

Specimen preparation and control over magnetic fields for CHIRALTEM experiments

2006-04-19

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1. Creation of Fe specimens on GaAs membranes
2. Specimen preparation by FIB
3. Switching of objective lens current on Tecnai microscopes
4. Summary/Prospect



Motivation: requirements for CHIRALTEM specimens

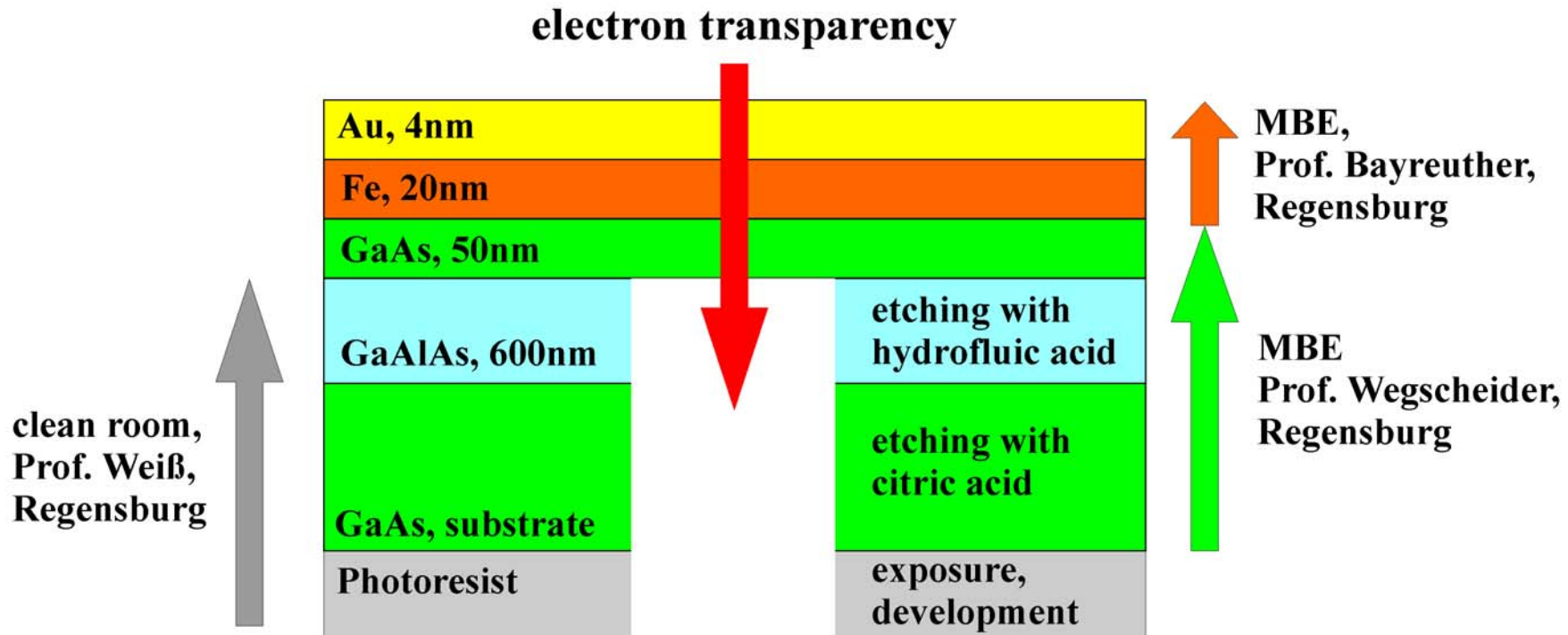
The „ideal“ CHIRALTEM specimen has to be:

- electron transparent
- ferromagnetic
- single crystal
- without thickness variations
- without background (membranes, etc.)
- large enough for XMCD



1. Creation of Fe specimen on GaAs membrane

Epitaxial growth of Fe on electron transparent GaAs membrane



1. Creation of Fe specimen on GaAs membrane

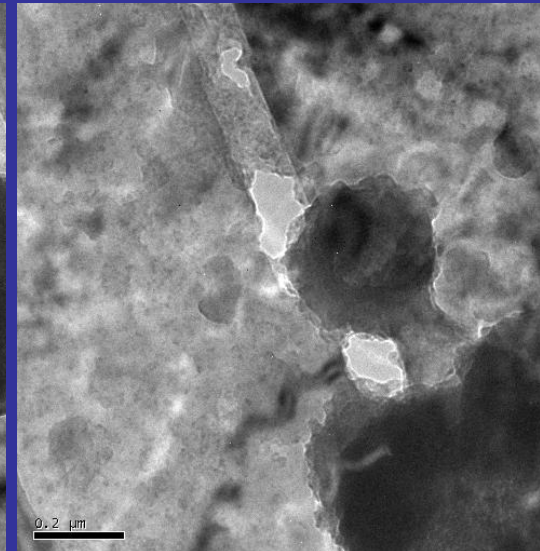
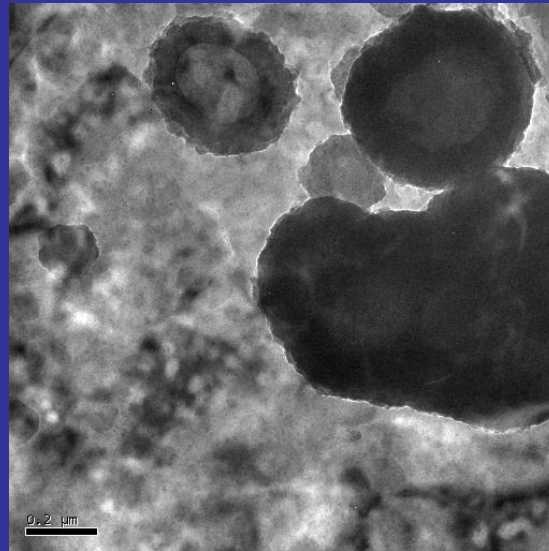
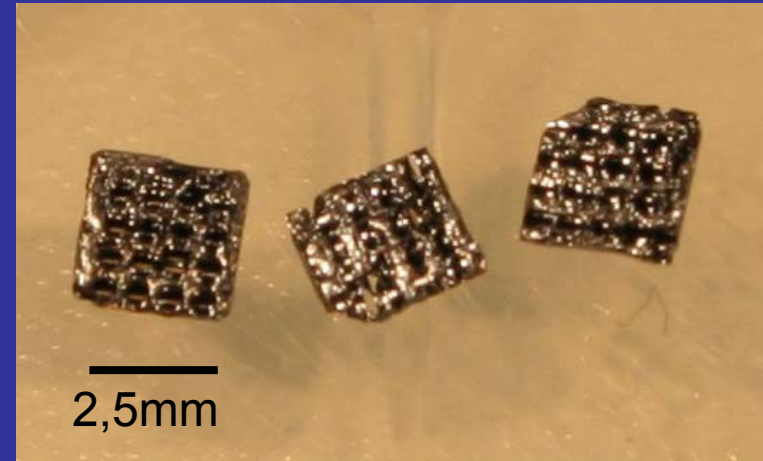
Theory:

- + epitaxial growth
- + every desired Fe layer thickness possible
- + thickness variation only a few monolayers

Reality:

- huge variations in thickness of GaAs-membranes
- specimens very brittle, not transportable
- diffraction pattern of GaAs dominant

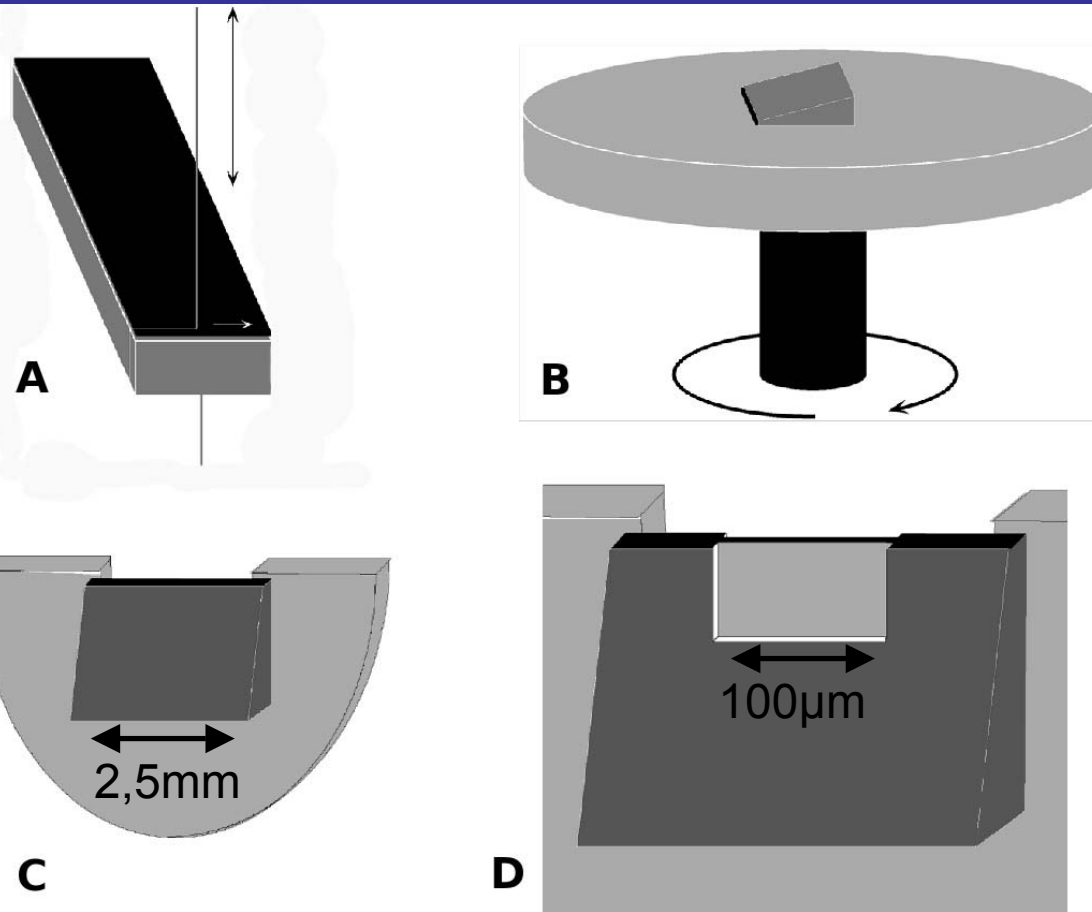
**No specimens for
CHIRALTEM!**



2. Specimen preparation by FIB

New attempt for CHIRALTEM specimen:

FIB preparation will be done in Bremen (Prof. Rosenauer, Dr. Kroeger)



Preparation steps:

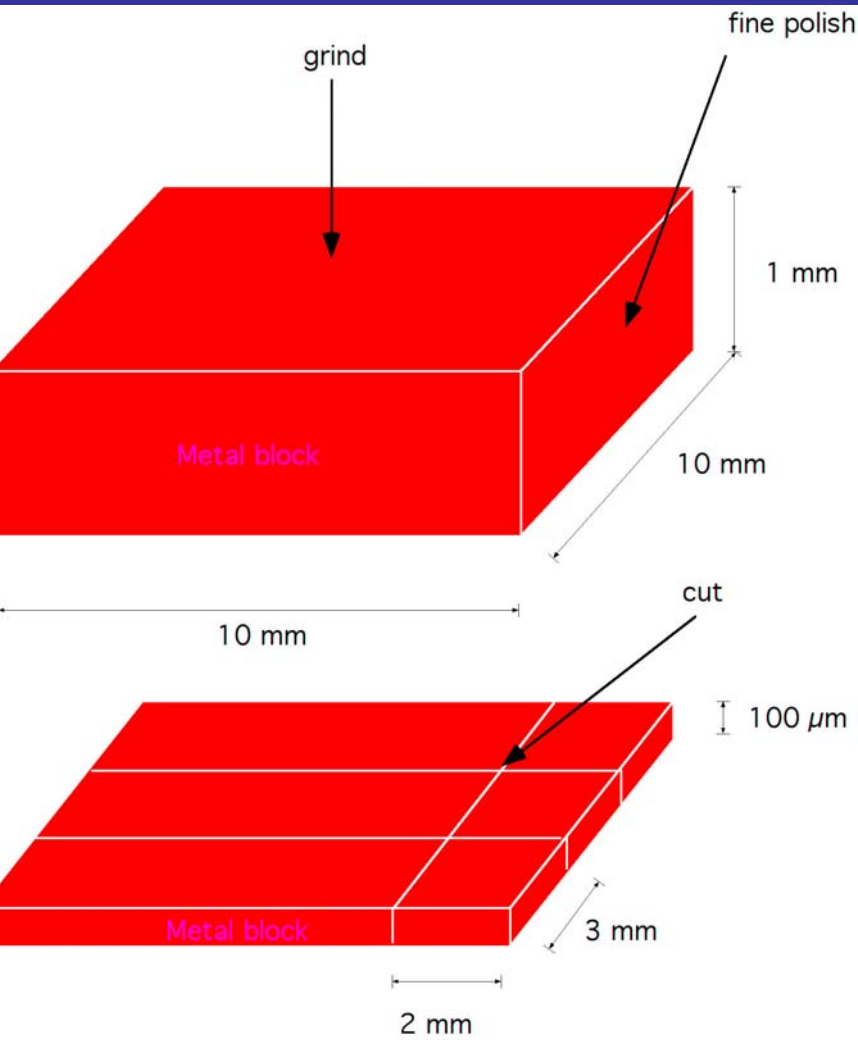
A – cutting a bulk monocrystal (e.g. Co), black side is finely polished

B - polishing on tripod

C - mounting on metal ring

D - cutting a window with focused ion beam (FIB)

2. Specimen preparation by FIB



Desired advantages:

- monocrystal specimen
- electron transparent
- flat specimen surface
- large enough even for XMCD

Possible disadvantages:

- modification of the crystal surface
- thickness not exactly predetermined
- expensive technique

Adequate monocrystals have already been cut and polished in Regensburg and have been sent to Bremen.

-> first feedback right ahead!

3. Switching of objective lens current on Tecnai microscopes



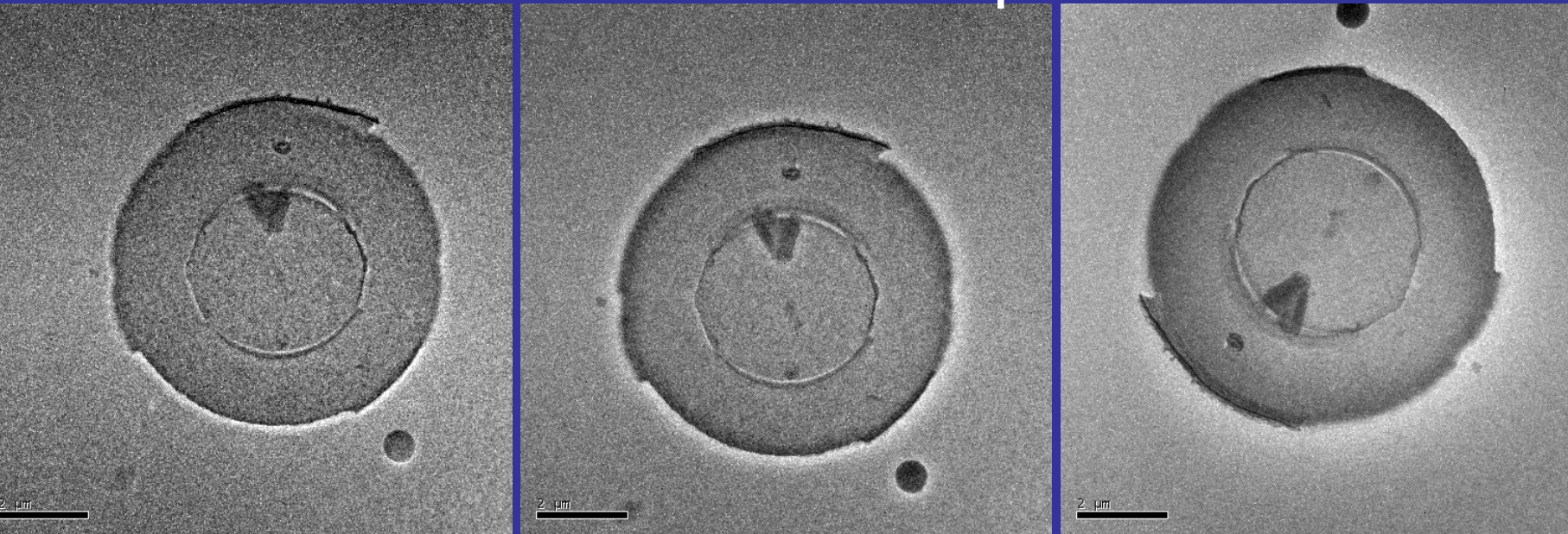
Dichroic effect should be reversed by inverted magnetisation ->
so commutate the objective lens !!



First try on CM30 with Lorentz lens:

- Replacing the objective lens power supply with external power supply
- reversal of the external power supply easily possible...

3. Switching of objective lens current on Tecnai microscopes



Permalloy dot (diameter about $8\mu\text{m}$):

left: SA3900x standard imaging mode,

middle: external power supply on objective lens at 11.49A,

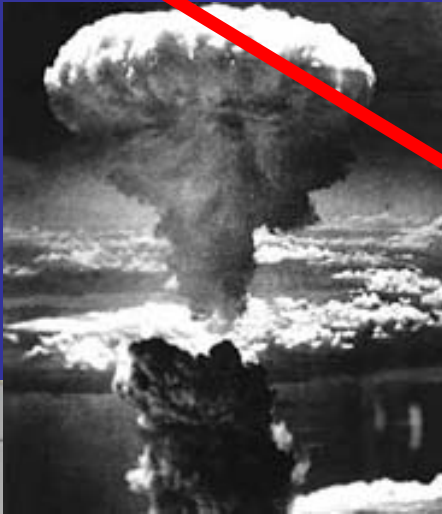
right: external power supply at -11.49A , C2 lens manually adjusted

First results on the CM30 -> necessity of improvements

3. Switching of objective lens current on Tecnai microscopes

Next approach: **TECNAI F30**

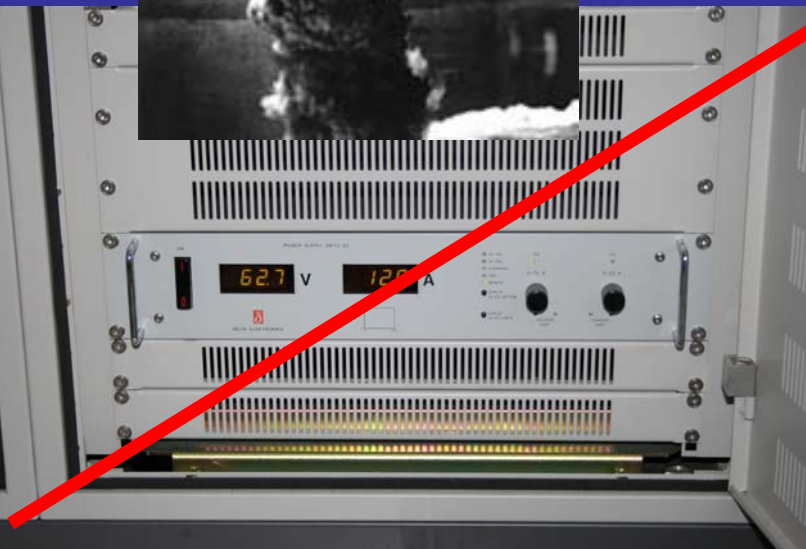
Commutation of the objective lens right after the (origin) power supply



vicious circle: TEM measures voltage at OL

-> commutation -> voltage at OL to low -> raise voltage

-> (measured) voltage gets lower -> raise voltage ->



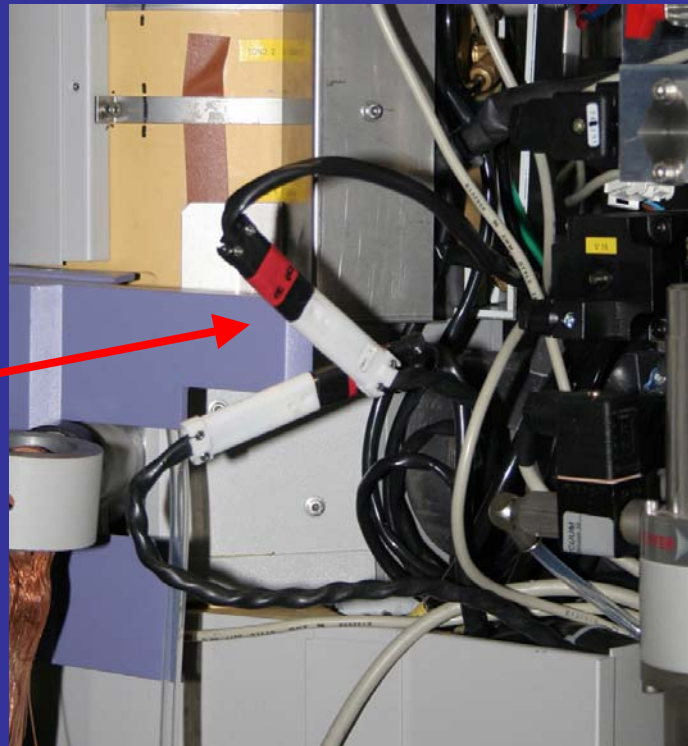
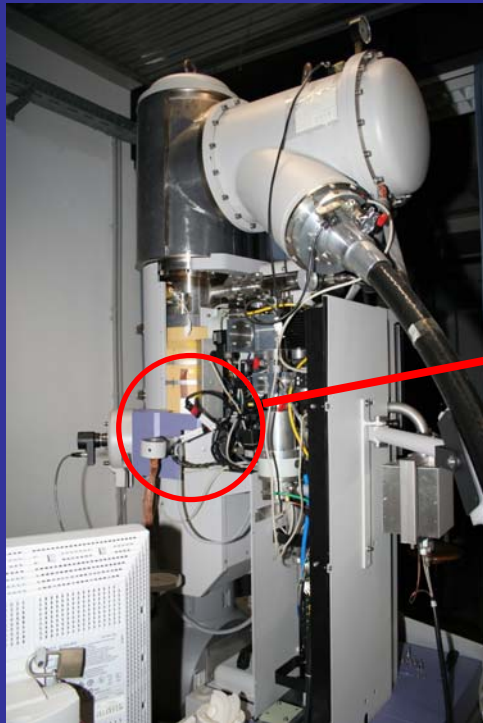
3. Switching of objective lens current on Tecnai microscopes

Next approach: **TECNAI F30**

~~Commutation of the objective lens right after the (origin) power supply~~

Better solution:

Commutation of both objective lens coils right at the coils!

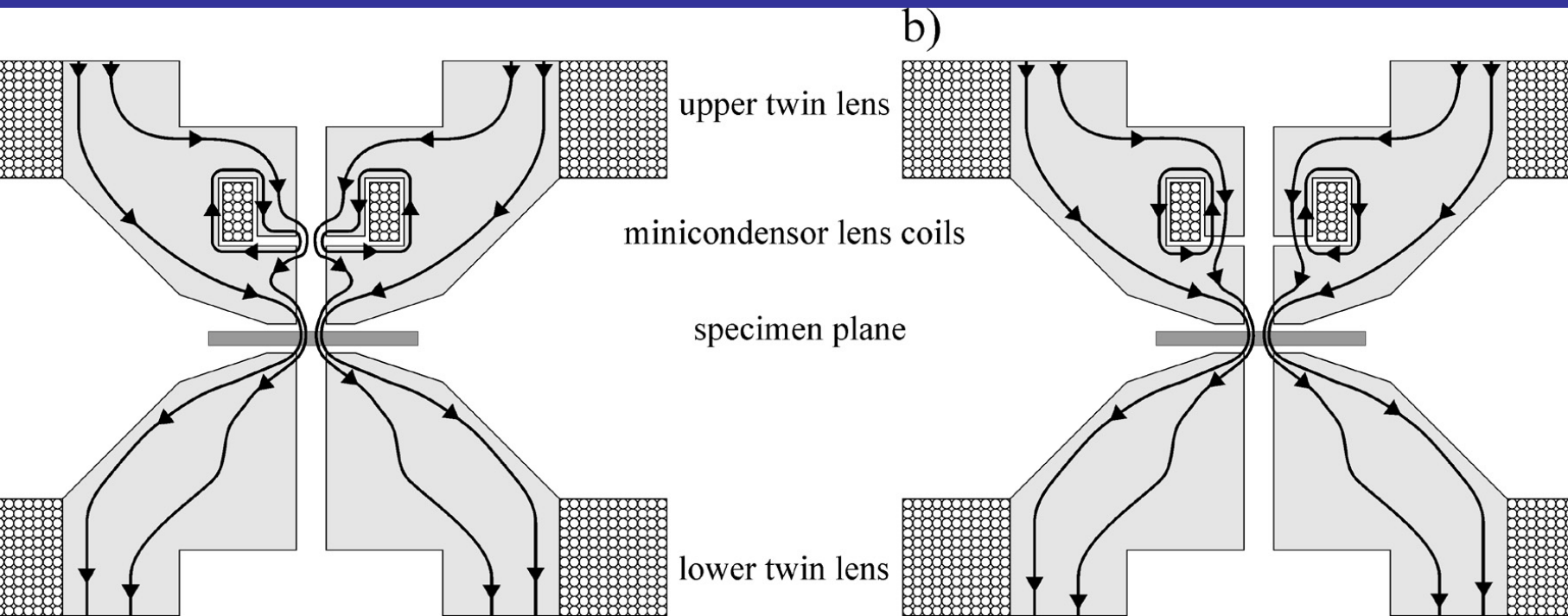


Installation of an adapter for manual commutation

objective lens has to be deactivated while switching !!!

3. Switching of objective lens current on Tecnai microscopes

Problem: Reversing OL current deactivates function of the minicondensor lens (C3)!
-> this is equivalent to switching from microprobe to nanoprobe



a) OL system in microprobe mode

b) OL system in nanoprobe mode

3. Switching of objective lens current on Tecnai microscopes

Problem: Reversing OL current deactivates function of the minicondenser lens (C3)!
-> equivalent switching from microprobe to nanoprobe

Possibility: switching to nanoprobe after commutation -> „normal“ TEM-image again

But: different alignment in nanoprobe mode, not all steps for alignment available

microprobe

System Status

Lens					
Spot size	29.20 %				
Intensity	50.02 %				
Minicondenser	92.75 %				
Objective	94.38 %				
Lorentz	0.00 %				
Diffraction	73.81 %				
Intermediate	39.51 %				
Projector 1	99.03 %				
Projector 2	100.00 %				

Gun deflector					
	X	Y	Perp X	Perp Y	All
Gun tilt	-0.0046	0.5787	U-X	0.0255	
Gun shift	0.0125	-0.2595	U-Y	0.3770	
Spot-dep. shift	0.0236	-0.0147	L-X	0.0303	
Gun tilt pp	5.5550	2.0994	L-Y	0.3054	
Gun shift pp	2.4200	2.3900	0.0246	-0.0276	L-Y

Beam deflector					
	X	Y	Perp X	Perp Y	All
Df tilt	0.0000	0.0000	U-X	-0.0071	
User shift	-0.0210	-0.1071	U-Y	-0.1015	
Rot Center	0.0018	0.1902	L-X	0.0007	
Align shift	0.0269	0.0943	L-Y	0.1815	
Beam tilt pp	3.6058	3.8344	-0.0219	-0.0034	
Beam shift pp	5.0960	5.1050			

Image deflector					
	X	Y	Perp X	Perp Y	All
Image-Beam shift	0.0000	0.0000	U-X	-0.1889	
User diff. shift	0.0000	0.0000	U-Y	0.0121	
User image shift	0.0000	0.0000	L-X	0.3581	
Align diff. shift	0.3537	-0.0221	L-Y	-0.0253	
Align image shift	0.3612	-0.0225			
Diff. shift pp	3.5160	3.5238	-0.0182	0.0550	
Image shift pp	3.5256	3.5552			
Meagn. corr.	0.0042	0.0032			
Det. alignment	0.0000	0.0000			
X-over corr.	0.0000	0.0000			

SA 66000 x
TEM Brightfield

nanoprobe

System Status

Lens					
Spot size	29.20 %				
Intensity	49.40 %				
Minicondenser	-92.75 %				
Objective	94.52 %				
Lorentz	0.00 %				
Diffraction	73.81 %				
Intermediate	39.51 %				
Projector 1	99.03 %				
Projector 2	100.00 %				

Gun deflector					
	X	Y	Perp X	Perp Y	All
Gun tilt	-0.0046	0.5787	U-X	0.0255	
Gun shift	0.0125	-0.2595	U-Y	0.3770	
Spot-dep. shift	0.0236	-0.0147	L-X	0.0303	
Gun tilt pp	5.5550	2.0994	L-Y	0.3054	
Gun shift pp	2.4200	2.3900	0.0246	-0.0276	L-Y

Beam deflector					
	X	Y	Perp X	Perp Y	All
Df tilt	0.0000	0.0000	U-X	0.0979	
User shift	-0.0192	-0.0326	U-Y	-0.0255	
Rot Center	0.1406	0.1138	L-X	-0.1135	
Align shift	-0.2191	-0.0557	L-Y	-0.0025	
Beam tilt pp	4.3495	4.3391	0.0147	-0.0181	
Beam shift pp	4.1401	4.1341			

Image deflector					
	X	Y	Perp X	Perp Y	All
Image-Beam shift	0.0000	0.0000	U-X	-0.1857	
User diff. shift	0.0000	0.0000	U-Y	0.0149	
User image shift	0.0000	0.0000	L-X	0.3516	
Align diff. shift	0.3474	-0.0275	L-Y	-0.0306	
Align image shift	0.3612	-0.0225			
Diff. shift pp	3.5160	3.5238	-0.0182	0.0550	
Image shift pp	3.5256	3.5552			
Meagn. corr.	0.0042	0.0032			
Det. alignment	0.0000	0.0000			
X-over corr.	0.0000	0.0000			

SA 66000 x
Nanoprobe

3. Switching of objective lens current on Tecnai microscopes

Problem: Reversing OL current deactivates function of the minicondenser lens (C3)!
-> equivalent switching from microprobe to nanoprobe

~~Possibility: switching to nanoprobe after commutation -> „normal“ TEM-image~~

**Best solution: concurrent commutation of the objective
lens and the minicondenser lens (C3)!**

3. Switching of objective lens current on Tecnai microscopes

Best solution: commutation also of the minicondensor lens (C3)!

Problem: minicondensor lens coil have no connectors at the colum...



... found it on the dual distribution board in the TEM cabinet!

manual commutation possibility

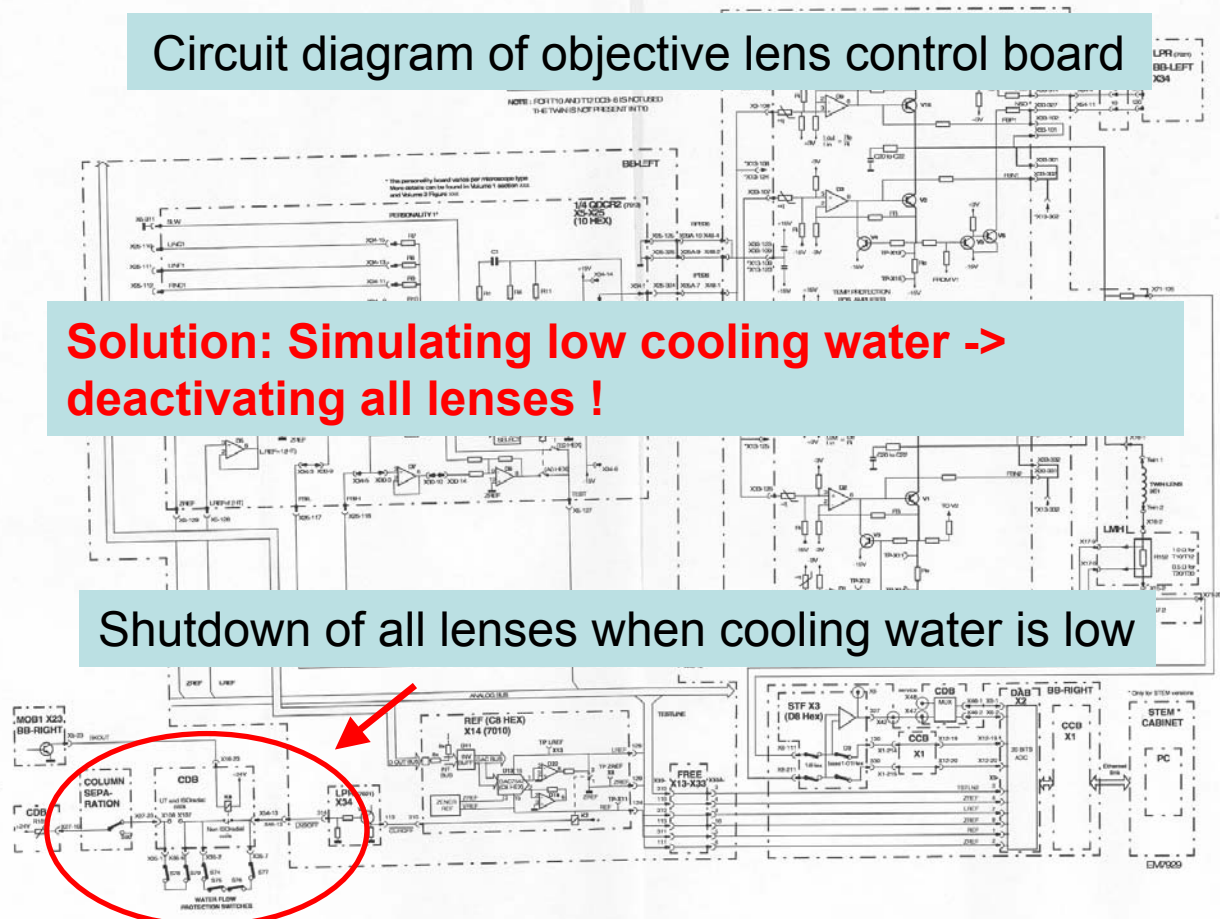
3. Switching of objective lens current on Tecnai microscopes

Problem: all lenses must be deactivated when commutating (using Lorentz-mode in Regensburg, not available in Vienna!)

Circuit diagram of objective lens control board

Solution: Simulating low cooling water -> deactivating all lenses !

Shutdown of all lenses when cooling water is low

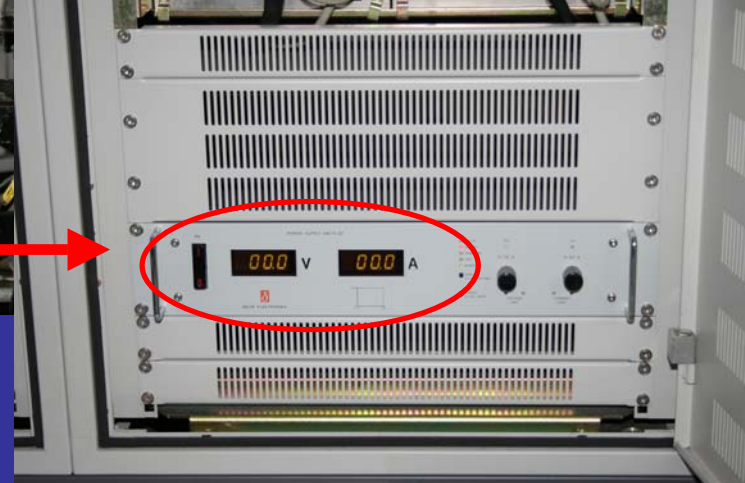
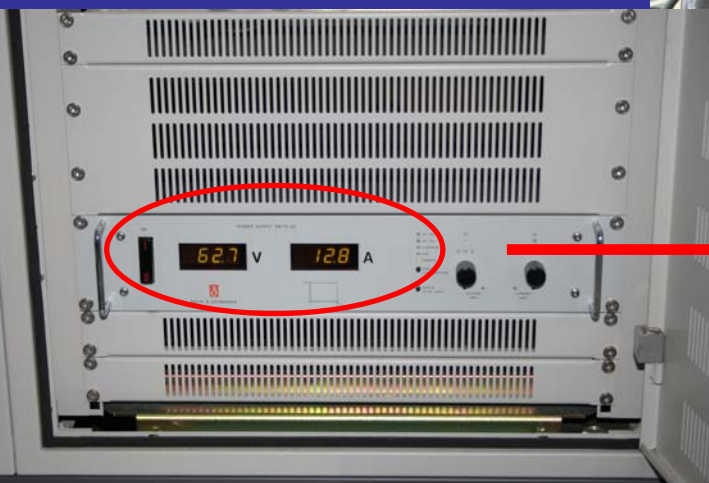
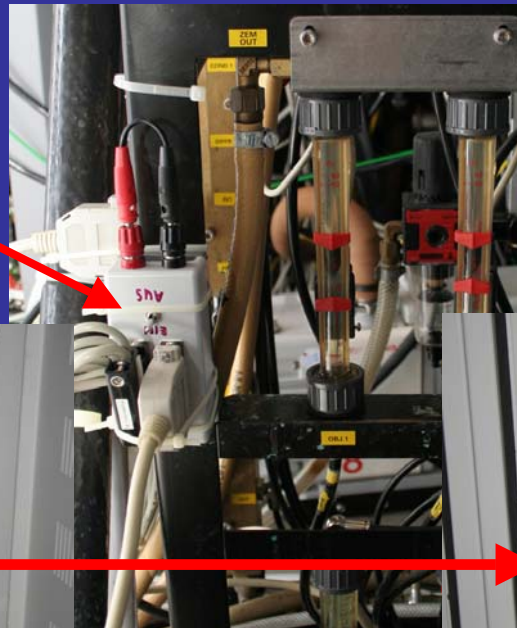


3. Switching of objective lens current on Tecnai microscopes

Problem: all lenses must be deactivated when commutation (using Lorentz-mode in Regensburg, not available in Vienna!)

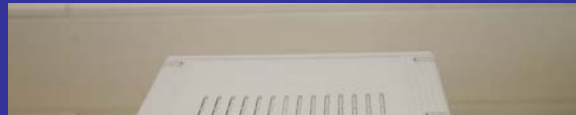
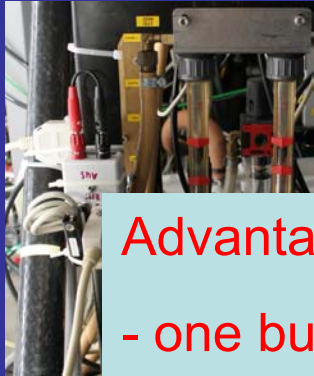
Solution: Simulating low cooling water -> deactivating all lenses !

Switch for all lenses, prepared for remote shutdown



3. Switching of objective lens current on Tecnai microscopes

Automatic unit for commutation of objective lens and minicondenser lens (prototype):



Advantages:

- one button for complete commutation cycle
- maloperation not possible
- system will be installed in a few hours (no wires to be cut!)
- system can be uninstalled in half an hour

1. All le

ens



2. Reversing obj. lens

4. All lenses on again

3. Switching of objective lens current on Tecnai microscopes

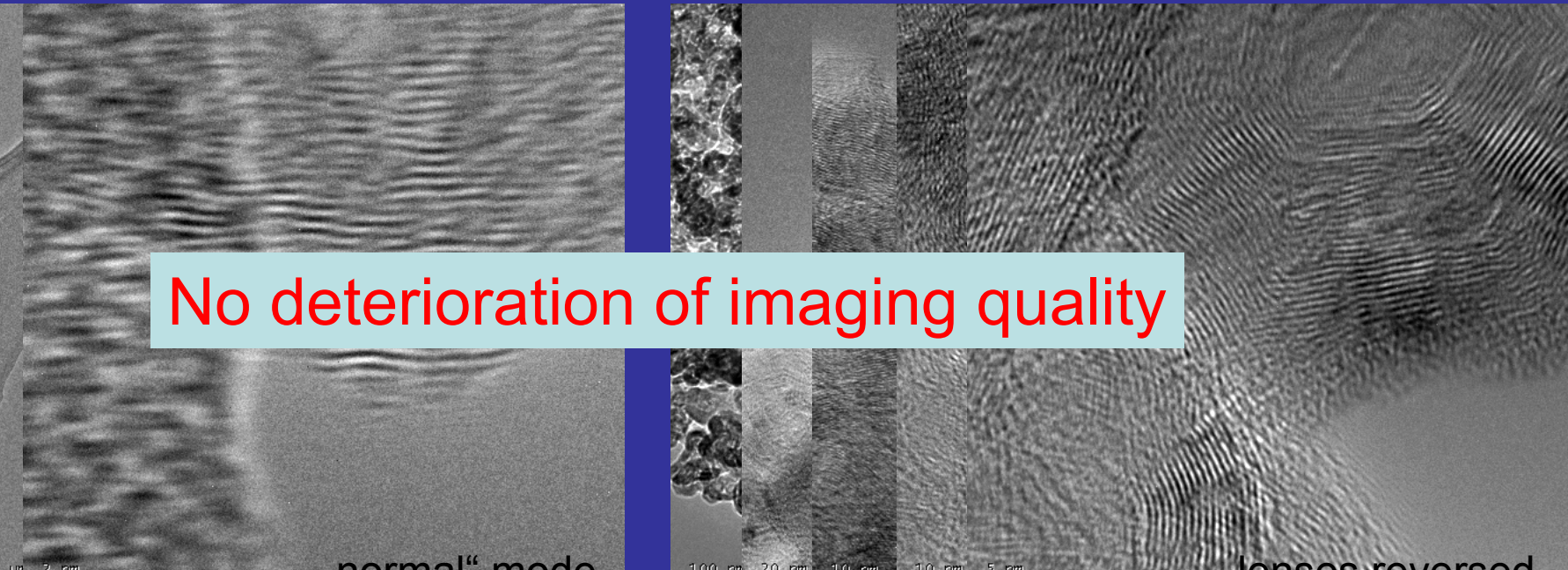
What about the image quality with commutated lenses?

3. Switching of objective lens current on Tecnai microscopes

Performance of the Tecnai microscope after commutation

-> Need for a separate alignment, focus, illumination, eucentric height, specimen area has to be adjusted manually

Example for high resolution: graphit particles (fringe distance 0,34nm)

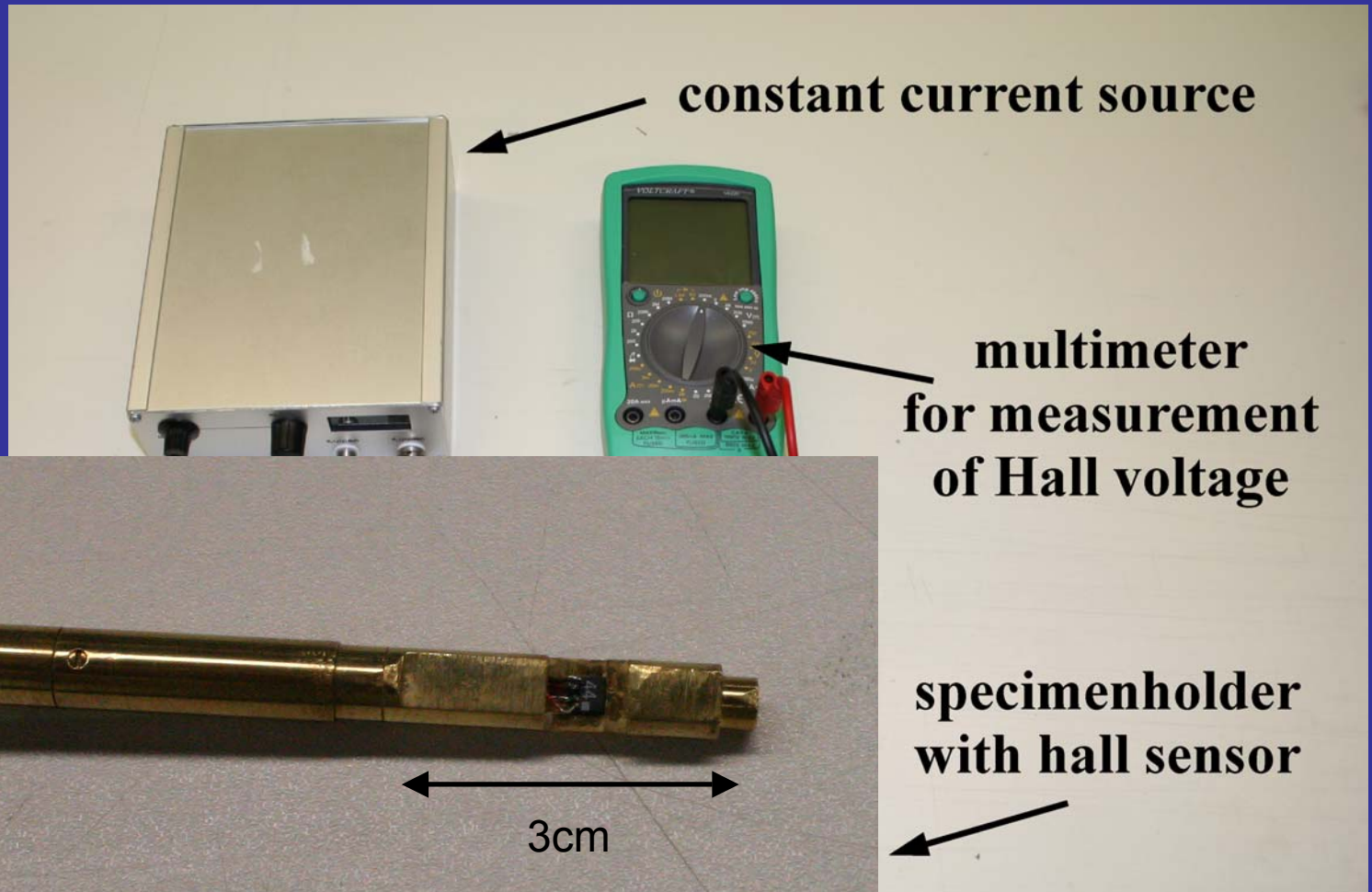


3. Switching of objective lens current on Tecnai microscopes

Is there any change after a commutation cycle?

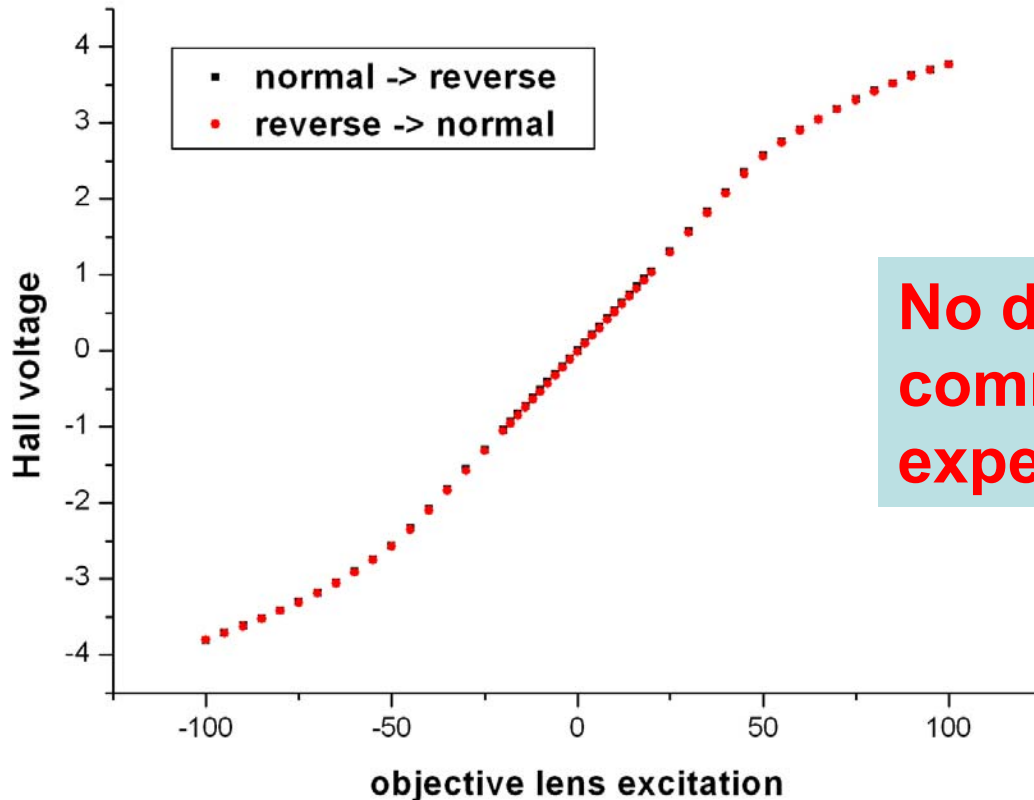
3. Switching of objective lens current on Tecnai microscopes

Measurement of the magnetic field in the specimen plane during the commutation cycle (object. lens AND C3 lens):



3. Switching of objective lens current on Tecnai microscopes

Measurement of the magnetic field in the specimen plane during the commutation cycle (object. lens AND C3 lens):



No hysteresis detectable

No disadvantages after commutation cycle expected!

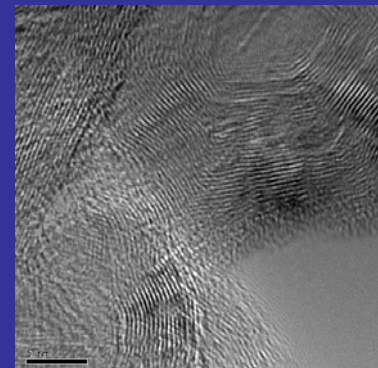
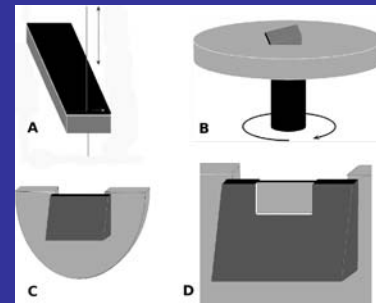
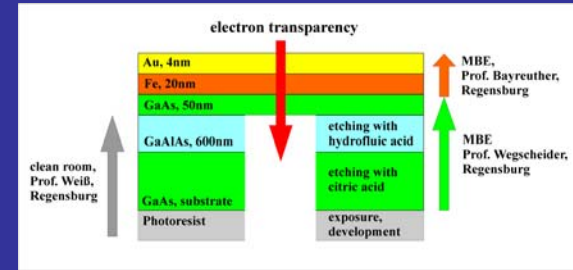
4. Summary / Prospect



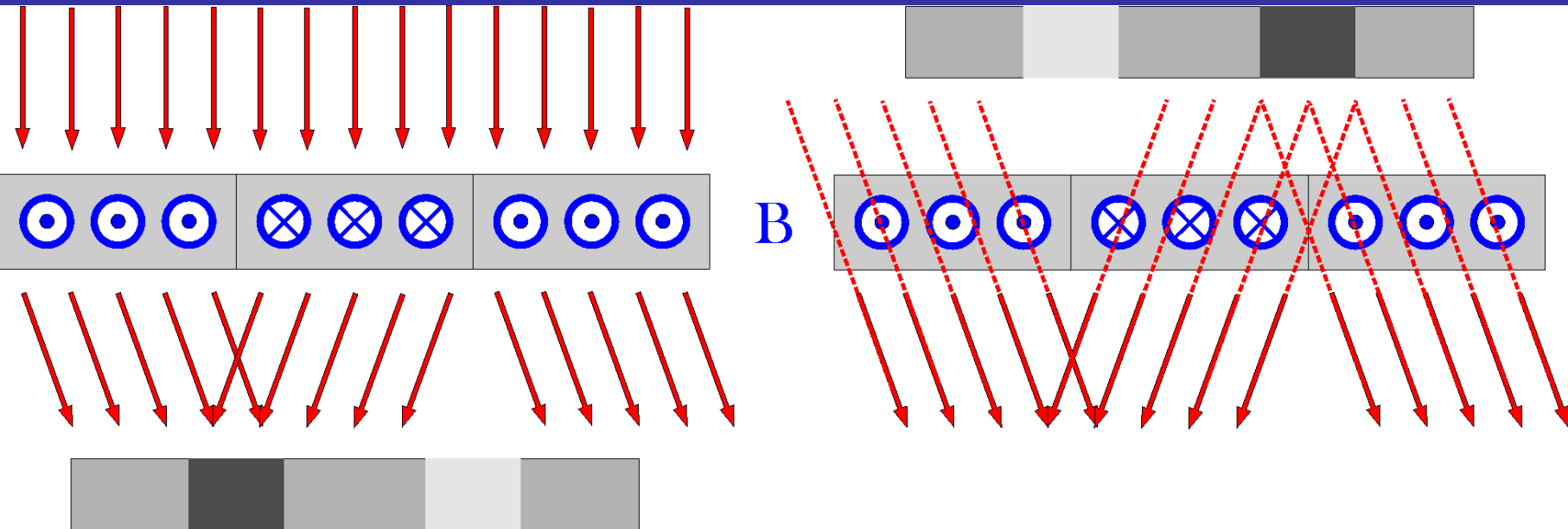
- Fe on GaAs probably not useful for CHIRALTEM...

- First CHIRALTEM specimen produced by FIB is just beeing prepared

- Objective lens switching procedure is working, switching unit will be available upon delivery of the connectors by FEI



Function of Lorentz microscopy



Fresnel imaging in overfocus...

... and in underfocus