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D2.1 Report on multilayer stacks fabrication

Responsible NCSRD (D. NIARCHOS)

Dissemination Level Public (PU)

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1 RES 1	CNRS Spintec	CNRS	France
2 RES 2	Catalan Institute of Nanotechnology	ICN	Spain
3 RES 3	Karlsruher Institut für Technologie	KIT	Germany
4 RES 4	National Center for Scientific Research Demokritos	NCSR D	Greece
5 RES 5	CEA LETI	LETI	France
6 IND 1	In Silicio	INSIL	France
7 IND 2	Singulus	SING	Germany
8 OTHER 1	Toplink Innovation	TLI	France
9 RES 6	Eidgenössische Technische Hochschule Zürich	ETHZ	Switzerland
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TAB 1	Micron Technology	MICRON	Italy
TAB 2	Tower Semiconductor	TOWER	Israel
TAB 3	European Nanoelectronics Infr. for Innovation	ENI2	Europe

Work programme topics addressed

Objective ICT-2011.3.1: Very advanced nanoelectronic components: design, engineering, technology and manufacturability

a) "Beyond CMOS technology"

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Abstract

The goal of WP2 is the fabrication of **single memory cells** and **cell arrays**. The first step for fabricating memory cells is the deposition of **continuous multilayer stacks** with appropriate structural and magnetic properties. Such stacks have been **sputter-deposited** by NCSRD and Singulus. NCSRD is focused on using **W** as spin-orbit coupling material (seed layer), while Singulus is focused on **Ta**. After deposition, the stacks are characterized structurally and magnetically. The deposition tool used at NCSRD is a **AJA ATC-2200-V** system. Singulus is using a **Singulus Timaris** platform.

During the first year, NCSRD has been focused in depositing W/CoFeB/MgO half Magnetic Tunnel Junctions (MTJs). Singulus has been depositing Ta/CoFeB/MgO half MTJs and Ta/CoFeB/MgO/CoFeB/Ta MTJs. Structural and magnetic characterization has been performed from both partners. Singulus has obtained perpendicular magnetization stacks after annealing of the as-deposited samples. NCSRD has obtained partial perpendicular anisotropy of the stacks and the annealing studies are continued in order to obtain homogeneous perpendicular anisotropy.

Section A: Fabrication
NCSRD

Substrates used : 100 mm wafers, Si/SiO_2 (500 nm)

Deposition technique: ultra high vacuum (base pressure $1{\cdot}10^{-9}$ Torr) magnetron sputtering using a AJA ATC-2200-V. It has been verified that uniformity across the wafers is ±3%. Metals (W and $Co_{20}Fe_{60}B_{20}$) have been deposited using DC bias, while oxides (MgO and AlOx) have ben deposited using RF bias. Ar deposition pressure is between 2 and 3 mTor and the substrate remains at room temperature. All substrates used where Si(001) wafers with a 500 nm thick thermal oxide. The DC power density on the CoFeB target was 1.2 Watt/cm^2 leading to a typical growth rate of 0.01 nm/sec.

Samples: NCSRD has been focused in depositing half-MTJs with W seed layer.

<u>Series A</u>: W/$Co_{20}Fe_{60}B_{20}$/MgO without AlOx capping and a W/$Co_{20}Fe_{60}B_{20}$/AlOx sample. These wafers have been characterized and there is no clear indication that there is perpendicular magnetic anisotropy (measurements below).
<u>Series B</u>: W/$Co_{20}Fe_{60}B_{20}$/MgO/AlOx samples. These wafers are currently being characterized by XRD/XRR, AHE and MOKE magnetometry (LPS, Orsay). Finally, these wafers have been used for micropatterning double Hall crosses.

There is an ongoing study on the effect of annealing on the structural and magnetic properties of the stacks. Annealing has been performed in UHV (pressure range 10^{-7} to 10^{-8} Torr).

Below there is a detail list of the fabricated samples:

Sample Code	Structure	Comments
	SERIES A	
SP61	W(6)/$Co_{20}Fe_{60}B_{20}$ (1) /AlOx (2)	AlOx capping
SP61A300	Annealing at 300^{o}C for 90 min	
SP61A350	Annealing at 350^{o}C for 90 min	
SP62	W(6)/$Co_{20}Fe_{60}B_{20}$ (1)/MgO(2)	
SP62A300	Annealing at 300^{o}C for 90 min	
SP62A350	Annealing at 350^{o}C for 90 min	
SP62A350A	Annealing at 350^{o}C for 90 min	
SP62A400	Annealing at 400^{o}C for 90 min	
SP63	W(6)/$Co_{20}Fe_{60}B_{20}$(0.5)/MgO(2)	0.5 nm CoFeB

SP63A350	Annealing at 350^oC for 90 min	
SP63A400	Annealing at 400^oC for 90 min	
SP64	W(6)/$Co_{20}Fe_{60}B_{20}$ (1)/MgO(2) – subst. pre-etch.	
SERIES B		
SP65	W(6)/$Co_{20}Fe_{60}B_{20}$ (1)/MgO(2)/AlOx(2) – subst. pre-etch.	
SP65A250	UHV annealing at 250^oC for 30 min	
SP65A300	UHV annealing at 300^oC for 30 min	
SP65A350	UHV annealing at 350^oC for 30 min	
SP66	W(3)/$Co_{20}Fe_{60}B_{20}$ (1)/MgO(2)/AlOx(2) – subst. pre-etch.	
SP67	W(9)/$Co_{20}Fe_{60}B_{20}$ (1)/MgO(2)/AlOx(2) – subst. pre-etch.	
SP68	W(12)/$Co_{20}Fe_{60}B_{20}$ (1)/MgO(2)/AlOx(2) – subst. pre-etch.	
SP69	W(15)/$Co_{20}Fe_{60}B_{20}$ (1)/MgO(2)/AlOx(2) – subst. pre-etch.	
SP76	W(6)/$Co_{20}Fe_{60}B_{20}$ (1)/MgO(2)/AlOx(2) – subst. pre-etch.	MgO @ 10 mTorr
SP77	W(6)/$Co_{20}Fe_{60}B_{20}$ (2)/MgO(2)/AlOx(2) – subst. pre-etch.	
SP78	W(6)/$Co_{20}Fe_{60}B_{20}$ (0.5)/MgO(2)/AlOx(2) – subst. pre-etch.	
SP78A250	UHV annealing at 250^oC for 30 min	
SP78A300	UHV annealing at 300^oC for 30 min	
SP78A350	UHV annealing at 350^oC for 30 min	
SP79	W(6)/$Co_{20}Fe_{60}B_{20}$ (1.5)/MgO(2)/AlOx(2) – subst. pre-etch.	
SP79A250	UHV annealing at 250^oC for 30 min	
SP79A300	UHV annealing at 300^oC for 30 min	
SP79A350	UHV annealing at 350^oC for 30 min	
SP80	W(6)/$Co_{20}Fe_{60}B_{20}$ (2.5)/MgO(2)/AlOx(2) – subst. pre-etch.	
SP81	W(6)/$Co_{20}Fe_{60}B_{20}$ (x)/MgO(2)/AlOx(2) – subst. pre-etch.	no CoFeB rotation (graded thickness)

Section A: Fabrication
Singulus

Substrates used : 50, 100, and 200 mm thermally oxidized Si wafers.

Deposition technique: The depositions were performed on the Singulus 10 target TIMARIS production platform using Linear Dynamic Deposition (LDD) by means of dc- and rf-magnetron sputtering (dc: all metallic materials, rf: ceramic MgO). The sputtering tool is characterized by outstanding reproducibility wafer to wafer and excellent uniformity (better $1\sigma<5\%$) of the key parameter resistance area (RA) product and tunnel magneto resistance (TMR) on 300mm wafers. Additionally, the sputtering concept gives the consortium the possibility to fabricate wedge layers with thickness controlled by software.

Samples: Singulus AG deposited MTJs using Ta seed layer. During the reporting term $Fe_{60}Co_{20}B_{20}$ / MgO / $Fe_{60}Co_{20}B_{20}$ MTJs with top hard magnetic Co/Pd multilayer were optimized using CIPT and vibrating sample magnetometer (VSM) on site by Singulus. The critical parameters that have been optimized are:

- the thickness range of the FeCoB layers to achieve perpendicular magnetization (Fig 1),
- the stacking sequence of the hard magnetic multilayer (Fig 2),
- the coupling of the hard magnetic multilayer to the top FeCoB layer (Fig 3),
- optimum annealing temperature (Fig 4). (annealing performed at Singulus site)

Based on these results, 61 MTJs with and without hard magnet with different Ta seed as well as wedge structures have been provided to Spintec and LETI on 50 mm, 100 mm and 200 mm wafers for patterning and additional investigations. Some of those structures had been annealed at Singulus at 240°C for 1h before sending to Spintec and LETI. For an overview see the following tables:

simple MTJs
20 Ta / 1.0 $Fe_{60}Co_{20}B_{20}$ / t MgO / 1.5 $Fe_{60}Co_{20}B_{20}$ / 5 Ta / 7 Ru

Wafer size	1.50 MgO		1.22 MgO
	w/- anneal	w/o anneal	w/o anneal
100mm	1	2	3
50mm	2	3	4
pieces	-	1	-

half MTJs with Ta seed variation
3-15 Ta / t $Fe_{60}Co_{20}B_{20}$ / 1.22 MgO / 1 Ru

Wafer size	0.8 FCB	1.0 FCB	
100mm	3	3	w/o anneal

simple MTJs with wedge free layer
20 Ta / 0.8-1.3 $Fe_{60}Co_{20}B_{20}$ / t MgO / 1.5 $Fe_{60}Co_{20}B_{20}$ / 5 Ta / 7 Ru

Wafer size	MgO 1.5nm	MgO 1.0nm	
200mm	1	1	w/o anneal
pieces	1	-	w/o anneal

MTJs with wedge free layer and in plane pinned top electrode
t Ta / 0.8-1.3 $Fe_{60}Co_{20}B_{20}$ / 1.5 MgO / 2.3 $Fe_{60}Co_{20}B_{20}$ / 0.85 Ru / 2.1 $CoFe_{30}$ / 20 PtMn / 5 Ta / 7 Ru

Wafer size	20Ta buffer	5Ta buffer	
200mm	1	1	w/o anneal

MTJs with Co/Pd hard magnet
t Ta / 1.0 $Fe_{60}Co_{20}B_{20}$ / 1.5 MgO / 1.3 $Fe_{60}Co_{20}B_{20}$ / 0.3 Ta / 5x [1.0 Pd / 0.3 Co] / 5 Ta / 7 Ru

Wafer size	20 Ta		10 Ta		5 Ta		
	w/- anneal	w/o anneal	w/- anneal	w/o anneal	w/- anneal	w/o anneal	
200mm	-	2	-	2		2	with 100nm Ta cap
100mm	2	2	-	-	2	2	
50mm	3	2	-	-	3	2	
pieces	-	1	-	-	-	-	

MTJs with Co/Pd hard magnet with wedge free layer different seeds
t Ta / 0.8-1.3 $Fe_{60}Co_{20}B_{20}$ / 1.5 MgO / 1.3 $Fe_{60}Co_{20}B_{20}$ / 0.3 Ta / 5x [1.0 Pd / 0.3 Co] / 5 Ta / 7 Ru

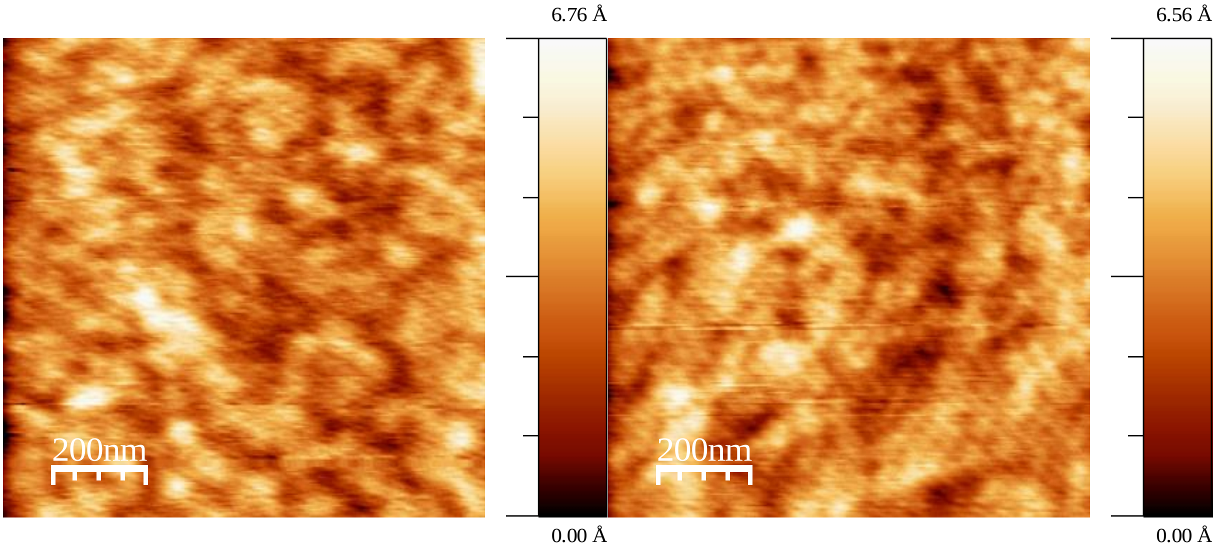
wafer size	5 Ta	20 Ta	5 Ta / 30 CuN /20 Ta	5 Ta / 30 CuN / 5 Ta	
200mm	2	2	1	1	w/o anneal

Section B: Characterization
NCSRD

Stacks have been characterized structurally (XRD, AFM) and magnetically (SQUID, Kerr magnetometry).

AFM

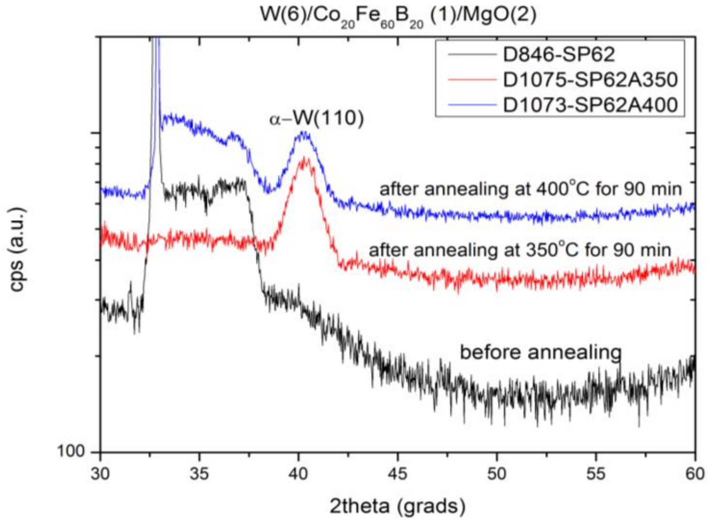
Atomic Force Microscopy was performed using a Veeco CP-II microscope and commercial AFM probes by Veeco.



AFM images of a Si / SiO2(500 nm) / W(6 nm) / CoFeB(1 nm) / MgO(2nm) / AlOx(2nm), before (left) and after (right) annealing at 350°C for 30 min. The RMS roughness is extremely low in both cases (0.1 nm), close to the sensitivity of the instrument.

XRD

XRD spectra where obtained using a Siemens D500 diffractometer with Cu-Kα radiation.



XRD spectra of a W(6 nm) / CoFeB (1 nm) / MgO (2 nm) sample, before and after annealing. The peak above 40^o corresponds to the α-W(110) planes. As-deposited samples have β-W structure, which transforms to α-W after annealing. The rest of the layers cannot be detected at our setup (new setup to arrive soon).

SQUID

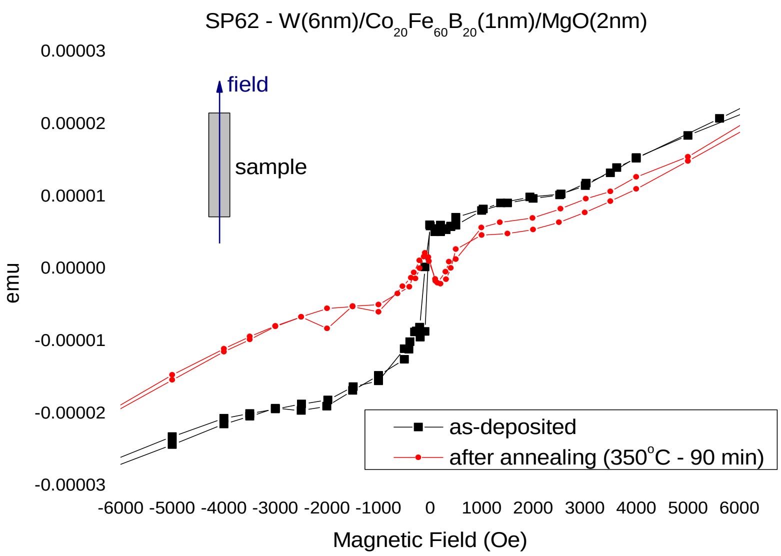


Figure: SQUID measurement (in-plane applied field). As-deposited: in-plane easy axis. After annealing (90 min at 350 deg): in-plane hard axis?

MOKE

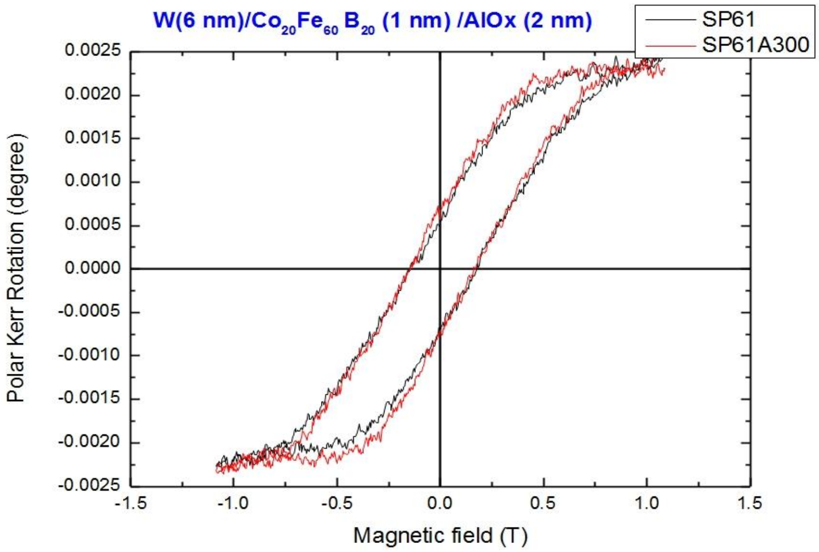


Figure: Polar Kerr magnetometry: there is no difference before and after annealing (90 min at 300 deg). The hysteresis cycles are open, but not square, indicating a mixed in- and out-of plane magnetization.

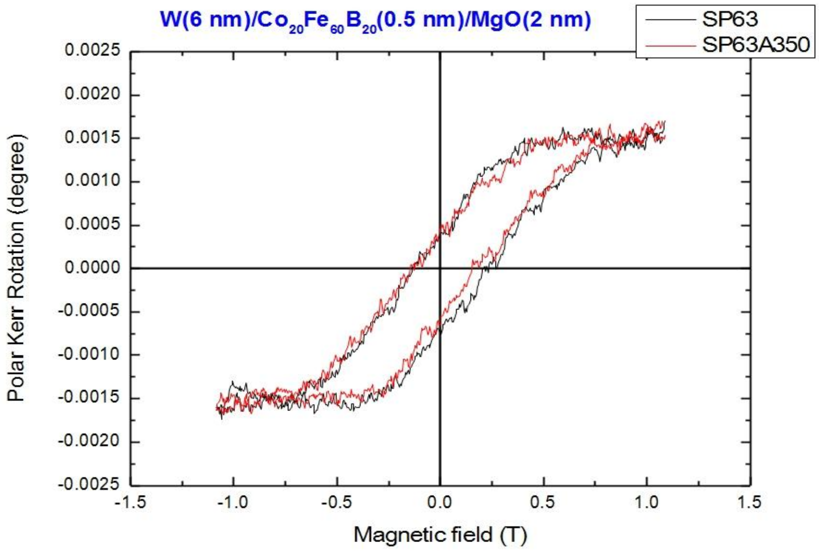


Figure: Polar Kerr magnetometry: there is no difference before and after annealing (90 min at 350 deg). As before, the hysteresis cycles indicate a mixed in- and out-of plane magnetization.

Section B:Characterization

Singulus

During the optimization runs the wafers deposited at the Singulus TIMARIS had been annealed and characterized magnetically as well as electriclally using VSM and CIPT measurements on Singulus site. Key results are shown below:

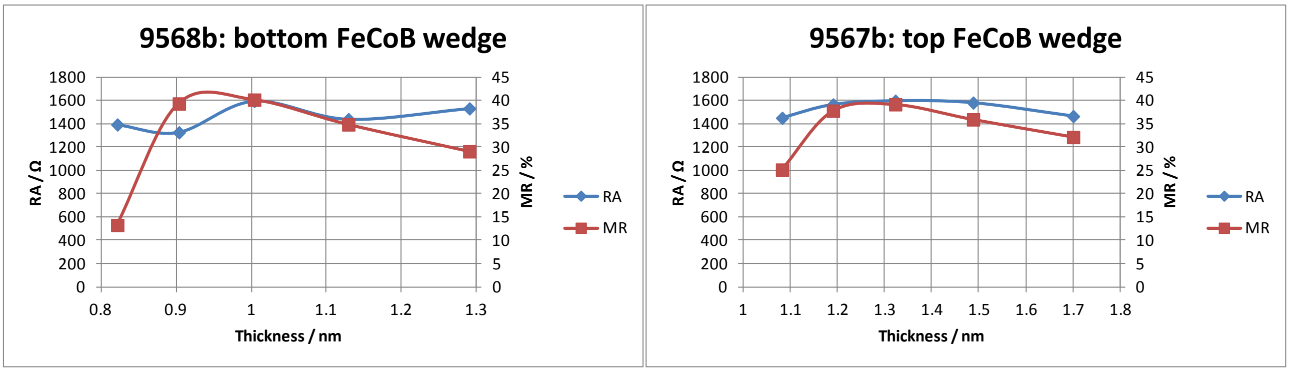


Figure: Out of plane TMR and RA values on 20 Ta / $Fe_{60}Co_{20}B_{20}$ / 1.50 MgO / $Fe_{60}Co_{20}B_{20}$ / 0.35 Ta / 5x [1.0 Pd / 0.3 Co] / 5 Ta / 7 Ru layer stack deposited on thermally oxidized Si annealed at 240°C for 1h (all thickness in nm). For the bottom FeCoB wedge the thickness of the top layer was fixed at 1.3nm for the top FeCoB wedge the thickness of the bottom layer was fixed at 1.0nm. Measurments are done using CIPT on unpatterned wafers.

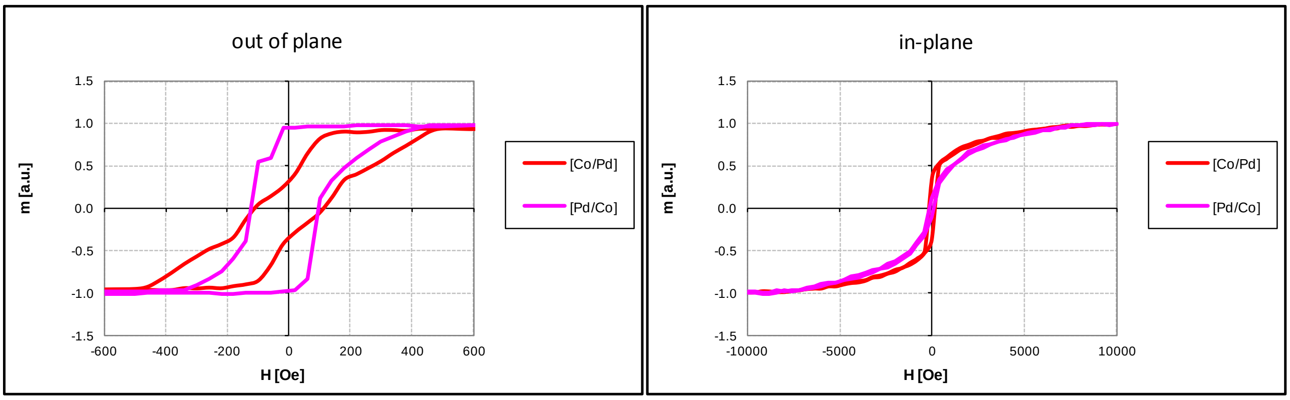


Figure: Out of plane and in-plane magnetic switching curves (VSM) with different stacking of the hard magnetic multilayer [ML]: 20 Ta / 1.0 $Fe_{60}Co_{20}B_{20}$ / 1.50 MgO / 1.3 $Fe_{60}Co_{20}B_{20}$ / 0.35 Ta / 5x [ML] / 5 Ta / 7 Ru layer stack deposited on thermally oxidized Si annealed at 300°C for 1h (all thickness in nm). Co was 0.3 nm Pd was 1.3nm. Layers using the [0.3 Co / 1.0 Pd] stacking showed no TMR.

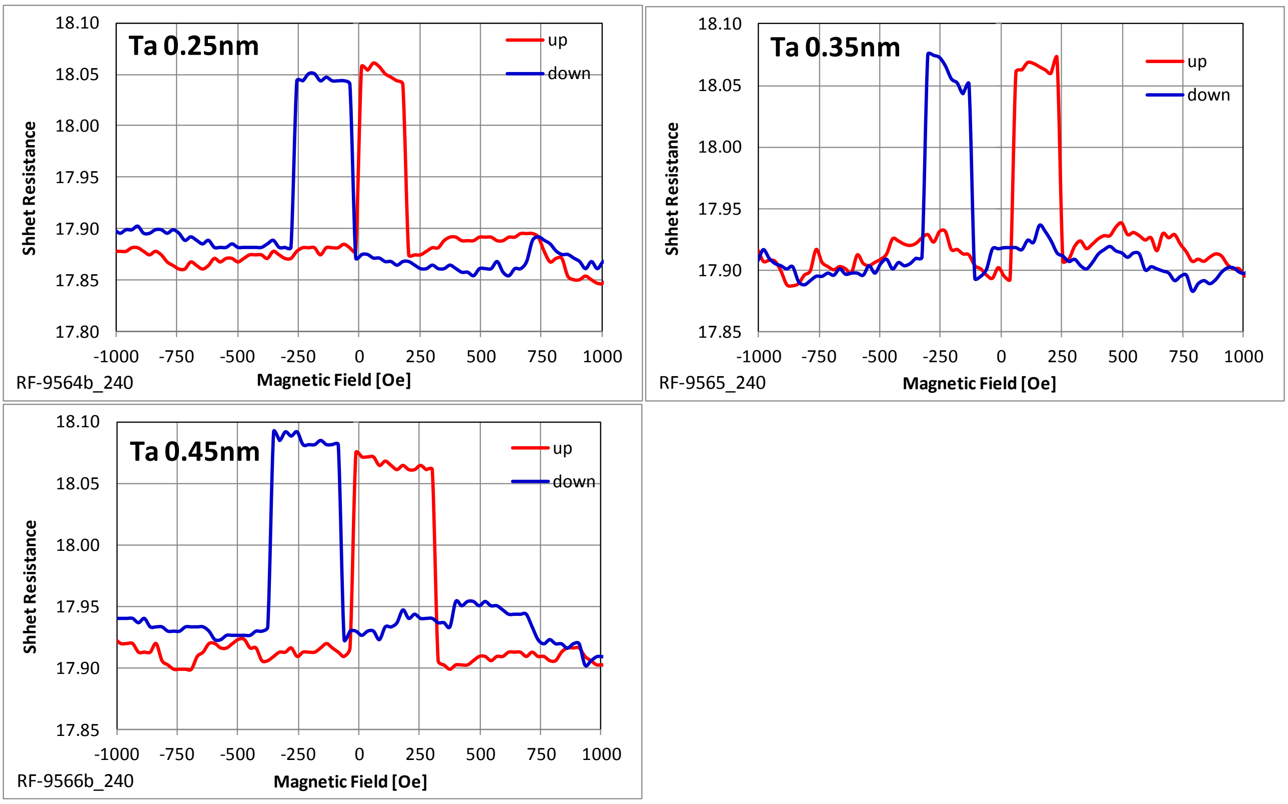


Figure: CIPT minor loops (field scans) in out-of-plane direction on 20 Ta / 1.0 $Fe_{60}Co_{20}B_{20}$ / 1.50 MgO / 1.3 $Fe_{60}Co_{20}B_{20}$ / t Ta / 5x [1.0 Pd / 0.3 Co] / 5 Ta / 7 Ru layer stack deposited on thermally oxidized Si annealed at 240°C for 1h (all thickness in nm). Measurements are done using CIPT on unpatterned wafers. Best switching is seen for a Ta thickness of 0.35nm. TMR values are comparable 36% with RA = 1.4kΩµm²

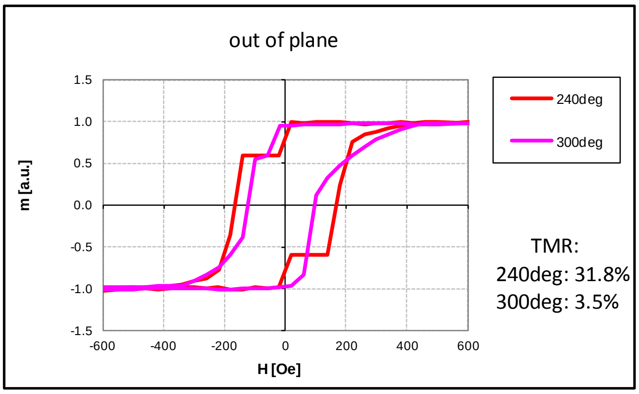


Figure: Out-of-plane field scans (VSM) on 20 Ta / 1.0 $Fe_{60}Co_{20}B_{20}$ / 1.50 MgO / 1.3 $Fe_{60}Co_{20}B_{20}$ / 0.27 Ta / 5x [1.0 Pd / 0.3 Co] / 5 Ta / 7 Ru layer stack deposited on thermally oxidized Si annealed at 240°C and 300°C for 1h (all thickness in nm).

Conclusion

Task 2.1 (Material growth and characterization) of WP2-SPOT is scheduled to last for the first 24 months of the project. The partners involved (Singulus, NCSRD, ICN, LETI) have to develop materials to meet the requirement for simultaneously maximizing SOT and TMR.

Within this framework, the objective of NCSRD and Singulus is to develop MTJ stacks that exhibit perpendicular anisotropy. During the first year of the project, Singulus has already obtained perpendicular anisotropy MTJ stacks utilizing a hard magnetic Co/Pt pinning on Ta seed layer. These layers show TMR. NCSRD has obtained mixed anisotropy MTJ stacks using W as seed layer. A strong materials development effort is ongoing at NCSRD for obtaining purely perpendicular anisotropy MTJs.