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1992 J. Phys.: Condens. Matter 4 L519

(http://iopscience.iop.org/0953-8984/4/40/004)

J. Phys.: Condens. Matter 4 (1992) L519–L524. Printed in the UK

LETTER TO THE EDITOR

Magnetic properties of Fe/Ag nano-multilayers

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Received 14 July 1992

Abstract. Fe/Ag multilayers were prepared by alternate vapour deposition. The microstructure, periodicity, thickness, chemical composition and the magnetic moment of the films were determined and measured by various methods. The magnetic moment per Fe atom in Fe/Ag multilayers was considerably enhanced with decreasing Fe layer thickness, and it was up to 2.60 μ_B at an Fe layer thickness of 1.2 nm. The enhancement may be attributed to the non-equilibrium Fe atomic configuration at the Fe/Ag interface, i.e. probably the epitaxial growth of the Fe layer on the Ag layer with an orientation relationship of $(001)_{Fe} \parallel (001)_{Ag}$, $[100]_{Fe} \parallel [110]_{Ag}$.

In the last few years, magnetic multilayered films on the nanometre scale with artificial periodicity have attracted much attention because they may exhibit anomalous magnetic properties. Among the various combinations of the elements, the Fe–Ag system, because of its immiscibility and because there are no intermetallic compounds between Fe and Ag, has aroused considerable interest [1–3]. Freeman and Fu *et al* [4,5] predicted that there would be strongly enhanced two-dimensional (2D) magnetism at surfaces and interfaces in transition metals on noble metals based on an all-electron total-energy local spin-density approach. Krompiewski *et al* [6] also foresaw that the magnetic moment of Fe/Ag multilayers could be up to 2.86 μ_B using a first-principles tight-binding linear muffin-tin orbital method. Although iron/noble metal multilayers have been studied by several authors [7–12], significant enhancement of the magnetic moment has not yet been observed. Recently only a slight enhancement of the magnetic moment in Fe/Ag superlattices and multilayers has been reported [13, 14]. In this letter, we report the changes of magnetic properties in Fe/Ag nano-multilayers and discuss the possibile mechanism of the magnetic moment enhancement.

The Fe/Ag multilayered films were prepared by depositing alternately pure iron (99.99%) and silver (99.99%) at rates of 0.1 to 0.2 nm s^{-1} onto NaCl single-crystal and glass substrates with a thickness of 0.1 mm in an electron-gun evaporation system with a vacuum of 5×10^{-7} Torr. The thickness of the silver layer was fixed at 9 nm, while the iron layer varied from 1.2 to 12 nm controlled by a quartz oscillator *in situ* in the system. The total thickness of the films was approximately 140 nm. The samples were analysed by transmission electron microscopy (TEM), selected-area electron diffraction (SAD) and electron microprobe to identify the structure and confirm the composition

0953-8984/92/400519+06$07.50

of the films. Rutherford backscattering (RBS) was also employed to measure the thickness and the periodicity of the samples. The magnetic properties were measured with a vibrating sample magnetometer (VSM) under a magnetic field of 4 kOe at room temperature. The size of the VSM samples was 5 mm × 5 mm. To reduce measuring error, four identical specimens were put together in one measurement to obtain the hysteresis loops. After measuring the magnetic properties, the alloy films were dissolved in 5 ml nitric acid (HNO_3), and a PLASMA-SPEC-I inductively coupled plasma atomic emission spectrum (ICP) was employed to determine the Fe content in the multilayers. An average magnetic moment per Fe atom was then calculated by using the above data.

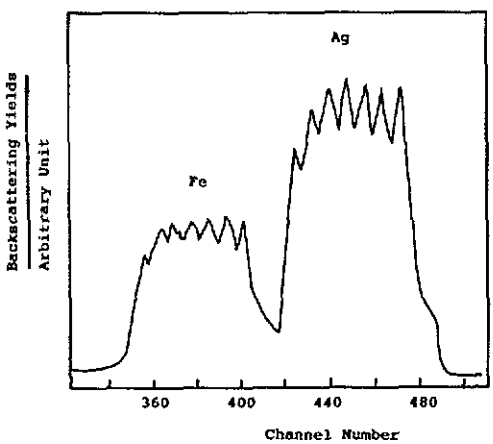

Figure 1. RBS spectrum of an $[Fe(11\ nm)/Ag(9\ nm)]_7$ film.

The average atomic concentration of all samples was determined by electron microprobe analysis, and the thickness and the periodicity of the films measured by RBS. The experimental results indicated that the composition, thickness and periodicity of all samples agreed with the designed ones. Figure 1 shows a Rutherford backscattering spectrum of an $[Fe(11\ nm)/Ag(9\ nm)]_7$ sample. The spectrum was obtained with 2.1 MeV He^+ ions. From the spectrum, one can see that the peak corresponding to the high channel is the peak of silver and the low channel is the peak of iron, and both the silver and iron spectra consisted of seven peaks, indicating the multilayered films to be of seven layers of silver and iron alternately. The total thickness of the sample was about 142 nm and the thickness of each layer of silver and iron was about 9.1 nm and 11.2 nm, respectively.

The magnetic properties of Fe/Ag multilayers were measured by a VSM with the precision of 2×10^{-6} emu. The VSM results indicated that all samples of the Fe/Ag multilayers in our experiment have the in-plane easy axis of magnetization. Firstly, the hysteresis loop of the substrate and holder was measured; the largest magnetic moment of the substrate and holder was about 4×10^{-4} emu, which was about 1–2 orders of magnitude lower than that of the Fe/Ag multilayers. We then measured the hysteresis loops of Fe/Ag multilayers, and the magnetization of the substrate and holder was deducted automatically by the computer during the measurement of the Fe/Ag multilayered films. Figure 2 shows the hysteresis loops of four Fe/Ag

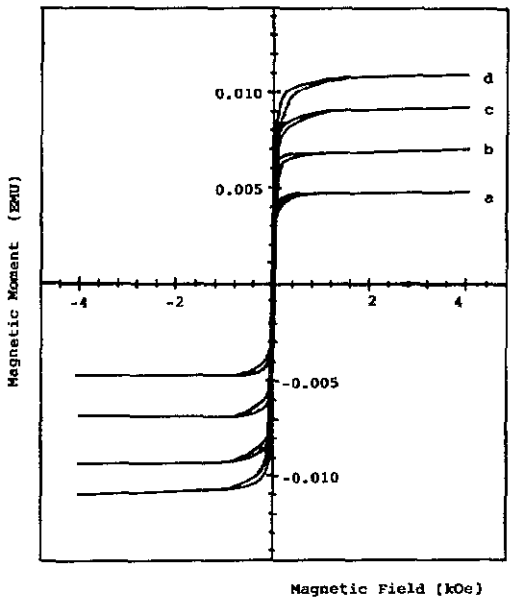

Figure 2. The hysteresis loops of Fe/Ag multilayers: (*a*) Fe(1.2 nm)/Ag(9 nm), (*b*) Fe(2.5 nm)/Ag(9 nm), (*c*) Fe(3.5 nm)/Ag(9 nm) and (*d*) Fe(7.8 nm)/Ag(9 nm).

Table 1. The magnetic moment per Fe atom in different Fe/Ag multilayers.

Specimen	σ_s (10^{-3} emu)	Weight of Fe (μg)	σ (μ_B)
Fe(1.2 nm)/Ag(9 nm)	4.735	17.95	2.60
Fe(2.5 nm)/Ag(9 nm)	7.075	28.85	2.42
Fe(3.5 nm)/Ag(9 nm)	9.350	39.39	2.35
Fe(5.6 nm)/Ag(9 nm)	11.350	46.65	2.40
Fe(7.8 nm)/Ag(9 nm)	11.100	47.15	2.32
Fe(11 nm)/Ag(9 nm)	14.400	62.00	2.29
Bulk Fe			2.15

multilayers under a magnetic field of 4 kOe, which was parallel to the film plane. Table 1 and figure 3 show the magnetic moment per Fe atom in Fe/Ag multilayered films. Apparently, the interference of the magnetic moment from the substrate and holder has negligible effect on the measured values of the Fe/Ag multilayers. Taking into account that the error involved in ICP measurement is 5%, the total measured error was lower than 6%. From figure 3, it can be observed that the magnetic moment per Fe atom in Fe/Ag multilayers was about the same as in the bulk Fe when the Fe layer thickness was about 11 nm, and that the magnetic moment per Fe atom was considerably enhanced with the decreasing of the Fe layer thickness and reached 2.60 μ_B, at an Fe layer thickness of 1.2 nm, while the Ag layer was fixed at 9 nm.

Figure 4 shows two hysteresis loops of the Fe/Ag multilayers measured with the magnetic field parallel and perpendicular to the film plane, respectively. From the figure it can be found that the saturation magnetization field, perpendicular to the film plane, depended on the thickness of the Fe layer in a varying manner when the Ag

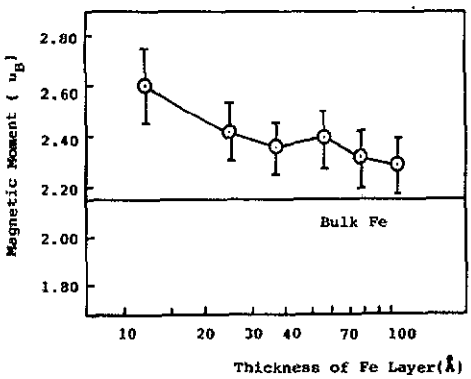

Figure 3. Magnetic moment of Fe/Ag multilayered films as a function of the thickness of the Fe layer.

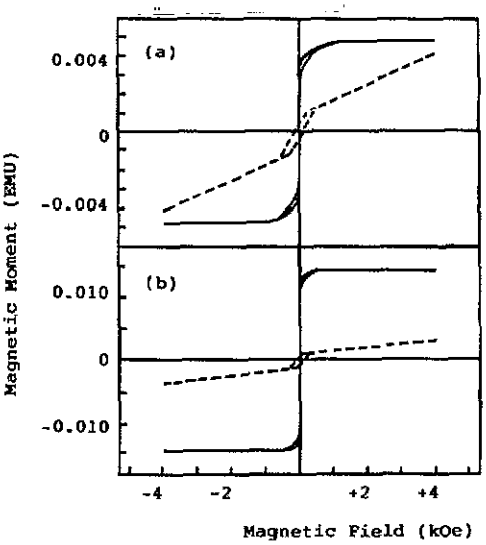

Figure 4. The hysteresis loops measured in a field parallel (solid line) and perpendicular (dotted line) to the film plane for (a) Fe(1.2 nm)/Ag(9 nm) and (b) Fe(11 nm)/Ag(9 nm).

layer was fixed at 9 nm. The saturation magnetization of the Fe(1.2 nm)/Ag(9 nm) multilayers was much easier than that of Fe(11 nm)/Ag(9 nm). It was revealed that as the thickness of the Fe layer decreased, there was an increasing tendency towards perpendicular magnetization in the Fe/Ag multilayers, which was similar to that reported by Gutierrez *et al* [2].

To probe the reason for the magnetic moment enhancement in Fe/Ag multilayers, the microstructure of the films was investigated by means of TEM SAD. Figure 5(a) shows a typical electron diffraction pattern of the Fe(4.5 nm)/Ag(9 nm) multilayers taken at room temperature. From this figure, it can be found that the diffraction rings of the sample were discontinuous, indicating that there were texture and some specific orientation relationships emerging in the films. It was found by routine calculation that the (001) plane of most of the silver grains was parallel to the film plane and that the (011) or (013) planes of the other silver grains was parallel to the film plane: while for the iron layer, only one texture orientation was observed, i.e. only the (001) plane of Fe were parallel to the film plane, shown in figure 5(b). From figure 5, it can also be found that there may be five orientation relationships between silver and iron grains, i.e.:

(1) $(001)_{Fe} \parallel (001)_{Ag}$, $[100]_{Fe} \parallel [110]_{Ag}$
(2) $(001)_{Fe} \parallel (011)_{Ag}$, $[100]_{Fe} \parallel [110]_{Ag}$
(3) $(001)_{Fe} \parallel (011)_{Ag}$, $[010]_{Fe} \parallel [1\bar{1}0]_{Ag}$
(4) $(001)_{Fe} \parallel (013)_{Ag}$, $[110]_{Fe} \parallel [010]_{Ag}$
(5) $(001)_{Fe} \parallel (013)_{Ag}$, $[1\bar{1}0]_{Fe} \parallel [100]_{Ag}$

and there may be four orientation relationships between silver and silver grains, i.e.:

(1) $(001)_{Ag1} \parallel (011)_{Ag2}$, $[110]_{Ag1} \parallel [110]_{Ag2}$
(2) $(001)_{Ag1} \parallel (011)_{Ag2}$, $[\bar{1}10]_{Ag1} \parallel [\bar{1}10]_{Ag2}$

(3) $(001)_{Ag1} \parallel (013)_{Ag2}$, $[100]_{Ag1} \parallel [100]_{Ag2}$
(4) $(001)_{Ag1} \parallel (013)_{Ag2}$, $[010]_{Ag1} \parallel [010]_{Ag2}$.

From the diffraction intensity of the films, it is thought that most of the Fe layers grew epitaxially on the (001) plane of the silver grains. A schematic diagram of an Fe/Ag bilayer is shown in figure 6, i.e. $(001)_{Fe} \parallel (001)_{Ag}$, $[100]_{Fe} \parallel [110]_{Ag}$.

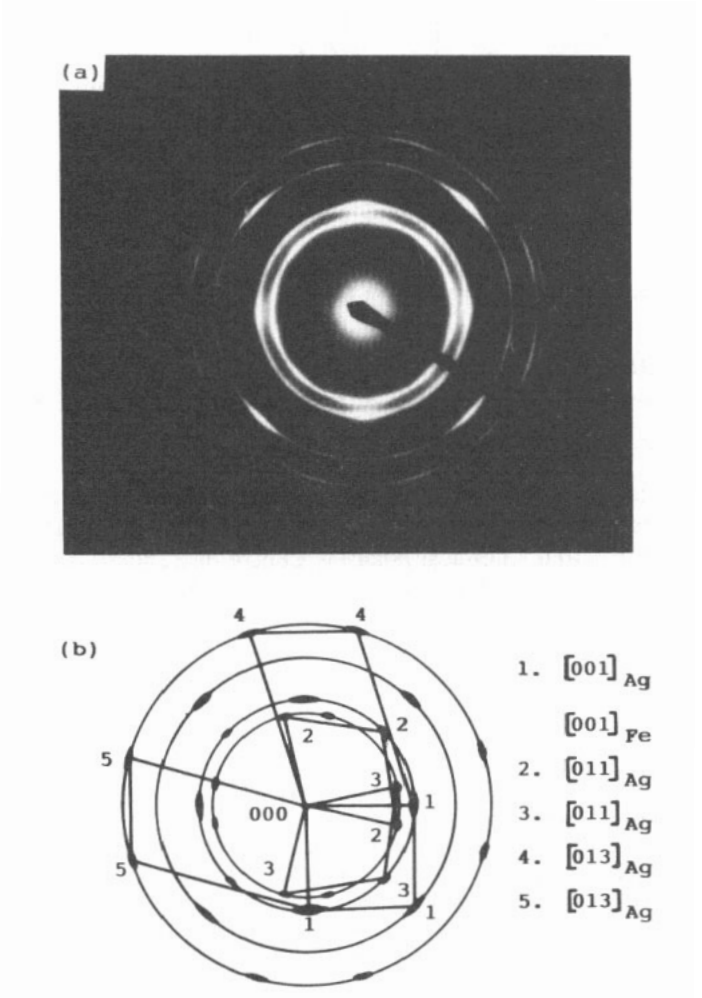

Figure 5. Electron diffraction patterns of Fe(4.5 nm)/Ag(9 nm) multilayers.

In summary, we have shown an enhanced magnetic moment as high as 2.60 μ_B in the Fe/Ag nano-multilayers. According to the calculation reported by Krompiewski *et al* [5], based on a first-principles tight-binding linear muffin-tin orbital method, the magnetic moment of the Fe/Ag(001) ultra-thin films can be 2.86 μ_B. The observed enhancement in Fe/Ag multilayers is therefore theoretically possible. Such properties, to our knowledge, have not been reported previously. The magnetic moment is

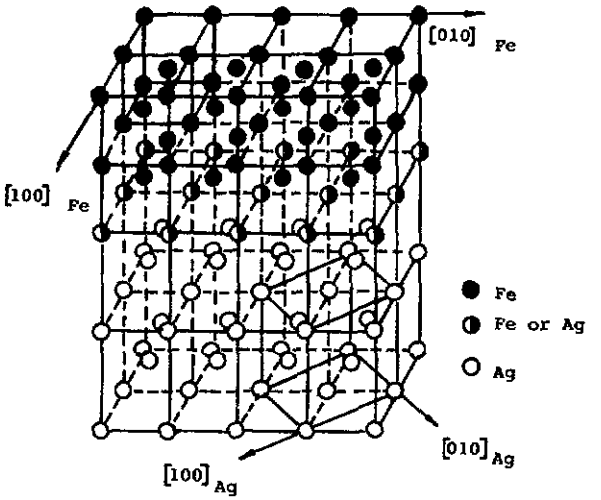

Figure 6. A schematic diagram of an Fe/Ag bilayer.

enhanced with the decreasing of the Fe layer thickness, and it is probably correlated with the epitaxial growth of an Fe layer on an Ag layer with an orientation relationship of $(001)_{Fe} \parallel (001)_{Ag}$, $[100]_{Fe} \parallel [110]_{Ag}$.

This work is supported in part by the National Natural Science Foundation of China. The authors are grateful to the Magnetism Laboratories, TEM Laboratories, Electron Microprobe Laboratories and Chemical Analysis Centre of Beijing University and the Institute of Materials Science of Tsinghua University for their assistance.

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