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EPITAXIAL GROWTH AND NEW PHASE OF SINGLE CRYSTAL Dy BY MOLECULAR BEAM EPITAXY*

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EPITAXIAL FILM GROWTH AND NEW PHASE OF SINGLE CRYSTAL Dy BY MOLECULAR BEAM EPITAXY

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Abstract

We have grown two novel epitaxial phases of dysprosium (Dy) on vanadium (V) by molecular beam epitaxy technique. Surface and bulk structures are studied by in-situ reflection high energy electron diffraction (RHEED) and X-ray diffraction techniques. The new hcp phases are ~4% expanded uniformly in-plane (0001), and ~9% and ~4% expanded out of plane along the c-axes for non-interrupted and interrupted deposition case respectively. We also observed (2x2), (3x3), and (4x4) Dy surface reconstruction patterns and a series of transitions as the Dy film thickness increases.

Introduction

Considerable amount of experimental and theoretical effort has been directed towards the study of novel structural, electronic and transport phenomena in surfaces, interfaces and superlattices.[1,2]

Modern technology such as molecular beam epitaxy (MBE) enables us to create high quality epitaxial thin films and artificially modulated superlattices. A metastable bcc-Co epitaxial film[3] was grown on GaAs showing magnetic properties similar to bcc α-Fe. Rare-earth superlattice systems

(such as Gd/Y[4] and Dy/Y[5]) have been prepared by MBE to study the propagation of the long range magnetic order by indirect coupling across non-magnetic materials. Theoretical predictions of anomalous magnetic behavior have been made in expanded transition metal (TM) films (Ni, Co, V, Fe)[6] and in TM interfaced with normal metals (Fe/Ag, Cr/Au, V/Ag)[7].

The present paper reports on novel epitaxial phases of dysprosium (Dy) on vanadium (V) by MBE. Surface and bulk structures were studied by in-situ reflection high energy electron diffraction (RHEED) and X-ray diffraction techniques. The new hcp phases are identified. Surface reconstruction patterns of Dy films are also observed for the first time.

Experimental results and Discussion

The samples were prepared in a Riber MBE metal deposition system equipped with in-situ RHEED with a typical base pressure of 4×10^{-11} Torr. The films were deposited from high purity V(99.9%) and Dy(99.99%) starting materials on temperature controlled (~900°C) sapphire $(11\bar{2}0)\alpha Al_2O_3$ substrates at a rate of 0.5-1.0 Å/sec, and with a pressure of 5×10^{-10} Torr during evaporation. The film surface structure was monitored using 10 KeV in-situ RHEED. Bulk structures were studied further by a 2-axis Rigaku DMAX II X-ray diffractometer or with a 2 KW Cu-Kα tube.

A vanadium film was first deposited on the sapphire substrate as a buffer layer,[8] under the proper growth conditions 1000 Å V film surface is atomically smooth and the film grows epitaxially as indicated by the streaked RHEED pictures in Fig. 1(a) and (b), and by X-ray diffraction. Dy film was then evaporated on the V film, either in an interrupted or uninterrupted fashion. A final layer of V (110) (~500 Å) was always avaporated to protect the Dy from oxidation.

Figure 1(c)-(f) shows Dy RHEED pictures at a thickness of 4 Å ((c),(d)) and 50 Å ((e),(f)) in two different azimuthal orientations $\langle 11\bar{2}0\rangle_{Dy}$ and $\langle 10\bar{1}0\rangle_{Dy}$ with interruption. The sharp RHEED streaks are indicative of layer by layer growth. The 2θ /θ X-ray diffraction result of 50 Å Dy film along the film normal is shown in Fig. 2(a). The X-ray results imply that the growth direction is <0001> as expected for hcp-Dy with a ~4% expansion (c_o = 5.88 Å) compared to the bulk C_o^{bulk} = 5.6510 Å.[9] Note that additional small peaks due to the finite size effects are observed in both sides of the main broad peak. This fact indicates that both sides of Dy and V interfaces are atomically smooth and chemically sharp. This is possibly due to the fact that the binary phase diagram of Dy and V shows them to be immiscible,[10] and because the many interruption (~2 min. stop for 5 Å deposition) may allow the Dy atoms to diffuse and form smooth layers.

The in-plane lattice constant of Dy (a_o = 3.72 Å) obtained from the RHEED streaks in Fig. 1(c)-(f) is approximately 4% expanded relative to the bulk a_o^{bulk} = 3.5915 Å.[9] Therefore in the interrupted deposition a new modified hcp phase a_o = 3.72 Å (+4%) and c_o = 5.88 Å (+4%) was grown. It is quite interesting to note that an expansion is observed in both directions, so simple geometric arguments combined with Poisson ratios can not possibly explain these results. If the growth is not interrupted the growth changes considerably. X-ray diffraction from the Dy film shows a larger perpendicular expansion c_o = 6.18 Å (+9.4%) (see Fig. 2b) and a thickness dependence ranging from a_o = 3.64Å (+1%) to a_o = 3.79 Å (+6%) for the in-plane spacing. This observation may relate to the fact that fast deposition (or non-interruption) stabilizes metastable and largely strained structures, expanded one in this case.

The Dy crystal surface show a modified in-plane structure, (2x2), (3x3) and (4x4) reconstruction patterns which are observed for the first time using RHEED. Figure 3 shows a series of RHEED pictures for increasing by film thickness in a non-interrupted deposition.

For the interrupted case (2x2) and (3x3) reconstructions are observed but not the (4x4) pattern. The a_o's obtained for the different thickness are a_o = 3.64 Å (+1%), 3.72 Å (+4%) and 3.79 Å (+6%) and out-of-plane c_o = 5.88 Å (+4%) and 6.18 Å (+9%) with (2x2), (3x3) and (4x4) reconstruction respectively. Clearly the reconstruction (pxp) pattern is correlated with the in-plane and out-of-plane crystal structures. For large p the structures (a_o's and c_o's) tend to expand.

At this point we don't know how the surface structure is modulated i.e. an unit cell structure. However the structure will generally get less dense to accomodate large unit cell unless atoms deviate the position in a complicated fashion. Obviously structures of this reconstructions and thier phase transitions have to be studied further in detail.

Epitaxial orientation relation of Dy(0001) to V(110) was determined by RHEED results (in Fig.1). $\langle 11\bar{2}0\rangle_{Dy}$ is parallel to $\langle 110\rangle_{V}$, which is equivalent to the Nishiyama-Wassermann orientation[11] in fcc(111)/bcc(110) systems. The same reientation is reported in Gd/Nb system.[12] On the other hand, a new orientation was recently found in Ce/V system.[8]

The magnetic properties in this new hcp expanded phase would be very intersting to study in variety of magnetic ordering (ferromagnetic, helical) similar to the theoretical prediction in transition metals.

Acknowledgment

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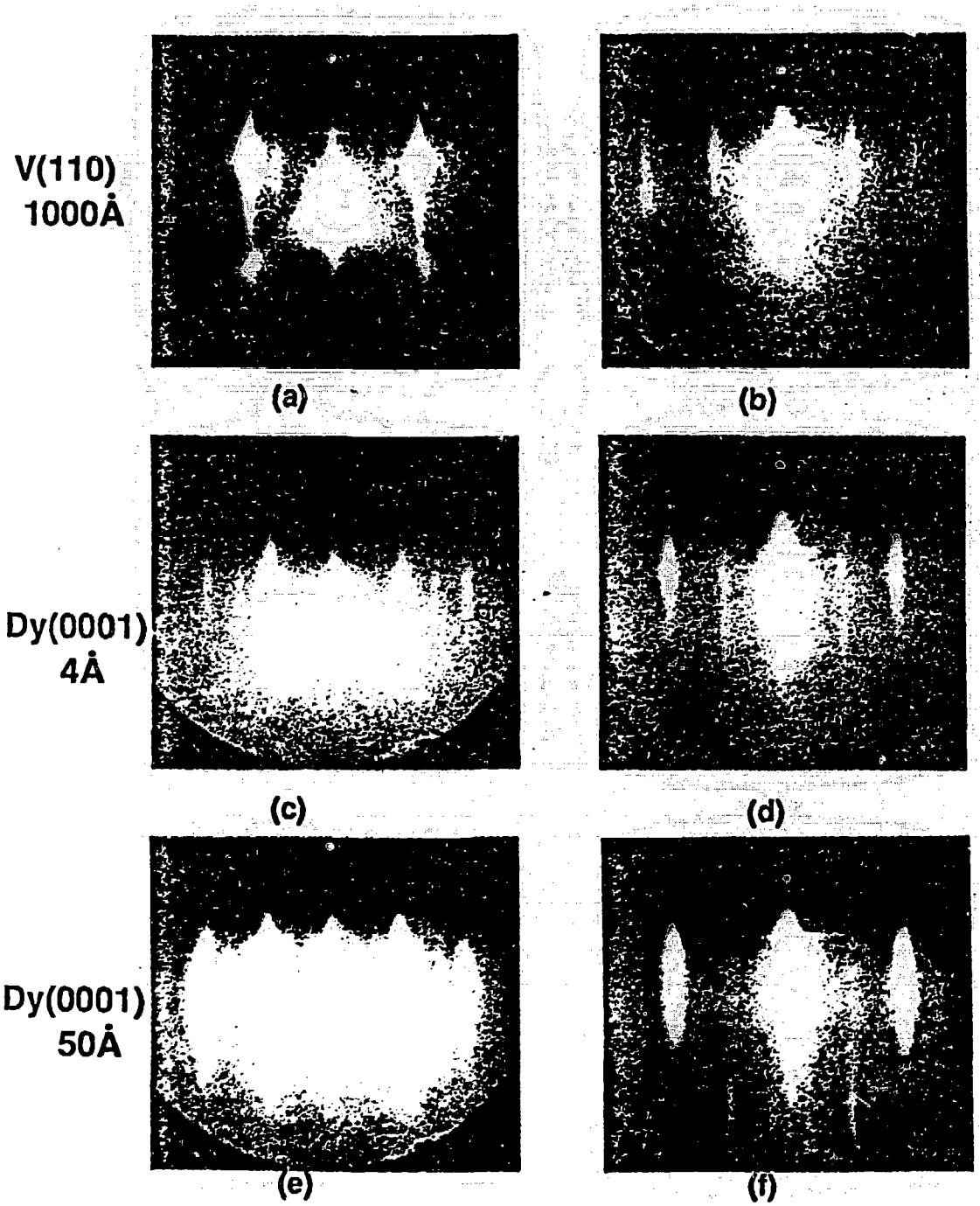
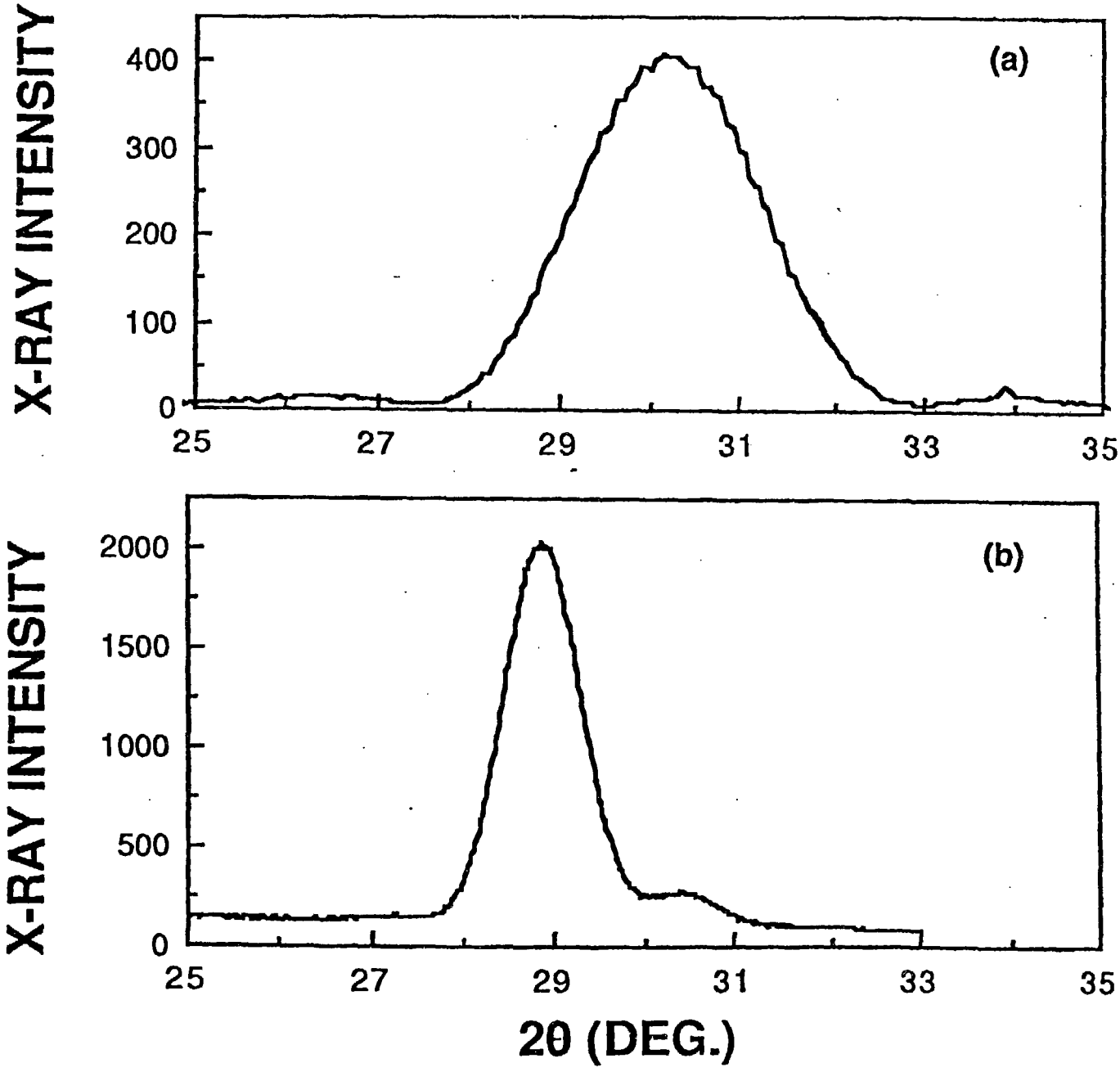
Figure Captions

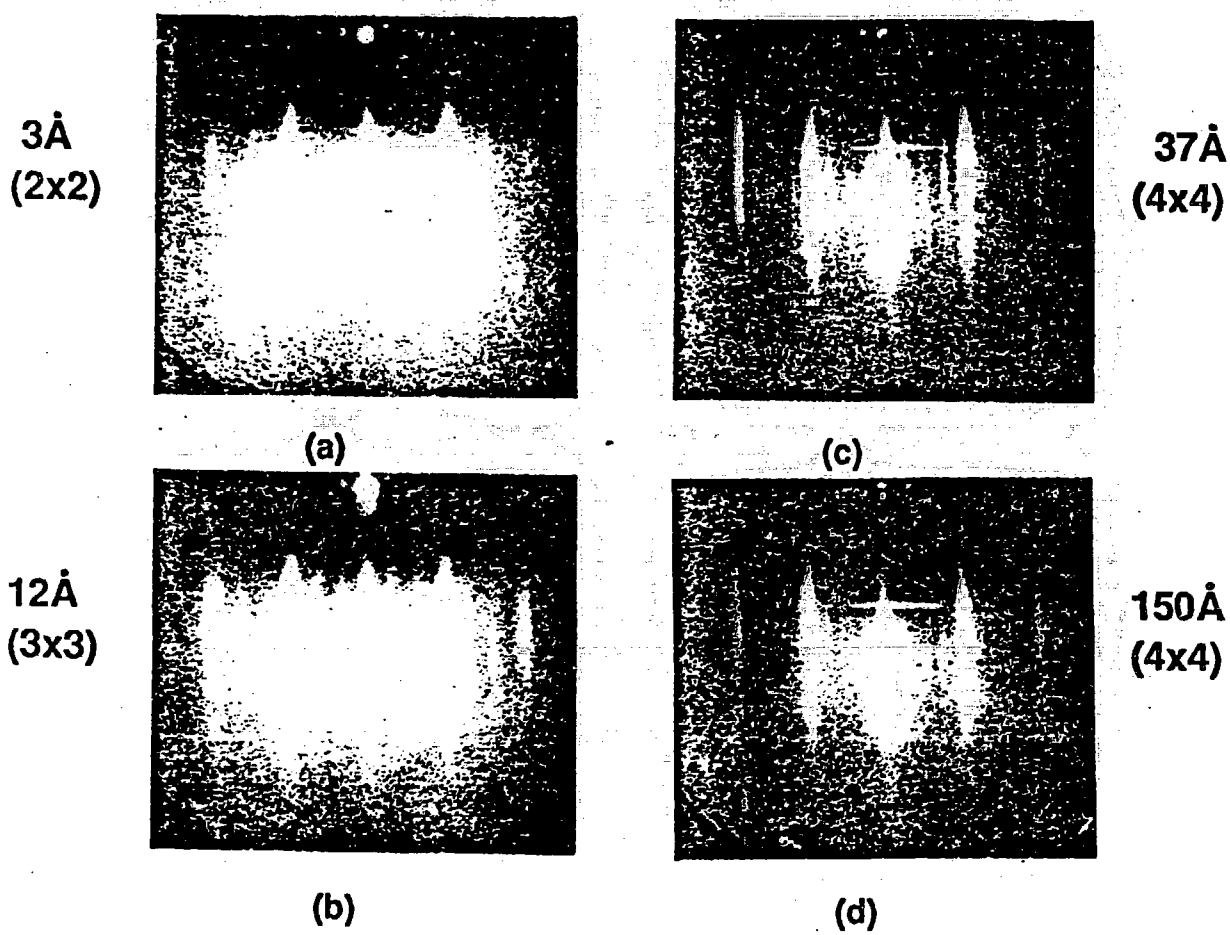
Fig. 1. RHEED patterns at 10 KV from (a) and (b) a 1000 Å-thick-V(110) film grown on a sapphire substrate α-Al_2O_3 $(1\bar{1}20)$ at 900°C, (c) and (d) 4 Å-thick-Dy(0001) film grown on V(110) in $\langle 11\bar{2}0\rangle_{Dy}$ and $\langle 10\bar{1}0\rangle_{Dy}$ orientation respectively, (e) and (f) 50 Å-thick-Dy(0001) film in the orientations corresponding to (c) and (d).

Fig. 2. $2\theta/\theta$ X-ray diffraction from (a) a 50 Å Dy(0001) film grown on V(110) in an interrupted fashion, and (b) a 50 Å Dy film grown on V(110) in a non-interrupted fashion.

Fig. 3 RHEED patterns at 10 KV from Dy(0001) film grown on V(110) at (a) 3 Å, (b) 12 Å, (c) 37 Å, and (d) 150 Å, showing (2x2), (3x3), (4x4), and (4x4) reconstruction patterns respectively.


V(110)
1000Å
(a)
(b)
Dy(0001)
4Å
(c)
(d)
Dy(0001)
50Å
(e)
(f)


X-RAY INTENSITY
400
300
200
100
0
(a)
25
27
29
31
33
35
X-RAY INTENSITY
2000
1500
1000
500
0
(b)
25
27
29
31
33
35
2θ (DEG.)


3Å
(2x2)
(a)
37Å
(4x4)
(c)
12Å
(3x3)
(b)
150Å
(4x4)
(d)