Structural studies of thin Mg films

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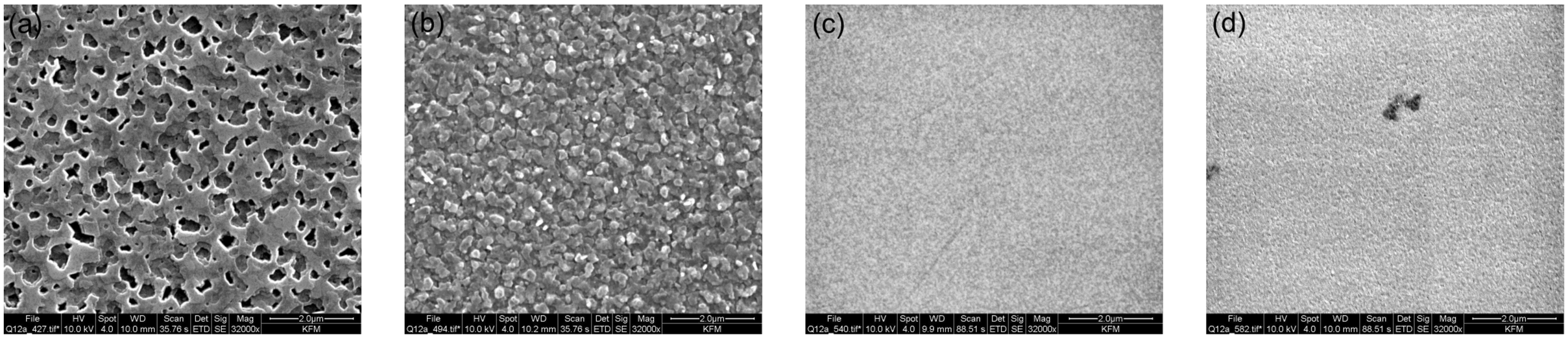
Introduction

* Mg is a perspective material suitable for hydrogen storage due to high hydrogen content (7.6 wt.%) in magnesium hydride, MgH_2. However kinetics of hydrogen sorption is sluggish due to low hydrogen diffusivity in MgH_2 ("Blocking effect").
* High density of grain boundaries typical for thin films may lead to improvement of hydrogen sorption rate. Hence Mg films microstructure and its dependence on deposition conditions is important to be studied.

Experimental

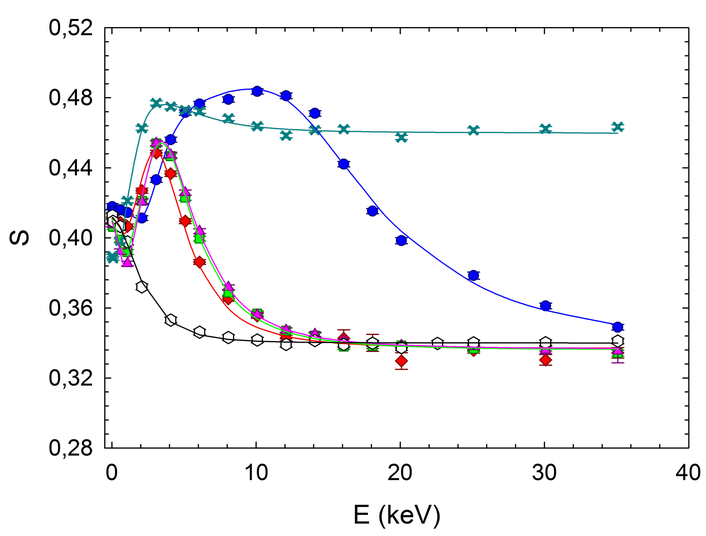
- Mg films was prepared by RF (13.6 MHz) magnetron sputtering.
- Fused silica, Si (100) and sapphire (0001) substrates were used.
- Films were deposited at two different temperatures (RT and 300°C), two different thicknesses ($\sim$ 1 μm and $\sim$ 100 nm) were prepared.
- 20 nm thick Pd cap was deposited on each film.
- SEM: FEI Quanta 200 scanning electron microscope.
- XRD: X-ray difractometer X'Pert PANalytical with Co anode and X'Celarator detector.
- VEPAS investigations were performed on a magnetically guided variable energy slow positron beam SPONSOR.

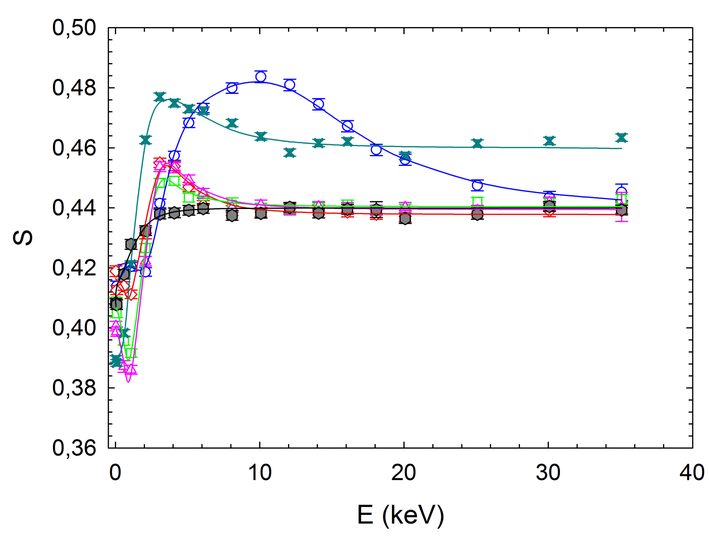
SEM micrographs



SEM micrographs (secondary electrons) of Mg films deposited on fused silica substrate. (a) deposited at RT (1540 nm thickness, mean grain size $\sim$ 410 nm); (b) deposited at 300°C (156 nm thickness, mean grain size $\sim$ 240 nm); (c) deposited at RT (121 nm thickness, mean grain size $\sim$ 70 nm); (d) deposited at RT and subsequently annealed at 300°C for 1 h (123 nm thickness, mean grain size $\sim$ 90 nm).
Films deposited on Si substrate exhibit virtually the same grain size as the films on fused silica. Films deposited on sapphire substrate exhibit always smaller grains compared to the films deposited on fused silica.

S-parameter curves and results of fitting

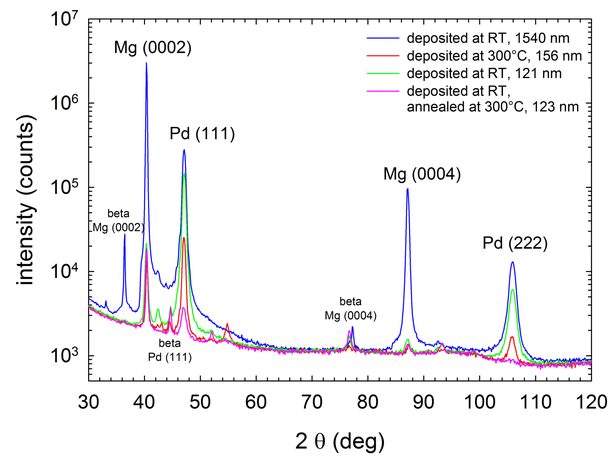




Dependence of the S parameter on positron energy for Mg films, sapphire and fused silica substrate and annealed bulk Mg. All data were fitted by VEPFIT code. For each Mg film S parameter, diffusion length L_+ and density of dislocations ϱ_D were calculated.

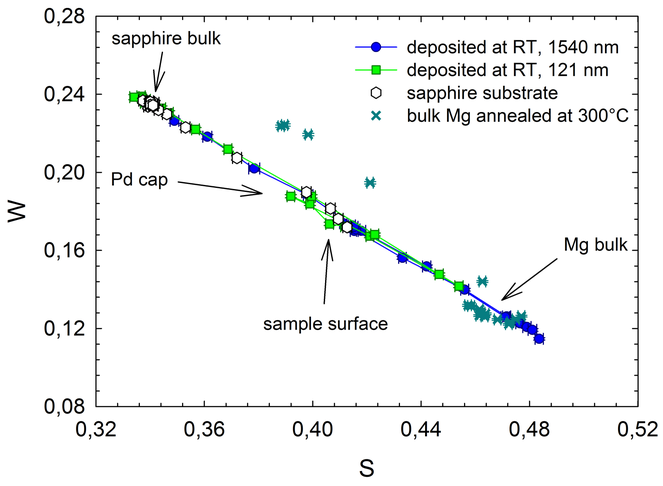
thickness	T_{dep}	T_{ann}	substrate	L_+ (nm)	S	ϱ_D ($10^{14}\mathrm{m}^{-2}$)
1540 nm	RT	–	sapphire	37(2)	0.4878(4)	12(2)
1540 nm	RT	–	fused silica	40(1)	0.4843(4)	10(1)
156 nm	300°C	–	sapphire	60(5)	0.483(2)	4.3(7)
156 nm	300°C	–	fused silica	77(7)	0.480(2)	2.4(9)
121 nm	RT	–	sapphire	35(4)	0.485(2)	14(3)
121 nm	RT	–	fused silica	39(5)	0.484(2)	11(4)
123 nm	RT	300°C	sapphire	50(5)	0.483(2)	6.4(8)
123 nm	RT	300°C	fused silica	52(6)	0.482(2)	5.9(9)
–	–	250°C	bulk Mg	196(9)	0.4634(5)	–

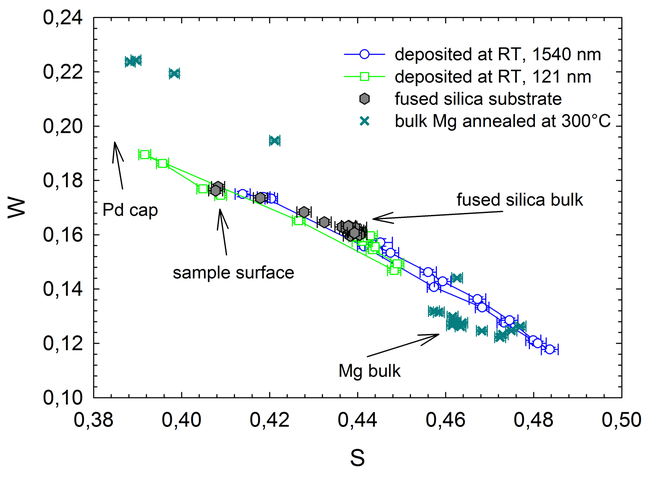
XRD diffractograms



XRD spectra of Mg films on fused silica. Only visible diffractions* came from Mg (0001) plane and Pd (111) plane. Films on sapphire (0001) and Si (100) has similar diffractograms with extra substrate peak.
* "Beta" peaks are caused by not completely filtered Co K_β line.

S-W plots





Conclusions

- A set of 4 different Mg films deposited on 3 different substrates was prepared by RF magnetron sputtering.
- Films deposited on fused silica and Si substrate exhibit larger grain size and lower concentration of defects than films deposited on sapphire substrate.
- Mean grain size increases with thickness of film. Most of defect presented in the film occurs at the interface of the film and the substrate.
- Mg films are strongly textured with preferred orientation of (0001) plane parallel to the surface.
- Films deposited at 300°C have larger grains and lower concentration of defects than films deposited at room temperature.
- Annealing at 300°C for 1 hour of films deposited at room temperature causes coarsening of grains and lowers the concentration of defects.