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LETTER TO THE EDITOR

Spin-reorientation phenomena in Dy/Yb superlattices

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Abstract. Thermal hystereses resulting from different cooling conditions, such as under a field or under zero-field, were observed in the temperature dependence of magnetizations for the Dy/Yb superlattices prepared by a molecular beam epitaxy method. In addition, strong magneto-crystalline anisotropy and spin-reorientation phenomena from the magnetic frozen states to the ferromagnetic states were observed in the Dy/Yb superlattices.

Our interest has focused on the structural and magnetic characteristics of the superlattices containing rare-earth metals. To date, we have shown that the BCC Yb high-temperature phase [1] and the FCC Sm metastable phase [2] can be stabilized by the epitaxial relationship with BCC Eu and FCC Yb, respectively. In these studies, it is worth noting that the non-thermodynamic structures of Yb and Sm are stable up to a thickness large enough to be considered a bulk state. In addition, it has been reported that unique thermal hystereses are observed in the magnetic properties of amorphous Eu/Mn [3] and BCC Eu/Yb [4] superlattices. We have concluded that the individual Eu layers in superlattices are magnetically coupled to each other and the Eu interlayer interaction contributes to the thermal hysteresis phenomena observed. The question remains as to why the superlattices containing Eu metal were the only ones to have such unique magnetism. This prompted us to study superlattices composed of other magnetic rare-earth metals.

In the present study, Dy/Yb superlattices were prepared using a molecular beam epitaxy (MBE) method. We report its magnetic characteristics as elucidated by means of a superconducting quantum interference device (SQUID).

Dy (99.99%) and Yb (99.99%) metals were deposited on the cleaved NaCl (100) surface at a rate of 0.2 Å s^{-1} under ultra-high vacuum at 6×10^{-10} Torr using two Knudsen cells in an MBE apparatus (ULVAC original model) whose ultimate pressure was 7×10^{-11} Torr. The growth temperature was kept at 290 K. The period of the superlattice was repeated 25 times for each sample. As the first step, a 200 Å thick Yb buffer layer was grown on a NaCl substrate. Following this, the Dy and Yb metals were deposited alternately. A 300 Å thick Au layer was deposited on the surface of all finished samples to protect them from oxidation. The DC magnetization was measured using a SQUID (Hoxan HSM-2000) with sample temperatures between 4.2 and 200 K. The

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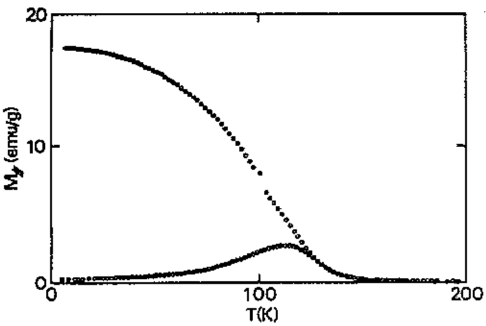


Figure 1. Temperature dependence of parallel magnetizations for a Dy (40 Å)/Yb (40 Å) superlattice measured under 50 G for field-cooled (under 50 G) and zero-field-cooled samples.

parallel magnetization ($M_{\parallel}$) was measured with the sample oriented parallel to the external applied magnetic field. The applied static magnetic field was up to ±5000 G. The total weight of Dy metal in the samples was determined by emission spectrochemical analysis (Nippon Jarrell-Ash ICAP-575). All magnetic data were corrected for magnetism arising from the Au protective layer, Yb component, NaCl substrate, and gelatine sample holder.

Reflection high-energy electron diffraction and x-ray diffraction (XRD) observations revealed that an FCC Yb single crystal layer was epitaxially grown on the cleaved (100) surface of the NaCl single crystal [2]. An epitaxial relationship in which the Yb (100) [100] is oriented parallel to the NaCl (100) [100] is obtained because of the small lattice mismatch (2.61%) between the FCC Yb and NaCl crystals. It was found that the Dy metal grown on the Yb layer possessed an HCP structure with the (102) and/or (110) plane parallel to the Yb (100) surface. The structure of the Yb layer deposited on the Dy layer was the same as that of the Yb layer epitaxially grown on the NaCl substrate. Very sharp and intense XRD peaks up to fifth order in the small-angle region suggested that the superlattice grew with smooth and clear interfaces. No impurities, which can give rise to magnetism, were detected by either electron probe micro analysis or Auger electron spectroscopy.

Thermal hystereses resulting from different cooling conditions were observed in the temperature dependence of magnetizations for the Dy/Yb superlattices as shown in figure 1. The magnetic-frozen states, characterized by the cusp in the temperature-dependent magnetization curve, were observed for zero-field-cooled superlattices. In contrast, the magnetization of field-cooled superlattices ferromagnetically increased with decreasing temperature. The thermal hysteresis, which is estimated by the difference between the magnetizations of field-cooled ($M_{F\parallel}$) and zero-field-cooled ($M_{Z\parallel}$) samples at 4.2 K, decreased exponentially as the Dy interlayer distance increased, as shown in figure 2. In addition, the freezing temperature, as defined by the cusp position, decreased with increasing Dy interlayer distance. As a result, the thermal hysteresis and cusp were not detected in Dy/Yb superlattices with Dy interlayer distances (Yb layer thicknesses) greater than 200 Å. Here note that the Yb metal which has the $4f^{14}$ configuration is non-magnetic. Based on our studies of the magnetic properties of Eu/Mn [3], Eu/Yb [4], and Dy/Yb superlattices, we can conclude that the individual magnetic rare-earth layers in these superlattices are magnetically coupled to each other. Furthermore, the thermal hysteresis phenomena, including the magnetic-frozen states, are associated with the magnetic rare-earth interlayer interaction. Such a long-range interaction must be a RKKY-type interaction which is mediated indirectly through the

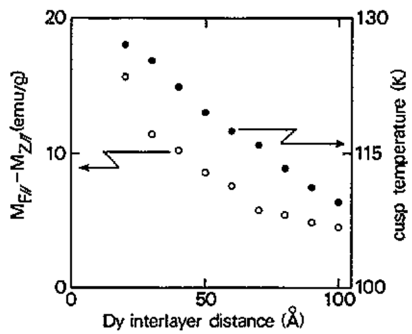


Figure 2. Thermal hysteresis (○) and cusp temperature (●) versus Dy interlayer distance for Dy (40 Å)/Yb superlattices.

polarization wave in the conduction bands of the rare-earth metals. Because the direct interaction between 4f spins which are localized in the inner core is negligibly small and is only effective over a short range.

With increasing magnetic field, the $M_{F\|} - M_{Z\|}$ value was gradually saturated as shown in figure 3. Similar behaviour has been observed in the Eu/Yb superlattices. The $M_{F\|} - M_{Z\|}$ value in the Eu/Yb superlattice was saturated until the magnetic field reached 50 G, above which it exponentially decreased [4]. As a result, the thermal hysteresis was not observed above 4.2 K under 300 G in the Eu/Yb superlattices. Such a critical field for the Dy/Yb superlattices was found to be higher than that of the Eu/Yb superlattices because the hysteresis in the Dy/Yb superlattices was clearly obtained under strong magnetic fields; as high as 5000 G. This difference in magnetic properties between Dy/Yb and Eu/Yb superlattices is likely to be due to the difference in strength of the interlayer interaction, such as Dy–Dy or Eu–Eu, which contributes to the thermal hystereses. This appears reasonable because the magnetic transition temperature of bulk Dy metal (179 K [5]) is higher than that of bulk Eu metal (91 K [6]). On the other hand, the freezing temperature decreased exponentially with increasing magnetic field as shown in figure 3. This shift of freezing temperature is very large and has not been observed before for spin-glass materials. In spin-glass materials, the magnetic-field dependence has been evident as a broadening of the cusp [7], where the freezing temperature changes little during measurement under static magnetic fields [8]. Therefore, it is implied that the Dy/Yb superlattices are not spin-glass materials. This is supported by the lack of time-dependent change of the magnetic states as described below, which is a characteristic normally found for spin-glass materials [9].

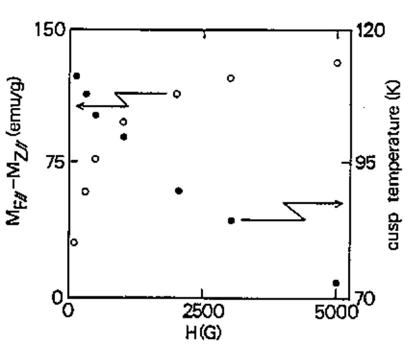


Figure 3. Thermal hysteresis (○) and cusp temperature (●) versus magnetic field for Dy (40 Å)/Yb (40 Å) superlattice.

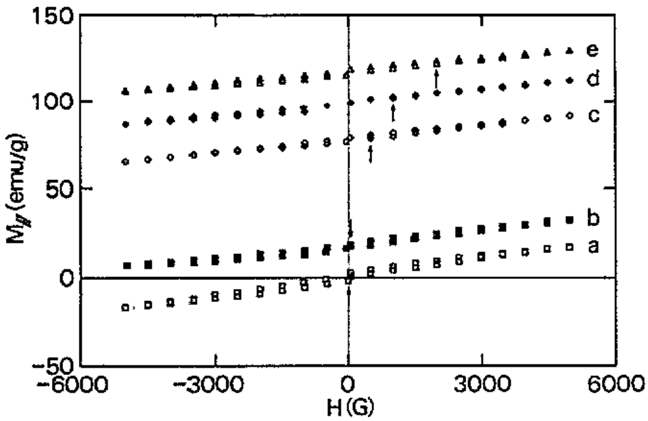
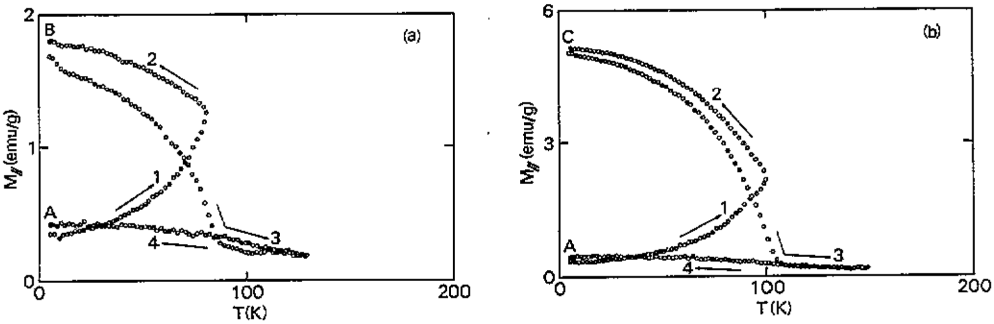


Figure 4. Dependence of Dy (40 Å)/Yb (40 Å) superlattice magnetizations at 4.2 K on magnetic-field cycling. The arrows indicate the starting points for the magnetic-field cycling on the samples: □, a, 0; ■, b, 50; ◇, c, 500; ◆, d, 1000; △, e, 2000 G.

The magnetic-field dependence of magnetization obtained at 4.2 K for a Dy (40 Å)/Yb (40 Å) superlattice is shown in figure 4. The arrows indicate the starting points for the magnetic-field cycling on the samples which were cooled from ambient temperatures to 4.2 K under the indicated fields. The superlattices which were cooled under a field possessed strong magnetic anisotropy. That is, the initial orientation of the magnetization was maintained in these samples, even though strong fields as high as 5000 G were applied in the opposite direction. This is due to the magnetocrystalline anisotropy associated with the interaction between the orbital angular momentum of 4f spin and the crystalline field of the lattice, which is well known in the bulk state of Dy metal [10].

When the Dy/Yb superlattices in the magnetic-frozen state, indicated by the letter A in figure 5(*a*)–(*c*), were heated to a set temperature (80 K (*a*), 100 K (*b*) and 125 K (*c*)) under 50 G, the frozen states gradually melted, as shown by arrow 1. Samples which were cooled back to 4.2 K under 50 G exhibited non-reversible temperature dependence on the magnetization and formed the B, C and D states (arrow 2). Here it is worth noting that the B, C and D states were maintained, even if the applied magnetic field became zero. This indicates that spin-reorientation from the magnetic-frozen state to the ferromagnetic state occurred in the Dy/Yb superlattices. In addition, the lack of time-dependent change of the magnetic states implies that the Dy/Yb superlattices are not spin-glass materials, as discussed above. When these samples, in their ferromagnetic states, were heated to set temperatures higher than the initial heating temperatures (80, 100 and 125 K) under zero field, their magnetizations reached the frozen level at 80, 100 and 125 K, respectively, as shown by arrow 3 in figure 5(*a*)–(*c*). Finally, the superlattices which were cooled back to 4.2 K under zero-field returned to the initial magnetic-frozen state A. It is very interesting that the desired magnetic states can be produced by controlling the initial heating temperature.

In summary, the magnetic-frozen states, characterized by the cusp in the temperature-dependent magnetization curve, were observed for the zero-field-cooled Dy/Yb superlattices. In contrast, the magnetization of field-cooled samples ferromagnetically increased as the temperature decreased. Such thermal hystereses resulting from different cooling conditions were also observed in the Eu/Mn and Eu/Yb superlattices and became more distinct with decreasing distance between the magnetic rare-earth layers. These results suggest that the magnetism of superlattices containing magnetic rare-earth metals possess thermal hystereses associated with the interlayer interaction. From the magnetic-field dependence, the Dy/Yb superlattices were found to



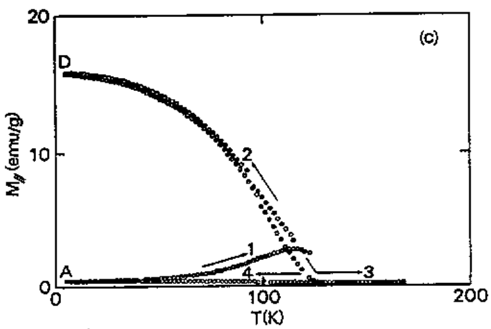


Figure 5. Spin-reorientation behaviour observed in the Dy (40 Å)/Yb (40 Å) superlattice. Samples in the A state were initially heated to the set temperatures: 80 (*a*), 100 (*b*) and 125 K (*c*), under 50 G as shown by arrow 1. These were cooled back to 4.2 K to form the B, C and D states (arrow 2). Next the samples were heated to set temperatures higher than 80, 100 and 125 K, under zero-field as shown by arrow 3 and finally cooled back to 4.2 K (arrow 4).

possess strong magnetocrystalline anisotropy. In addition, the spin-reorientation phenomena from the magnetic-frozen states to the ferromagnetic states were observed in the Dy/Yb superlattices.

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