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LETTER TO THE EDITOR

^{57}Fe conversion electron Mössbauer spectroscopy studies of Fe/Pd multilayers

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Abstract. Fe/Pd multilayers with different thicknesses of Pd layers prepared by e-beam evaporation have been studied by ^{57}Fe conversion electron Mössbauer spectroscopy (CEMS) and x-ray diffraction (XRD). A structural transition of Fe layers from BCC to FCC in Fe/Pd multilayers was found with the thickness of Pd layers $d_{Pd} \geqslant 36$ Å. The CEMS measurements indicated that the hyperfine field H_{hf} of the inner parts of the Fe layers was almost the same as that of a single-layer Fe film and H_{hf} of the interface Fe was enhanced slightly from that of a single-layer Fe film. The moments of Fe layers were independent of the thickness of Pd layers and the structure of Fe layers, which suggested that the FCC Fe had the same magnetic moment as BCC Fe in Fe/Pd multilayers. The isomer shifts and quadrupole splitting increased abruptly for $d_{Pd} \geqslant 36$ Å. This would correlate with the structural transition of the Fe layers in Fe/Pd multilayers.

Recently, interest in Fe/Pd magnetic multilayers has increased strongly [1–9]. These studies mainly focused on interface anisotropy, spin polarization of Pd layers and interlayer coupling. Pd being a nearly ferromagnetic metal, its polarization effect has an influence on the magnetic properties of Fe layers and Fe/Pd multilayers. Mössbauer spectroscopy has proved to be a useful tool in investigating the surface and interface effects because of the atomic nature of this probe and is suited for determining H_{hf} both in the interior of a layer and at the interface. Hosoito *et al* [5] performed a depth-sensitive Mössbauer study of Pd/Fe/Pd sandwiches and found that H_{hf} was reduced from the bulk and that about 30% of the Fe layer was paramagnetic at 300 K. Boufelfel *et al* [6] studied the Fe(110)/Pd(111) superlattices from Mössbauer spectra and found that H_{hf} increased compared to that of bulk Fe, caused by the strong interface Pd polarization, and no paramagnetism of Fe layers was observed. This difference may be related to the structure of the Fe layers. So the structure and magnetic properties of Fe layers in Fe/Pd multilayers is needed for further investigation. In this letter, we report on Mössbauer and x-ray diffraction studies on Fe/Pd multilayers prepared by e-beam evaporation.

Fe/Pd multilayers were synthesized by using e-beam evaporation. The chamber was initially evacuated to 1×10^{-7} Torr and was at 5×10^{-7} Torr during evaporation. Both the deposition rates, about 0.6–0.8 Å s^{-1} for Fe and 1.0 Å s^{-1} for Pd, and the layer thickness were monitored by a water-cooled quartz crystal oscillator (IL400). Two types of material, 0.1 mm thick glass and kepton, were used as substrates for various measurements and the substrate was kept at room temperature during deposition. A series of multilayer samples

was prepared with the Fe layer thickness fixed at 20 Å and the Pd layer thickness varying from 6 Å to 60 Å systematically. The bilayer number was fixed at 25. The structural characterizations of the samples were carried out by both large- and small-angle x-ray measurements on a Rigaku diffractometer with an 18 kW rotating anode x-ray generator using a Cu target. The room-temperature ^{57}Fe conversion electron Mössbauer spectra of the samples were recorded by using a gas-flow (CH_3COCH_3) proportional counter, a 20 mCi ^{57}Co/Pd source (in the constant-acceleration mode), and a 1024-channel multichannel analyser. The spectrometer was calibrated with a standard α-Fe foil. The Mössbauer spectra were fitted by using the standard MUSFUN program on a PDP 11/34 computer system.

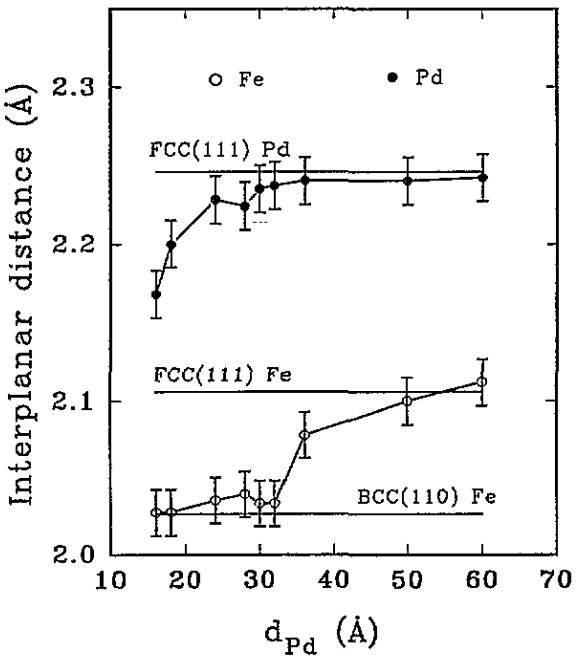

Figure 1. Interplanar distances of Fe layers and Pd layers as a function of d_{Pd}.

The small-angle x-ray reflectivity of the Fe/Pd multilayers indicated that the samples had a high-quality layered structure with small-angle spectra showing Kiessig fringes and up to third-order Bragg peaks. The crystalline-structure properties of Fe and Pd layers were checked by high-angle x-ray diffraction and two or three superlattice peaks on each curve can be seen. By fitting and analysing the above experimental results the superlattice periods, the interplanar distances and the coherent length were obtained. Figure 1 shows the dependence of the interplanar distances of Fe layers and Pd layers on the Pd layer thickness d_{Pd}. The interplanar distances of the Pd layer are compressed from that of bulk FCC Pd(111) planes and decrease as the Pd layer thickness decreases, while the interplanar distances of Fe layers are expanded from that of bulk BCC Fe(110) planes and increase with increasing Pd layer thickness. There is a jump at about $d_{Pd} = 36$ Å to that of the bulk FCC Fe(111) planes. In Fe/Pd multilayers, Pd layers are FCC(111) texture, and Fe layers are BCC(110) texture for $d_{Pd} < 36$ Å and FCC(111) texture for $d_{Pd} \geqslant 36$ Å. The detailed discussion of the structural character will be published elsewhere [10].

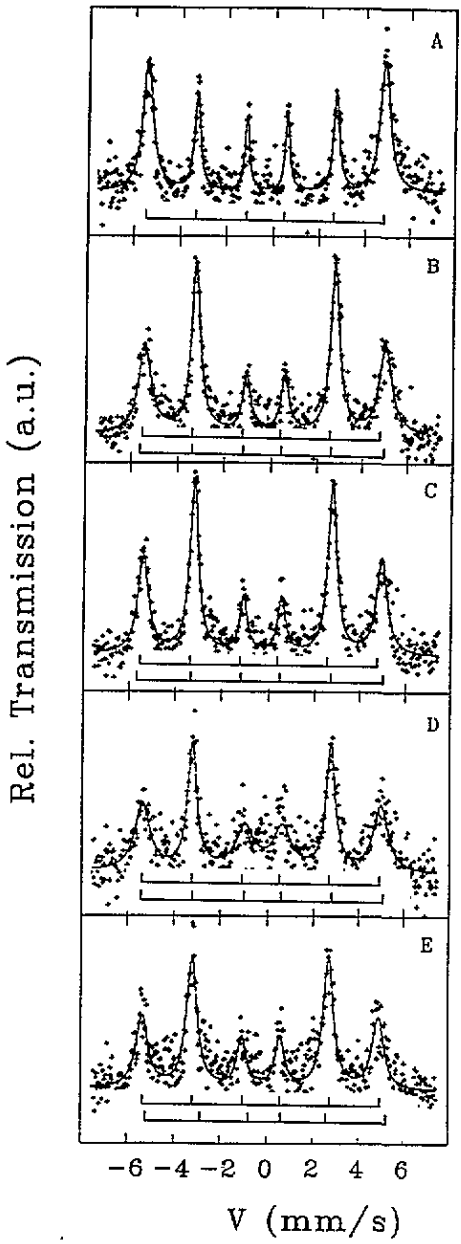

Figure 2. ^{57}Fe conversion electron Mössbauer spectra of Fe/Pd multilayers and a 1000 Å Fe film at room temperature: (A) 1000 Å Fe film; (B)–(E) samples with $d_{Pd} = 8$ Å, 18 Å, 28 Å and 36 Å, respectively.

Figure 2 shows the room-temperature ^{57}Fe conversion electron Mössbauer spectra of Fe/Pd multilayers and a single-layer 1000 Å Fe film for comparison. From figure 2 one can see that a six-line spectrum exists in these typical Fe/Pd multilayers. Each of the Mössbauer spectra of Fe/Pd multilayers can be fitted well with two six-line spectra. The

hyperfine field of the first set is the same as that of the 1000 Å Fe single-layer film (H_{hf} = 330 kOe); this may be attributed to the inner part of the Fe layers. The hyperfine field of the second subspectrum is slightly larger than that of the 1000 Å single Fe film, so the second subspectrum may be related to the interface part of the Fe layers. Figure 3 shows the dependence of the hyperfine field of the second subspectra on the thickness of the Pd layers. The hyperfine field first increases with d_{Pd} and reaches a maximum for d_{Pd} = 18 Å and then decreases slightly with further increase of d_{Pd}. This slight increase in H_{hf} is perhaps due to the polarization effect of Pd layers [6]. The CEMS results indicated obviously that the moments of Fe layers in Fe/Pd multilayers were almost the same as that of the Fe single-layer film and the magnetic moments of FCC Fe layers are also the same as that of BCC Fe layers in Fe/Pd multilayers. It is an interesting phenomenon that the structural transition in Fe layers does not affect the magnetic moments of Fe layers in Fe/Pd multilayers. At present, the explanation of this phenomenon is not known. In our samples, no paramagnetism of Fe layers was found in concordance with the results of Boufelfel *et al* [6].

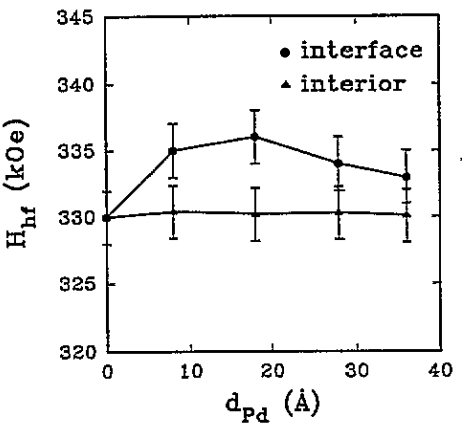

Figure 3. The hyperfine field of Fe/Pd multilayers as a function of d_{Pd}.

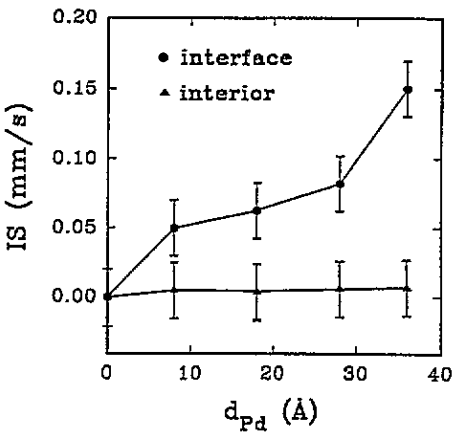

Figure 4. The isomer shift of Fe/Pd multilayers as a function of d_{Pd}.

Figure 4 shows the dependence of the isomer shifts (ISs) (with respect to α-Fe) of Fe/Pd on the thickness of the Pd layers. The ISs of the first subspectra of Fe/Pd multilayers have no significant changes compared to that of the 1000 Å single-layer Fe film. The ISs of the second subspectra are positive and increase with the thickness of the Pd layers. For d_{Pd} = 36 Å, the IS has a large increment and this may be related to the structural transition of the Fe layers. It is indicated that in Fe/Pd multilayers the s-like electrons of the interface parts of Fe layers transfer to the Pd layers and the thicker the Pd layers are, the more electrons are transferred. This implies that the Fermi surface of Fe layers and Pd layers in Fe/Pd multilayers has a large difference from bulk ones. This would affect the magnetic properties of Fe/Pd multilayers [11]. Figure 5 shows the quadrupole splitting (QS) of Fe/Pd multilayers and an interesting trend can be seen. The QSs of the two sets of subspectra of Fe/Pd multilayers become larger than that of a single-layer Fe film and there is also an abrupt increase for d_{Pd} = 36 Å. The QSs of the second set of subspectra are larger than those of the first ones. Since the QS arises from the presence of an electric-field gradient at the ^{57}Fe nucleus, the relative size of the splitting correlates with the degree of asymmetry

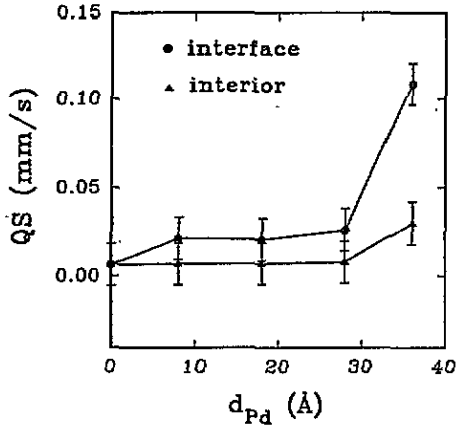

Figure 5. The quadrupole splitting of Fe/Pd multilayers as a function of d_{Pd}.

in the local electronic distribution. For $d_{Pd} \leqslant 28$ Å there is no distinguishable QS for the first set of subspectra, indicating a highly symmetric electronic environment in the interior sites of the Fe layers. The interface sites of the Fe layers have an asymmetric electronic environment. For $d_{Pd} = 36$ Å the QSs of the two sets of subspectra have obvious changes and this may be correlated with the structural transition of the Fe layers, implying the large difference in electronic environment between BCC Fe and FCC Fe. In the investigation of Boufelfel *et al* [6], no significant IS or QS were found, perhaps due to the difference in the Fe layers' structure. In our case, the interplanar distances of Fe layers and Pd layers have large changes with d_{Pd} but the structure of Fe layers and Pd layers in Boufelfel's samples had no such changes. It is interesting that the change of the Fe layers' structure does not affect H_{hf} but affects the IS and QS.

Very recently, Sakurai has found that in Fe/Pt multilayers the structure of Fe layers transits from FCC to BCC with increasing Fe layer thickness (near 10 Å) [12]. This is a very interesting conclusion. In Fe/Pd multilayers, a similar transition from FCC to BCC Fe with increasing Fe layer thickness (near 16 Å) was also found for a fixed Pd layer thickness (30 Å) [13]. Due to the differences between Pt and Pd, the thickness of Fe layers at which the structure transition occurs would be different for Fe/Pt and Fe/Pd multilayers. On the other hand, for a fixed Fe layer thickness in Fe/Pt multilayers, a transition from BCC to FCC Fe would also be found with increasing Pt layer thickness.

In summary, the structure of Fe layers in Fe/Pd multilayers depends on the thickness of the Pd layers, and changes from BCC(110) to FCC(111) for $d_{Pd} \geqslant 36$ Å. The Mössbauer study indicates that the magnetic moments of FCC Fe are almost the same as that of BCC Fe, but the abrupt changes in the ISs of the interface parts of the Fe layers and the QS for $d_{Pd} = 36$ Å correlate with the structural transition of the Fe layers.

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