CHARACTERISATION OF NdFeB THIN FILMS PREPARED ON (100)Si SUBSTRATES WITH SiO_2 BARRIER LAYERS

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ABSTRACT

This work presents a systematic study of the deposition and characterization of NdFeB films on substrates of Si(100) and of SiO_2 layer thermally grown on Si(100) held at RT, 360 °C or 440 °C. The post-deposition annealing is performed at 600 or 800 °C in vacuum. The films are characterised using the analytical techniques of RBS, SIMS, XRD, OM and SEM. Results indicate that SiO_2 is, in deed, an excellent diffusion barrier layer till 600 °C but becomes relatively less effective at 800 °C. Without this barrier layer, interdiffusion at the Si-NdFeB film interface leads to formation of iron silicides, α-Fe and B exclusion from the diffusion zone, in competition with the formation of the magnetic NdFeB phase.

INTRODUCTION

Deposition of permanent magnetic thin films on single crystal silicon substrates is an essential requirement for the incorporation of permanent magnet features into some micro electro mechanical systems (MEMS) fabricated by the planar process. However, deposition or annealing at high temperatures is required to grow the magnetic phases; e.g. NdFeB films with good magnetic properties have been deposited on several substrates (glass, sapphire, quartz, Nb or Ta) by using sputter deposition at substrate temperatures of about 600 °C or post deposition annealing [1-3]. Our recent work [4] on post deposition annealing of NdFeB films on Si(100) has shown that a)massive interdiffusion at the interface is a major problem and b)thin layers of SiO_2 can reduce this interdiffusion. Thermally grown SiO_2 which is an integral part of MEMS processing could therefore make an excellent diffusion barrier. This work presents a systematic study of this hypothesis.

EXPERIMENTAL

NdFeB films (0.1 and 2 μm thick) are sputter deposited using a dc magnetron operating in pure Ar at a pressure of 1 Pa. The target composition was $Nd_2Fe_{14}B$. The substrates of Si(100) and of SiO_2 layer (1.2μm thick) thermally grown on Si(100) are held at RT, 360°C or 440°C. The post-deposition annealing is performed for 20 m at 600 or 800 °C in vacuum ~ 10^{-5} Pa. The films are characterised using the analytical techniques of RBS, SIMS, XRD and SEM.

RESULTS AND DISCUSSION

Films deposited on Si(100): The nominal composition of the films deposited on Si(100) substrates at RT or 360°C is estimated from RBS to be $Nd_{1.9}Fe_{13}B_4$. However, RBS analysis of films deposited at 440 °C indicated atomic diffusion at the Si substrate-NdFeB film interface. The SIMS depth profiles for a film deposited at RT (Fig.1a) show B quite well; and those for a film deposited at 440 °C (Fig. 1b) indicate a clear outward migration of B or Nd and inwards migration of Fe. The effect of annealing the NdFeB films deposited on Si (100) can be seen in Fig. 2 for different deposition temperatures. The films deposited at RT (Fig.2a) undergo considerable interfacial diffusion of Fe and Si, which increases with the annealing temperature. The XRD shows formation of silicides of Fe. The Nd shows strong surface segregation but much weaker inwards diffusion after 800 °C anneal. The anneal behaviour of films deposited at 360 °C (not shown) is similar. The anneal behaviour of films deposited at 440 °C (Fig.2b) shows considerable difference for Nd migration particularly after the 800 °C anneal when it show a double peak formation. It may be noted that there is no evidence of any oxidation of the film during vacuum annealing. The detailed examination of RBS, SIMS and

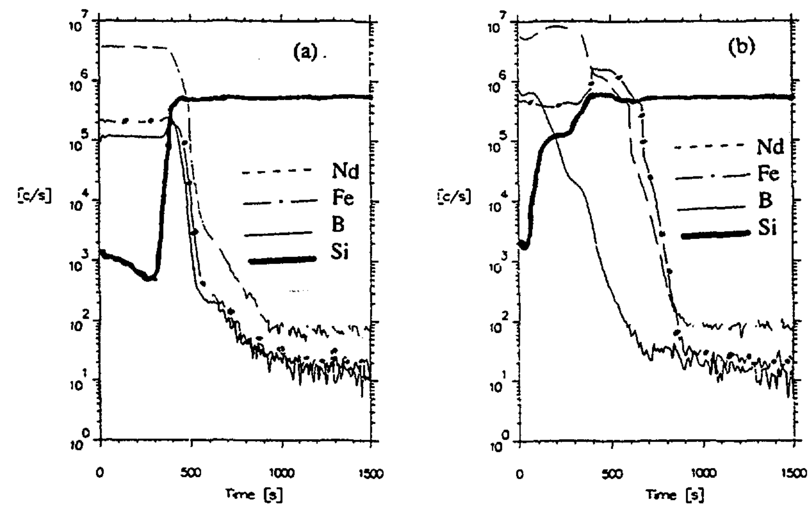

Fig.1. SIMS spectra of films deposited at (a) R.T (b) 440°C

XRD data obtained from the same films indicates- a) the as deposited films at RT and 360 °C are amorphous, b) but those deposited at a temperature of 440 °C and all films after annealing exhibit a massive interdiffusion at the Si-NdFeB film interface leading to formation of iron silicides,α-Fe and B exclusion from the diffusion zone, in competition with the formation of the magnetic NdFeB phase (no lines for NdFeB are seen in XRD). A diffusion barrier layer is therefore required for crystallising the magnetic phase of NdFeB on Silicon.

Films deposited on barrier layer of SiO_2: RBS analysis of films deposited on silicon substrate with an SiO_2 barrier layer didn't show any interdiffusion between the film and substrate. The SIMS data (not shown) obtained from the same films confirms the suppression of B diffusion observed earlier in Fig.1b. The stability of this diffusion barrier action has been studied for the annealing temperature of 600 °C (Fig. 3a) and 800 °C (Fig. 3b). The post deposition annealing at 600 °C results (Fig.3a) when compared with those in Fig.2 clearly demonstrate the excellent stability of the SiO_2 as a diffusion barrier. A small amount of Si can be seen to be present at the surface near the Si edge. This could be due to a small number of pin holes in SiO_2. However, after annealing at 800 °C (Fig.3b), significant changes in the Fe, Nd and Si depth profiles near the film surface can be noticed. Emergence of the new oxygen peak near the surface edge may arise from formation of surface oxides of Fe or Nd or Si. Silicide formation appears to be absent as deduced from the absence of Fe diffusion into Si.

Problems: Thicker films (2 μm) delaminate after undergoing thermal treatment beyond 360°C. This may be due to several reasons - stress relief, mismatch of the thermal expansion coefficient between SiO_2 or Si and FeNdB phases, formation of silicides or α-Fe at the interface etc.

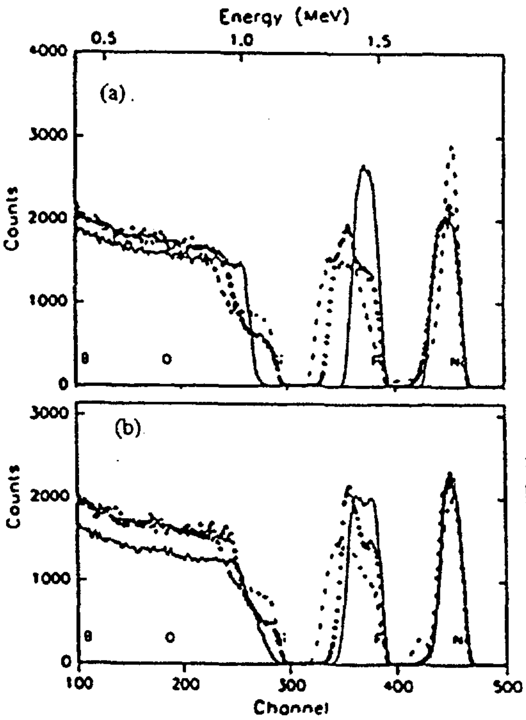

Fig.2. RBS spectra of films (a)Asdeposited at R.T(—) annealed at 600°C (·) and 800°C (--)
(b) Asdeposited at 440°C(—), annealed at 600°C (·) and (b) 800°C

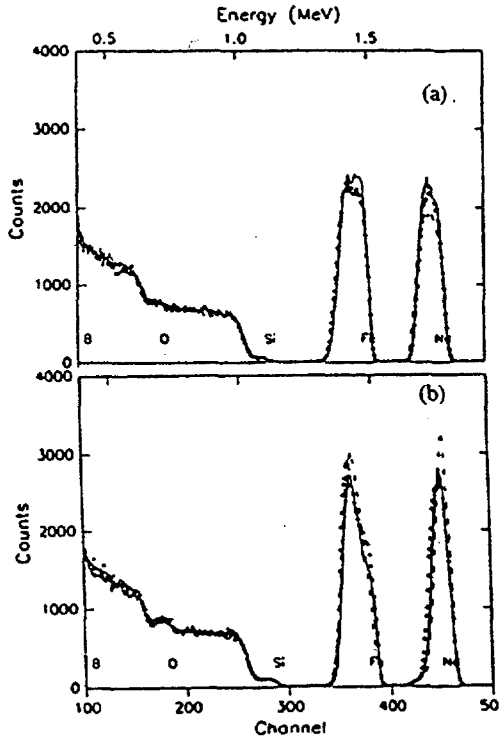

Fig.3.RBS spectra of films deposited on silicon with SiO_2 barrier layer at R.T(—), 360°C(·),440°C(--)and annealed at (a) 600°C and (b) 800°C

Further work is in progress to optimise conditions to overcome this undesirable delamination effect for thicker films. Magnetic properties are also under investigation.

CONCLUSIONS

1. Thermally grown SiO_2 makes a good diffusion barrier layer up to 600 °C. 2. However some slight surface degradation of the NdFeB films sets in after annealing at 800 °C. Interfacial diffusion is still minimal. 3. Without the diffusion barrier layer, interdiffusion at the Si-NdFeB interface leads to the formation of $FeSi_x$, α-Fe phases and exclusion of B from the intermixing zone.

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REFERENCES

1. F.J.Cadieu, J. Appl. Phys., 61, 4105 (1987).
2. T.Araki and M.Okabe, Proc. of IEEE 9th Annual Int. workshop on MEMS, 244, (1996)
3. K.D.Aylesworth, Z.R.Zhao, D.J.Sellmyer and G.C.Hadjipanayis, J. Magn. Mater., 82, 48 (1989).
4. G.K.Muralidhar, B.Window, D.K.Sood and R.B.Zmood, J. Mater. Sci. (In press)