprisingly, the effect does not depend on the precise position of the Co layer, i.e., whether it is placed at the Fe/Pd interface (shown in Fig. 3) or 5 Å below the interface. The addition of a second Co layer, resulting in a $[Fe_{(8\ Å)}/Co_{(4\ Å)}/Fe_{(8\ Å)}/Co_{(4\ Å)}/Fe_{(8\ Å)}/Pd_{(30\ Å)}]/$

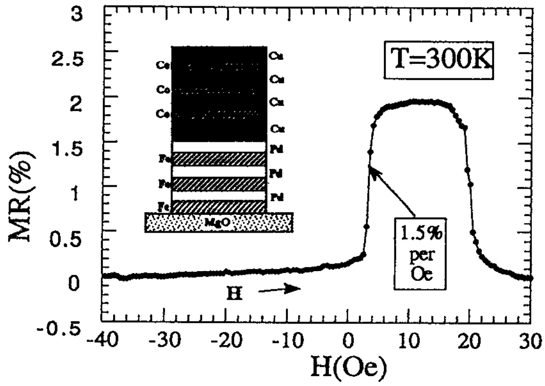

FIG. 4. Magnetoresistive response at $T=300$ K of a $[(Fe/Pd)_3/Pd]/[Cu/(CuCo)_3/Cu]$ structure. Inset: schematic drawing of the multilayer structure.

$[Cu_{(30\ Å)}/Co_{(20\ Å)}/Cu_{(30\ Å)}]$ structure, does not increase the magnetoresistance.

In a second approach, we have simply increased the number of Fe/Pd interfaces by replacing the Fe/Pd layer with a Fe/Pd multilayer. A series of samples was grown with different numbers of Fe/Pd bilayers. It is not trivial to determine precisely the role of the additional Fe/Pd interfaces in these samples, because the quality of the growth in the entire structure may be affected by the respective thicknesses of the first few layers. Moreover, in order to compare the magnitudes of the magnetoresistive responses, we must be sure that the other parameters, such as the structure of the nonmagnetic Pd/Cu layer or the quality of the Cu/Co interface, are identical in each case. Nevertheless, the observed results are quite spectacular, regardless of the small structural differences that may exist. Here we present out results obtained for a series of samples of the form $[Fe/(Fe/Pd)_n/Pd]/[Cu/(Cu/Co)_3/Cu]$, with $n=1,2,3$, or more. One should note that in this structure the soft magnetic layer is formed by a Fe/Pd multilayer, and the hard layer by a Co/Cu multilayer. It has been chosen because it presents a good contrast between the two coercive fields. For $n=1$, as discussed above, we always observed a value of $\Delta R/R$ around 0.4%. This value increases notably for $n=2$ (i.e., three Fe/Pd interfaces) with $\Delta R/R=0.8\%$, and up to $\Delta R/R=1.5\%$ for $n=3$. For a higher number of interfaces the effect appears to saturate. In Fig. 4 we show the best magnetoresistance curve obtained at $T=300$ K for a $[Fe/(Fe/Pd)_3/Pd]/[Cu/(Cu/Co)_3/Cu]$ structure grown directly on (100)MgO. The 2% amplitude leads to a slope at low fields close to 1.5% per Oe.

To verify that the change of $\Delta R/R$ was, in fact, due to the increasing number of the Fe/Pd interfaces, and not simply to the increase in the total Fe thickness, we have also studied a structure with a wedge-shaped Fe layer. The structure is $[Fe/Pd_{(50\ Å)}/Fe_{(wedge\ 20-70\ Å)}/Pd][Cu/(Cu/Co)_3/Cu]$. The first Pd layer is made thick, to ensure that the first Fe/Pd interface will not participate in the spin-valve magnetoresistive effect. The result is that we do not observe any increase of $\Delta R/R$ with increasing total Fe thickness. On the contrary, the effect decreases slowly with a downward step at about 45 Å, which may be due to a structural change of the Fe layer at these large thicknesses. The lack of increase in $\Delta R/R$ shows clearly that the varying thickness of the Fe layer does not significantly modify the magnetoresistance, and that the effect described above can be attributed principally to the increasing number of Fe/Pd interfaces.

In conclusion, we have used the soft magnetic properties of epitaxial Fe/Pd bilayers grown on MgO to fabricate epitaxial "spin-valve" structures, which display sensitive magnetoresistive responses to low fields. Hysteresis loops and magnetoresistance curves of the "spin-valve" structures obtained by epitaxial growth of Fe/Ag, Co/Ag, or Co/Cu bilayers on top of a Fe/Pd bilayer, clearly indicate the successive rotation of the two separate magnetic components. Magnetoresistive sensitivities of up to 0.3% per Oe have been obtained at 4.2 K. By increasing the number of active Fe/Pd layers, we have notably increased the magnetoresistive effect of the structures, and obtained sensitivities of up to 1.5% per Oe at 300 K.

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