Thermally-induced phase transitions in Pt/Tb/Fe trilayers

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Introduction

Structural phase formation induced by thermally-activated diffusion processes and intermixing of layered structures is a promising reaction pathway for the synthesis of unexpected (metastable) thin film materials. Furthermore, the sequence of structural phase transitions during annealing can substantially differ from those predicted by corresponding bulk phase diagrams due to the underlying grain boundary diffusion mechanisms as well as surface and interface effects.

In case of complex multilayer stacks the control of intermediate and final phase products formed at different stages of thermal processing becomes a relevant issue. In particular, it is important to understand the sequence and temperature intervals of structural phase transitions in such stacks.

In this study, we focus on the various stages of thermally-induced phase transitions in Pt/Tb/Fe trilayers, which were investigated by x-ray diffraction, transmission electron microscopy, secondary neutral mass spectrometry and Auger electron spectroscopy depth profiling, as well as by magnetic properties measurements using superconducting quantum interference device-vibrating sample magnetometry.

Methodology

Trilayers of Pt(15 nm)/Tb(10 nm)/Fe(15 nm)/sub. were prepared via dc magnetron sputtering at room temperature on thermally oxidized SiO2(100 nm)/Si(1 0 0) substrates from pure Fe (99.99 %), Pt (99.99 %), and Tb (99.9 %) targets. The as-deposited films were post-annealed at different temperatures up to 620 ° C in vacuum (10-3 Pa) applying an average heating rate of 0.5 ° C/s.

Structural properties as well as phase composition of the as-deposited and post-annealed samples were investigated by x-ray diffraction **(XRD)** in (θ-2θ) geometry using a Rigaku Ultima IV diffractometer with Cu-Kα radiation. Cross-sectional transmission electron microscopy **(TEM)** imaging was performed on a Philips CM-200 FEG TEM operated at 200 kV. In parallel to TEM imaging, local chemical analysis was performed by energy dispersive x-ray **(EDX)** spectroscopy. In addition, chemical depth profiling was carried out by secondary neutral mass spectrometry **(SNMS)** in order to evaluate the elemental distribution through the film depth after. Furthermore, Auger electron spectroscopy **(AES)** measurements were accomplished using a Jamp-9500F device. In-plane magnetic hysteresis loops of the samples were measured at room temperature in magnetic fields up to ±70 kOe using superconducting quantum interference device-vibrating sample magnetometry **(SQUID-VSM).**

Results

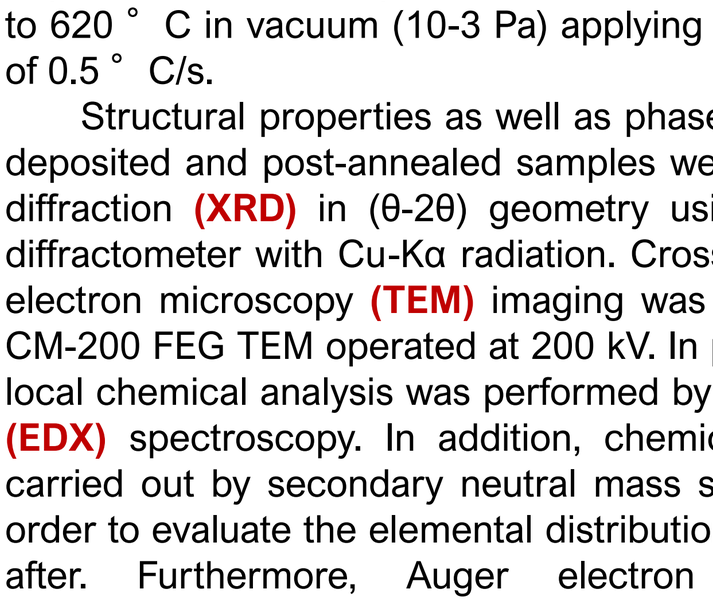

Figure 1 presents TEM cross-section images of the as-deposited Pt/Tb/Fe stack obtained in dark (a) and bright (b) field mode.

Figure 1. *Cross-section TEM images of the as-deposited Pt/Tb/Fe trilayer obtained in (a) dark and (b) bright field mode; TEM images and corresponding EDX maps (c, d) after deposition and after post-annealing at (e, f) 280 °C, (g,h) 500 °C, and (i, j) 550 °C.*

Results

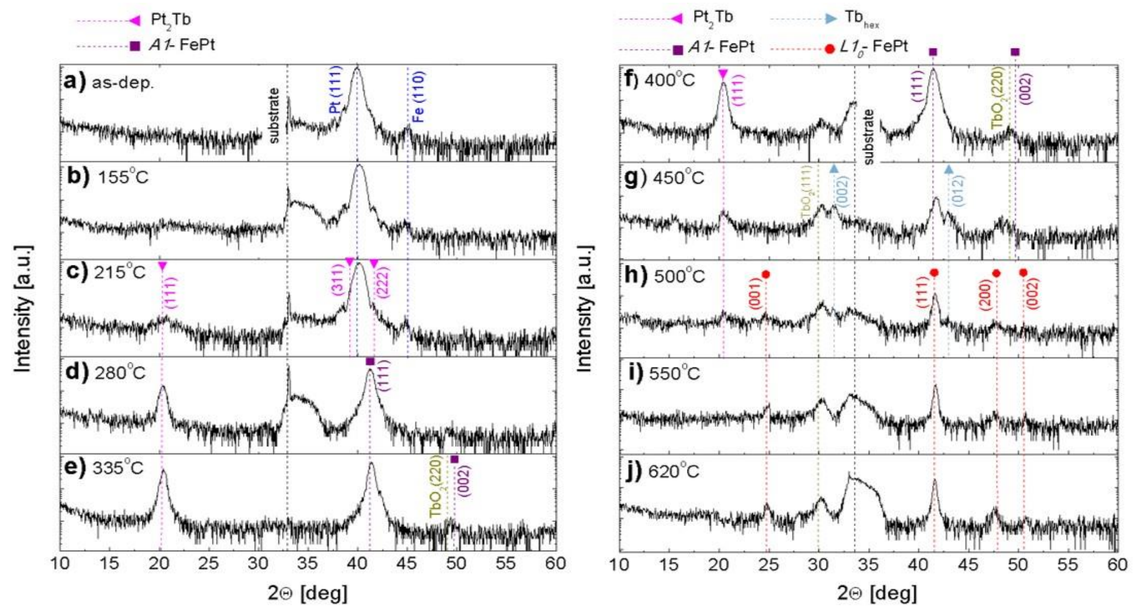

Figure 2. *XRD Θ–2Θ patterns of Pt/Tb/Fe films (a) after deposition and (b-j) after post-annealing up to different temperatures as labelled.*

From the analysis of the experimental results the following sequence of phase transitions is found in Pt/Tb/Fe trilayers upon annealing:

Pt/Tb/Fe (deposited)

$\xrightarrow{215\,^{\circ}C}$ Pt + Pt_2Tb + Fe

$\xrightarrow{280\,^{\circ}C}$ TbO_2 + Pt_2Tb + *A1*-FePt

$\xrightarrow{450\,^{\circ}C}$ TbO_2 + Tb + Pt_2Tb + *A1*-FePt + $L1_0$-FePt

$\xrightarrow{500\,^{\circ}C}$ TbO_2 +Tb + Pt_2Tb + $L1_0$-FePt

$\xrightarrow{550\,^{\circ}C}$ TbO_2 + $L1_0$-FePt.

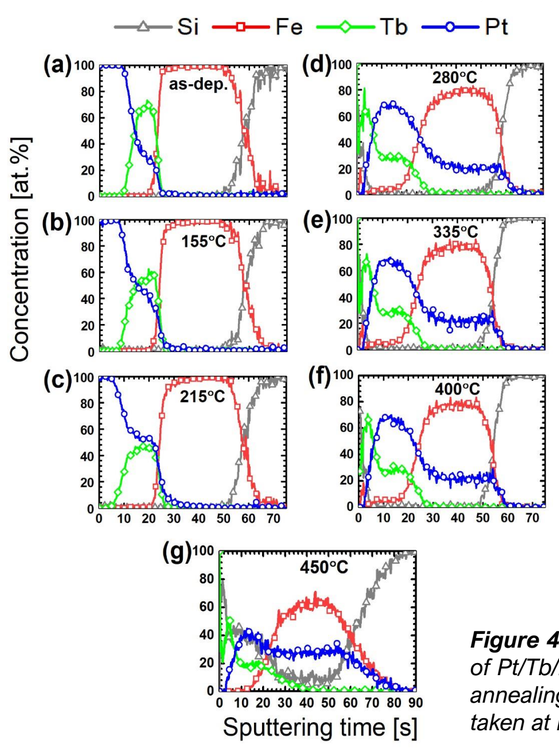

Figure 4. *SNMS composition profiles versus sputtering time of Pt/Tb/Fe trilayers (a) after deposition and (b-g) after post-annealing to different temperatures as labelled. Profiles were taken at room temperature.*

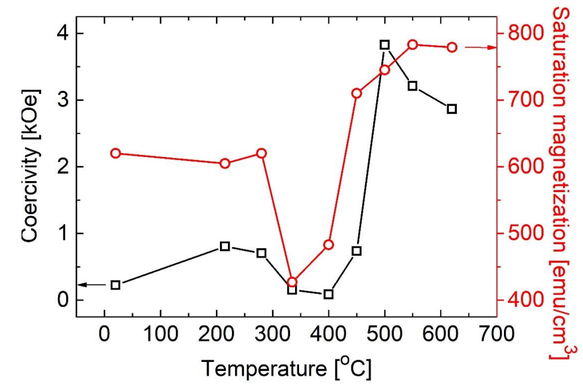

Figure 3. *Coercivity and saturation magnetization of Pt/Tb/Fe trilayers as a function of annealing temperature.*

Conclusion

In this study, phase transitions in Pt/Tb/Fe trilayers occurring during post-annealing up to 620 °C were investigated by various techniques including x-ray diffraction, transmission electron microscopy, secondary neutral mass spectrometry, Auger electron spectroscopy depth profiling, and magnetic properties measurements. At an annealing temperature of 215 °C pronounced Pt and Tb diffusion intermixing is observed leading to the formation of the binary Pt_2Tb phase. Further annealing up to 280 °C is accompanied by the appearance of the chemically disordered *A1*-FePt phase and significant Tb segregation to the outer surface forming an oxide layer. The chemically ordered L10-FePt phase starts to form in parallel to the disappearance of the Pt_2Tb phase at 450 °C. The final phase product at 620 °C is characterized by the coexistence of two remaining phases, $L1_0$-FePt and TbO_2.

Acknowledgment

This work was financially supported by the German Research Foundation (DFG Grant number AL618/34-1) and by the GINOP-2.3.2-15-2016-00041 project co-financed by the European Union and the European Regional Development Fund.

NANO-2020, Lviv, Ukraine, 2020

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