Barium ferrite thin film media with perpendicular *c*-axis orientation and small grain size

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Barium ferrite thin films with perpendicular *c*-axis orientation and small grain size (about 300 Å) were successfully fabricated with careful control of sputtering conditions. The *c*-axis orientation of barium ferrite thin films is most sensitive to the oxygen partial pressure during deposition. All samples with oxygen gas during deposition have a random *c*-axis texture, as indicated by existence of both weak (00*l*) peaks and (106) peaks. All the samples without oxygen gas during deposition show only strong (00*l*) peaks, which indicate excellent perpendicular *c*-axis orientation. Transmission electron microscopy results show that oxygen gas promotes the growth of in-plane and/or randomly oriented grains. The effect of the Pt interlayer on the barium-rich films was also studied. The Pt interlayer was found to be very effective in improving *c*-axis orientation of barium-rich films. A relative increase in perpendicular nucleation sites over in-plane and/or random nucleation sites contributes to the improvement in perpendicular *c*-axis orientation. [S0021-8979(00)60808-3]

Barium ferrite thin film media are attractive candidates for ultrahigh density overcoat free perpendicular magnetic recording. One of the major challenges is to reduce grain size in order to reduce media noise. The grain size was reported to be typically larger than 1000 Å for perpendicular oriented barium ferrite.[1–3] Reducing the grain size for barium ferrite thin film was achieved by doping a small amount of CoTi, Cr_2O_3.[3,4] But once doping with other elements as above, barium ferrite thin films became randomly oriented.[3,4] Barium ferrite films with higher barium content were shown to have smaller grain size and random *c*-axis orientation.[5] It is difficult to control the perpendicular *c*-axis orientation with barium-rich films. Perpendicular *c*-axis orientation was only achieved by two-step annealing for barium ferrite films with a thickness 300 Å or less on Pt underlayer.[6] Both fine grain size and perpendicular orientation of the *c* axis are desirable microstructural characteristics for barium ferrite used as perpendicular thin film media. In this study, effects of processing parameters and Pt interlayer on the 10 wt % barium-rich barium ferrite were investigated.

Barium ferrite thin films and Pt films were deposited by rf diode sputtering in a Leybold Z-400 sputtering system. A base pressure of less than 1×10^{-6} Torr was achieved before deposition. The Pt underlayer was deposited in pure Ar gas, with a pressure of 5.0 mTorr and a deposition rate of about 100 Å/min. For all the films, a 500-Å-thick Pt underlayer was first deposited onto a thermally oxidized silicon substrate. The purpose of the Pt underlayer is to promote the perpendicular orientation. 10wt % barium-rich nonstoichiometric barium ferrite thin films were then deposited in a mixture of Ar and O_2 on the Pt underlayer. The flow rate ratio of Ar and O_2 mixture was changed with the total pressure fixed at 5.7 mTorr. All the deposited barium ferrite films were annealed in a rapid thermal annealing furnace at a temperature of 800 °C for about 60 s to fully crystallize the films.

The effects of processing conditions on the magnetic properties of stoichiometric barium ferrite thin films have been previously reported.[2] In our work, we found that the processing conditions have very different effects for barium-rich films than for the stoichiometric films. Thus the effects of processing parameters such as oxygen partial pressure, chamber pressure, and substrate bias on the magnetic properties of barium-rich films were investigated.

The oxygen partial pressure was found to have the largest effect on the *c*-axis orientation of the barium-rich thin films. Two samples with different oxygen partial pressure were made on a 500-Å-thick Pt underlayer, and their magnetic properties were studied. Sample A was sputtered with an oxygen gas partial pressure of 0.7 mTorr, while sample B was sputtered without additional oxygen gas. The total gas pressure during sputtering was fixed at 5.7 mTorr. The barium ferrite film thickness for both samples A and B was 600 Å. The *MH* loops for samples A and B are shown in Figs. 1(a) and 1(b), respectively. Sample A shows similar in-plane and perpendicular squareness of around 0.7, while sample B shows a much better perpendicular orientation, with a perpendicular squareness of 0.9 and an in-plane

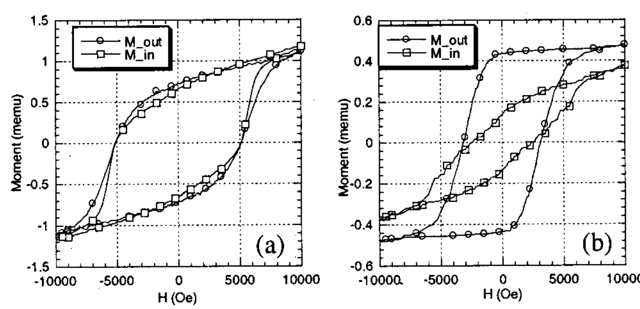

FIG. 1. In-plane *MH* loop (*M*_in) and perpendicular *MH* loop (*M*_out) for (a) sample A and (b) sample B.

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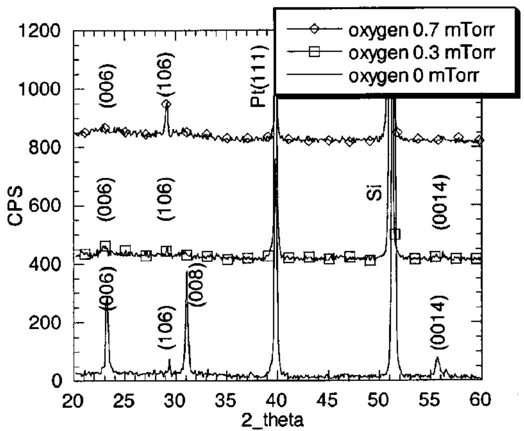

FIG. 2. X-ray diffraction results for 600-Å-thick barium-rich barium ferrite thin films with different oxygen partial pressure on a 500-Å-thick Pt underlayer.

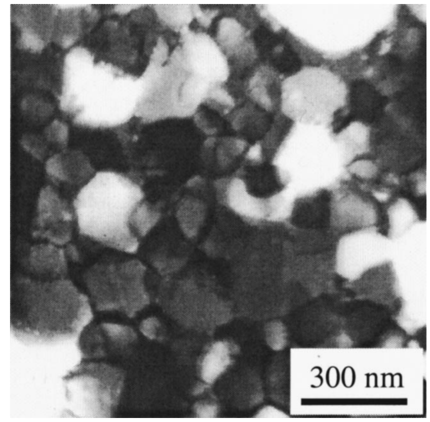

FIG. 4. TEM plan-view BF micrograph for a 600-Å-thick stoichiometric barium ferrite thin film on a 500-Å-thick Pt underlayer.

squareness of 0.3. The saturation magnetization (M_s) of both samples A and B is about the same, 200 and 210 emu/cc, respectively. Sample A has a perpendicular coercivity of about 5000 Oe, while sample B has a perpendicular coercivity of about 3000 Oe, as shown in Figs. 1(a) and 1(b).

X-ray diffraction results for 600-Å-thick barium ferrite thin films with a 500-Å-thick Pt underlayer are shown in Fig. 2. The oxygen partial pressure was varied from 0 to 0.7 mTorr. For all the samples without oxygen gas, strong (00*l*) peaks are observed, which indicate excellent perpendicular *c*-axis orientation. As the oxygen partial pressure increases, (00*l*) reflection peaks become weaker. At the same time, the (106) reflection peak becomes a little stronger with respect to (00*l*) peaks. There is no obvious preferred orientation for all the films prepared with a partial pressure of oxygen, while there is strong perpendicular *c*-axis orientation for films prepared without oxygen partial gas.

TEM plan-view bright field (BF) micrographs for samples A and B are shown in Fig. 3. For sample A, both acicular and platelet grains are observed. The acicular grains have an average size of 600 Å in length and 250 Å in width, as shown in Fig. 3(a). The platelet grains have an average size around 300 Å. For sample B, plateletlike grains dominate with an average size of about 300 Å, as shown in Fig. 3(b). The acicular grains are either *c*-axis in-plane oriented or randomly oriented. The electron diffraction from one acicular grain shown at the top-left corner of Fig. 3(a) clearly shows *c*-axis in-plane orientation. The plateletlike grains are *c*-axis perpendicularly oriented, as confirmed by the electron diffraction pattern from one platelike grain shown at the top-left corner of Fig. 3(b). The above results clearly indicate that oxygen partial pressure affects the grain growth and grain orientation during annealing. Oxygen partial gas seems to enhance the growth of the in-plane and randomly oriented acicular grains, thus worsening the perpendicular *c*-axis orientation.

To compare the grain size, a 600-Å-thick barium ferrite thin film with stoichiometric composition was also made on a 500-Å-thick Pt underlayer. TEM BF micrograph for the stoichiometric film is shown in Fig. 4. The grain size was measured to be between 1600 and 2000 Å. Comparing Figs. 3(a) and 4, we can see that barium-rich films have a much reduced grain size compared to the stoichiometric barium ferrite films.

The effects of chamber pressure on the barium-rich barium ferrite films were also studied. The films show no obvious dependence on the pressure from a range of 2.5 to 10 mTorr, when oxygen gas was used as a second gas during

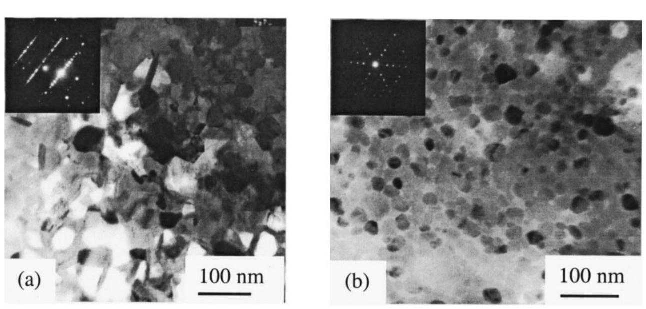

FIG. 3. TEM plan-view BF micrographs and electron diffraction patterns for (a) sample A and (b) sample B.

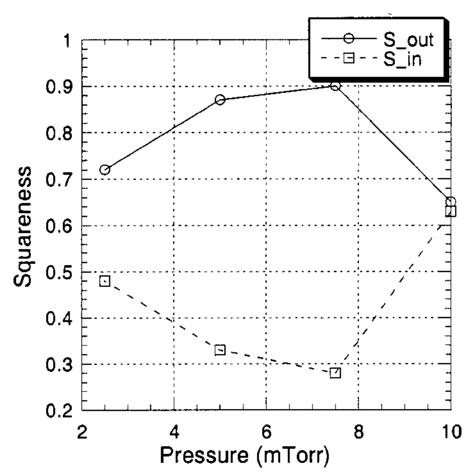

FIG. 5. Pressure dependence of in-plane squareness (S_in) and perpendicular squareness (S_out) for 600-Å-thick barium-rich barium ferrite thin films on a 500-Å-thick {Pt} underlayer.

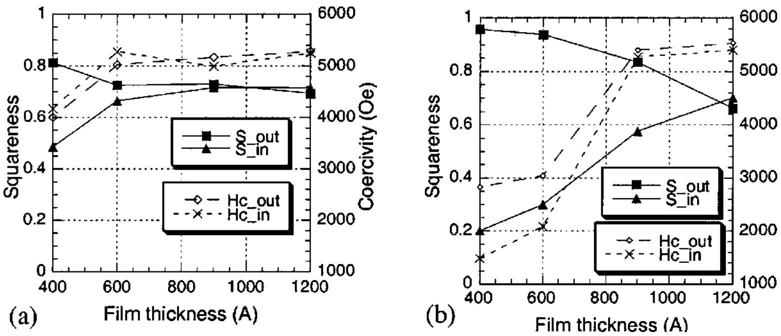

FIG. 6. Thickness dependence of in-plane squareness (*S*_in), perpendicular squareness (*S*_out), in-plane coercivity (H_C_in), and perpendicular coercivity (H_C_out), of barium rich barium ferrite thin films:(a) films prepared with an argon partial pressure of 5.1 mTorr and an oxygen partial pressure of 0.6 mTorr; (b) films prepared with pure argon gas with a pressure of 5.7 mTorr.

sputtering. Without oxygen gas during sputtering, the chamber pressure was found to affect the magnetic properties of barium ferrite films. The relationship between remanent squareness and chamber pressure for barium ferrite prepared without oxygen partial pressure is shown in Fig. 5. Within the range from 5.0 to 7.5 mTorr, the perpendicular squareness is about 0.9, while the in-plane squareness is about 0.3. As the chamber pressure decreases to 2.5 mTorr or increases to 10 mTorr, the perpendicular squareness decreases to about 0.7, while the in-plane squareness increases to about 0.55. So barium ferrite thin films with similar good perpendicular *c*-axis orientation can be achieved in a pressure range from 5.0 to 7.5 mTorr. The perpendicular *c*-axis orientation becomes worse as the chamber pressure deviates from above pressure range. The effects of substrate bias on the barium-rich barium ferrite films were also studied. The films show no obvious dependence on the substrate bias voltage with a range from 0 to −150 V either with an oxygen partial pressure or without oxygen gas during sputtering.

The thickness dependencies of remanent squareness and coercivity of barium-rich barium ferrite thin films with and without oxygen gas are shown in Figs. 6(a) and 6(b), respectively. Barium ferrite thin films deposited with oxygen gas partial pressure of 0.7 mTorr show random *c*-axis orientation when the thickness is 600 Å thick or larger. The 400-Å-thick barium ferrite shows a weak perpendicular orientation. Barium ferrite thin films deposited without oxygen gas have a perpendicular *c*-axis orientation up to a thickness of 1000 Å. The 400-Å-thick barium ferrite shows a very strong perpendicular *c*-axis orientation as indicated by a higher perpendicular squareness of 0.95.

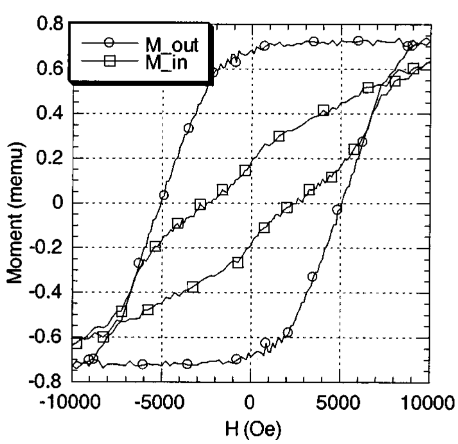

FIG. 7. In-plane and perpendicular *MH* loops of 900-Å-thick barium-rich barium ferrite thin films with a 150-Å-thick Pt interlayer.

Even with optimized sputtering conditions, there is a gradual deterioration of perpendicular *c*-axis orientation as the thickness of barium ferrite films increases, as shown in Fig. 6(b). The reason for the deterioration of the *c*-axis orientation with the thickness was attributed to the increase of random nucleation sites over perpendicular nucleation sites, due to the increase of bulk nucleation sites with the film thickness.[6,7] We have found that the Pt interlayer is effective in improving the *c*-axis orientation on the stochiometric barium ferrite thin films.[7] Thus, the effect of a Pt interlayer was also studied for the barium-rich films. The 900-Å-thick barium ferrite media without a Pt interlayer has a perpendicular squareness of about 0.82 and an in-plane squareness about 0.55, as indicated in Fig. 6(b). The *MH* loops of 900-Å-thick barium ferrite thin films with a 150-Å-thick Pt interlayer are shown in Fig. 7. The film with a Pt interlayer shows an improved *c*-axis orientation, as indicated by the increase of perpendicular remnant squareness to above 0.9, and the decrease of in-plane squareness to about 0.25. Thus, a Pt interlayer is also very effective in improving the *c*-axis orientation for barium-rich films. A relative increase in perpendicular nucleation sites over in-plane and/or random nucleation sites contributes to the improvement in perpendicular *c*-axis orientation for films with a Pt interlayer.[6,7]

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