JST-DFG workshop on Nanoelectronics
January 22, 2009, Karasuma Kyoto Hotel

Nanostructure and properties of magnetic materials

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National Institute for Materials Science
and
International Center for Materials Nanoarchitronics

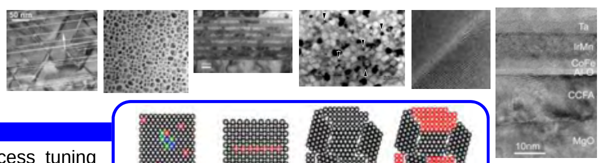
TOYOTA NEDO CREST

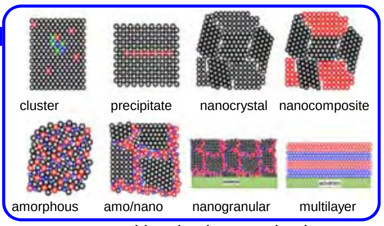
Development of Nanostructured Materials

-understanding propety-structure relationships
-understanding the roles of alloying elements

Process
- •sputtering
- •rapid solidification
- •mechanical milling
- •sintering
- •themomechanical
- •phase transformation

process tuning



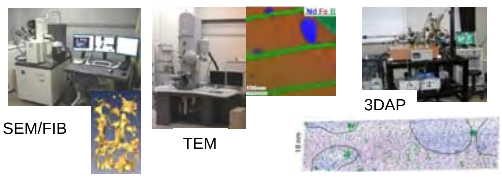


Properties
- ･magnetic
- ･spintronics
- ･mechanical

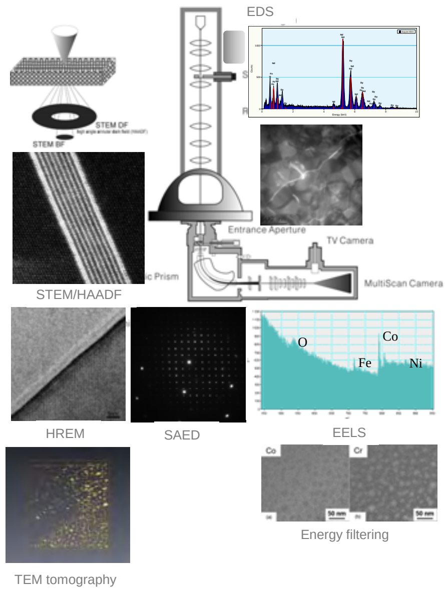
structure & properties

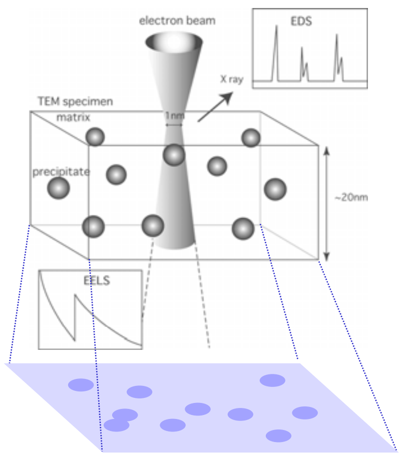
multiscale characterization

Nanostructured Metallic Materials
- •permanent magnets
- •nanocrystalline soft magnetic materials
- •Magnetic recording media
- •spintronics materials
- •spintronics devices -TMR, CPP-GMR
- •nanostructured high strength alloys



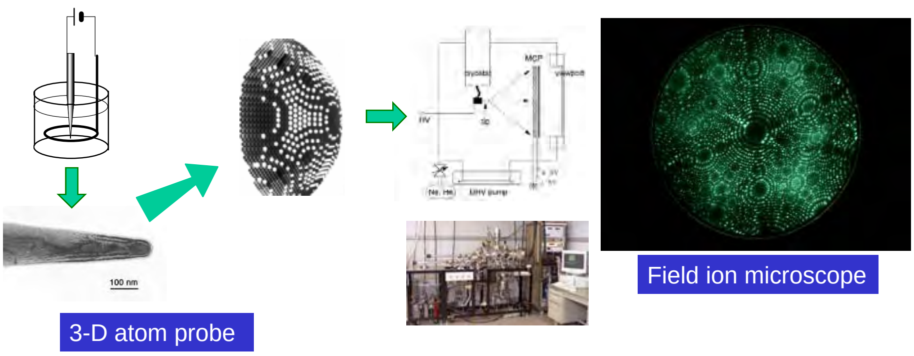
Chemical analysis by TEM

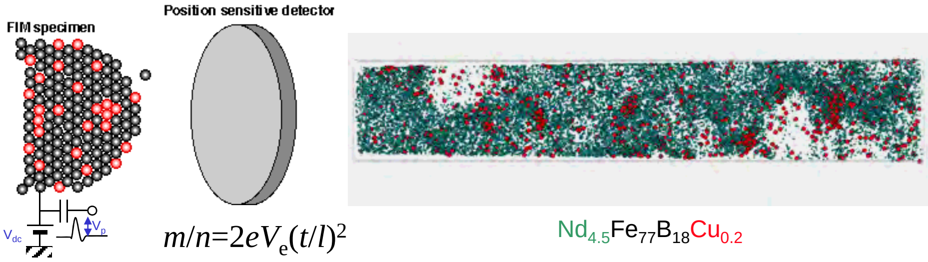




TEM image – 2D projection

3D atom probe

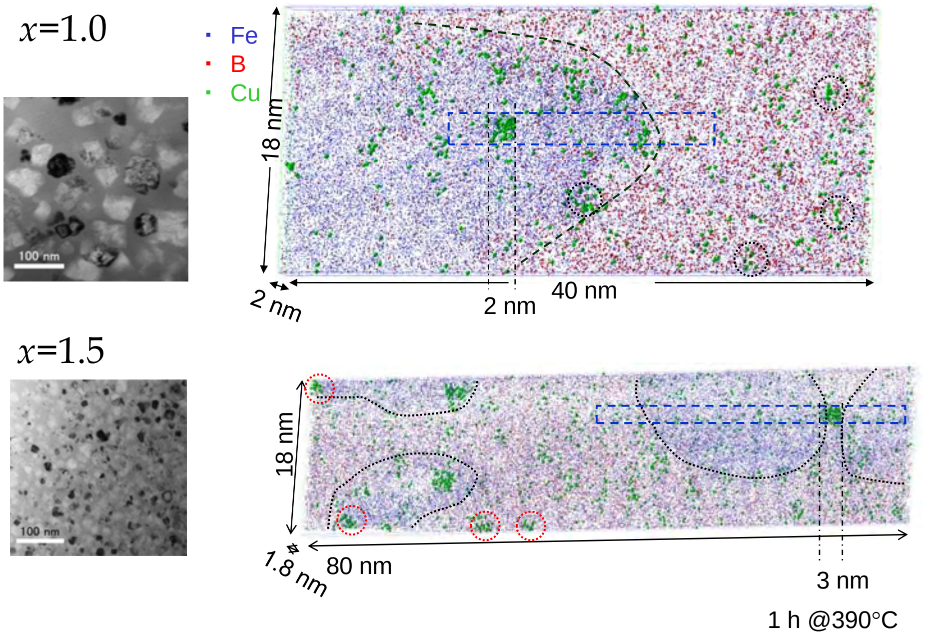




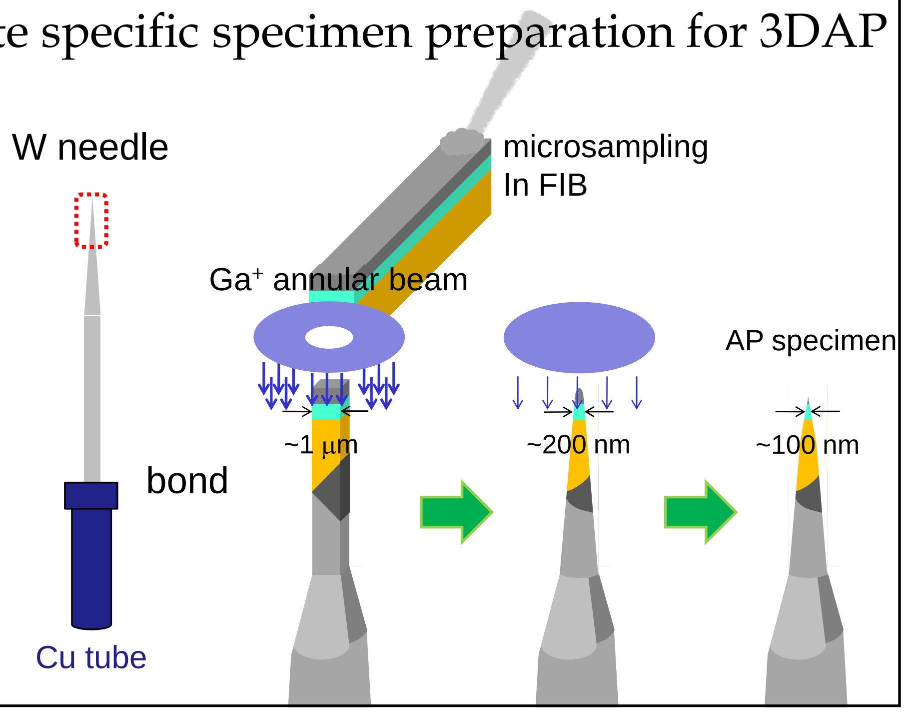
$m/n = 2eV_e(t/l)^2$

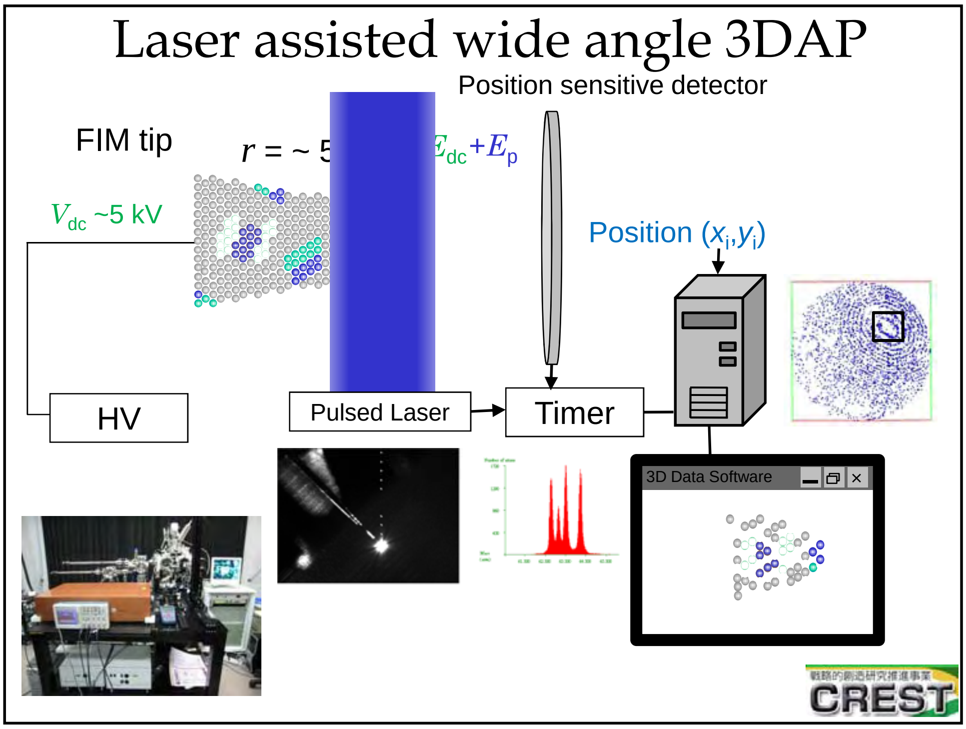
$Nd_{4.5}Fe_{77}B_{18}Cu_{0.2}$

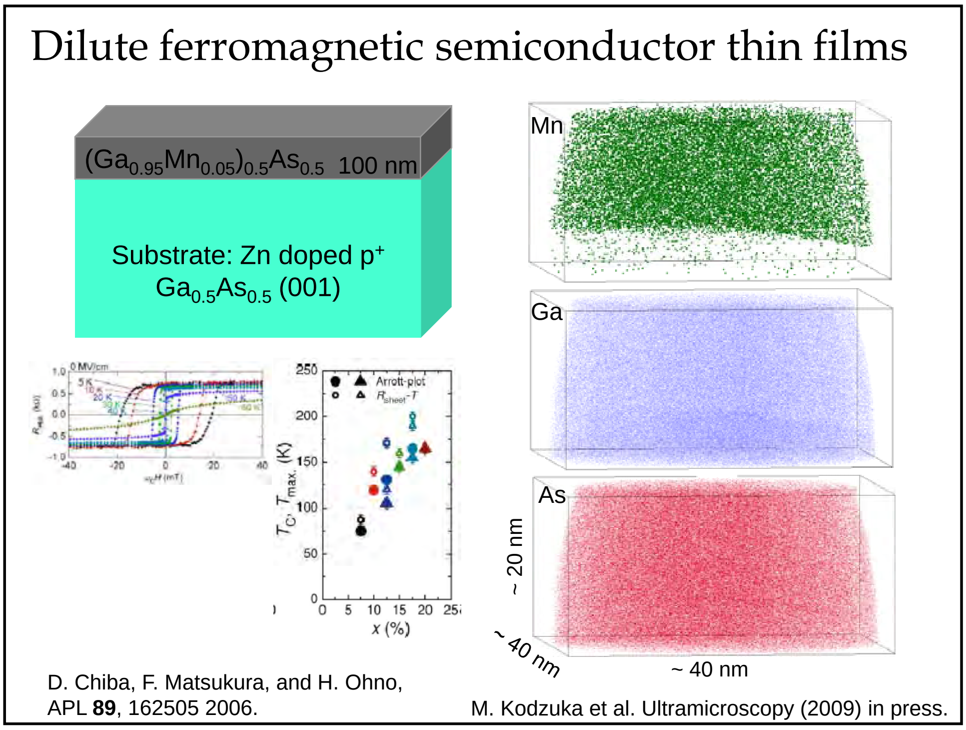
$(Fe_{0.85}B_{0.15})_{100-x}Cu_x$ nanocrystalline softmagnets



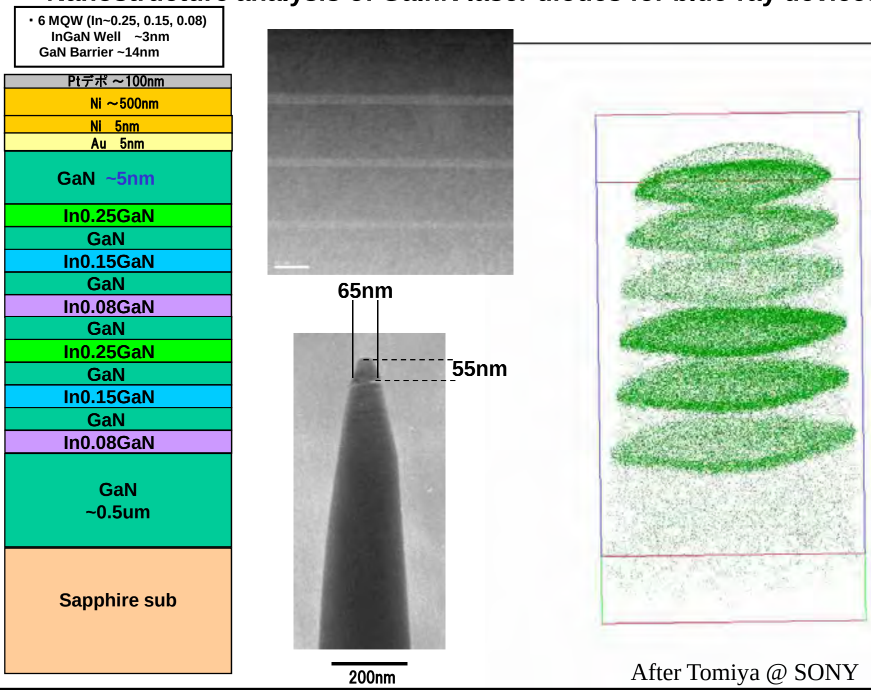
Site specific specimen preparation for 3DAP




Laser assisted wide angle 3DAP
Position sensitive detector
FIM tip
r = ~ 5
Edc+Ep
Vdc ~5 kV
Position (xi,yi)
HV
Pulsed Laser
Timer
3D Data Software
CREST

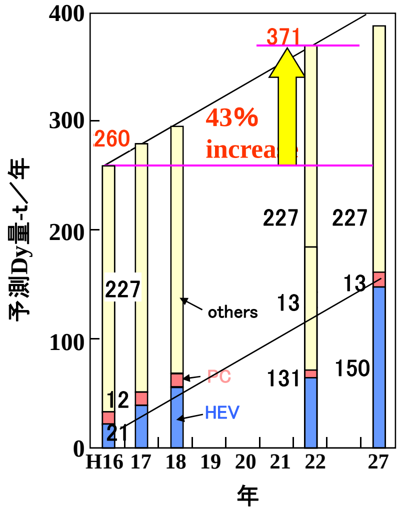

Dilute ferromagnetic semiconductor thin films
(Ga0.95Mn0.05)0.5As0.5 100 nm
Substrate: Zn doped p+ Ga0.5As0.5 (001)
Mn
Ga
As
~ 20 nm
~ 40 nm
~ 40 nm
Arrott-plot
x (%)
D. Chiba, F. Matsukura, and H. Ohno, APL 89, 162505 2006.
M. Kodzuka et al. Ultramicroscopy (2009) in press.

Nanostructure analysis of GaInN laser diodes for blue-ray devices

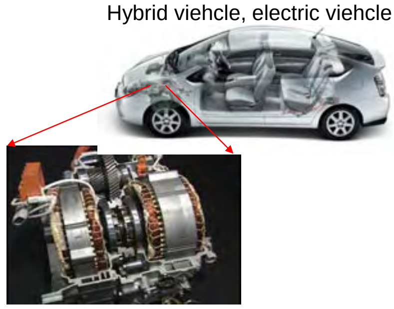


After Tomiya @ SONY

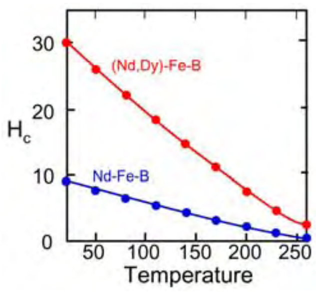
Demand of permanent magnets for HV&EV



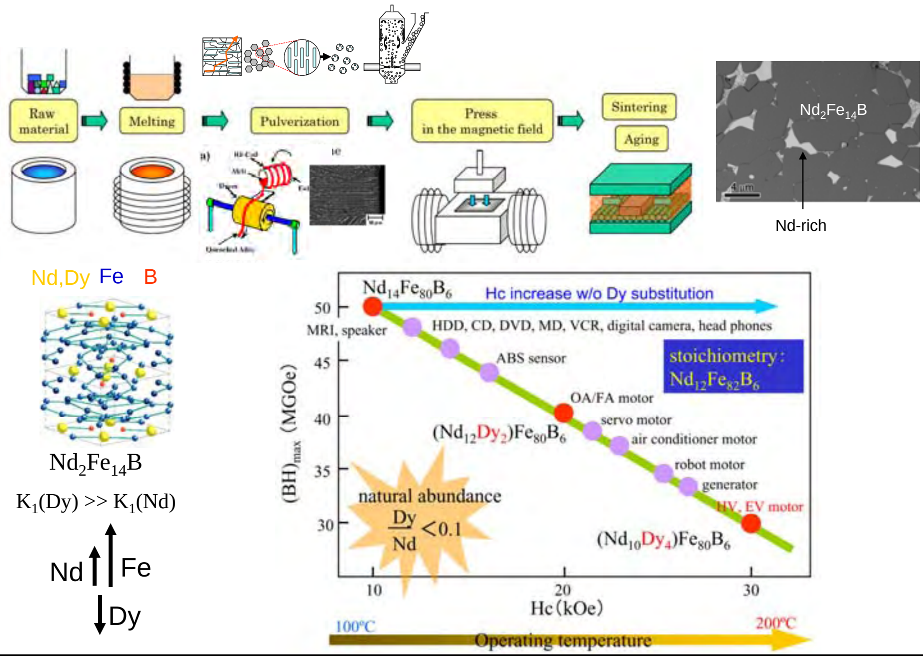
Hybrid viehcle, electric viehcle



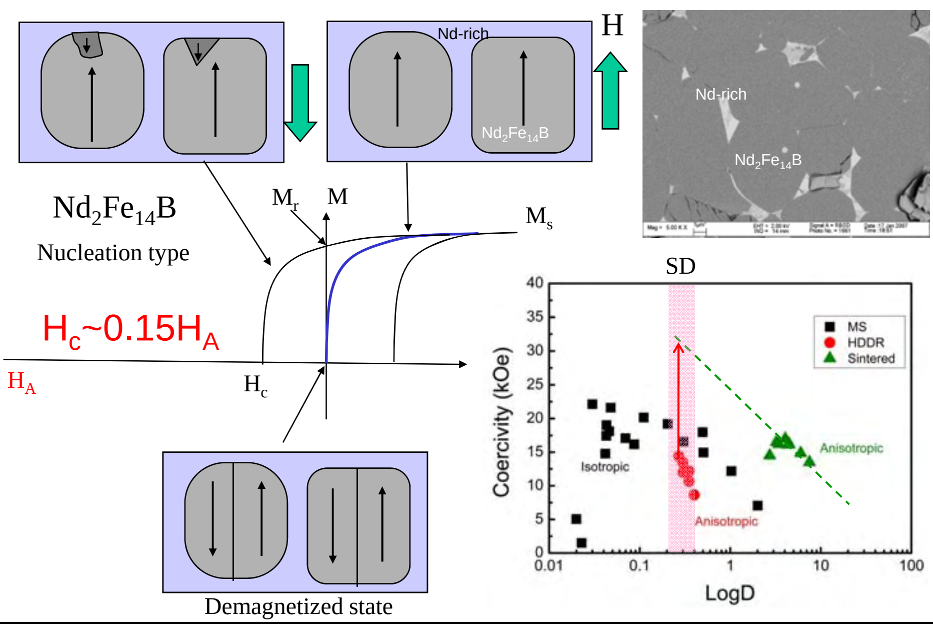
Operation temperature: 200°C



Nd-Fe-B sintered magnets

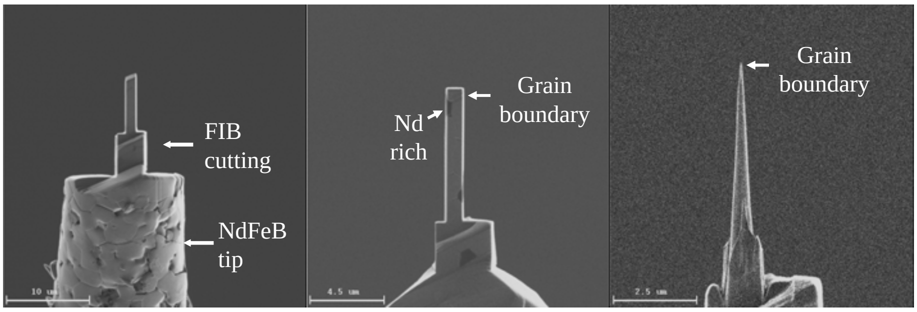


Magnetization reversal by the nucleation mechanism



FIM specimen preparation using FIB

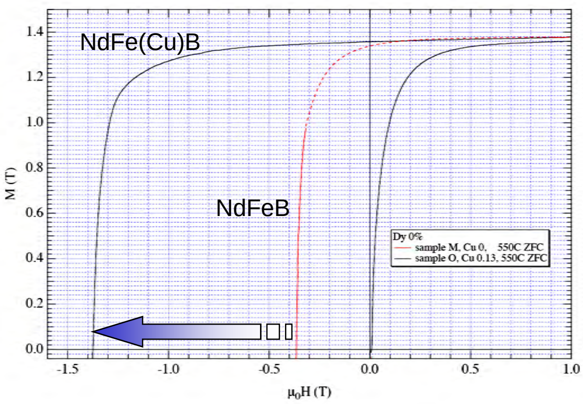
FIB fabrication	Microstructure before annular milling	Annular milling from the top to sharp tip



Question #1
Why Hc increase by trace Cu addition?

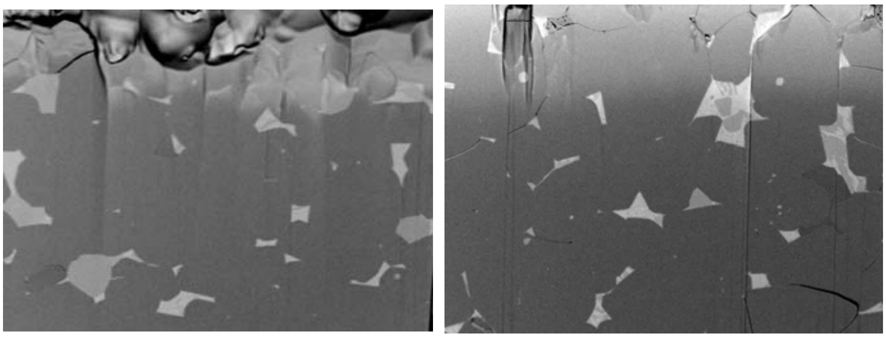
Sample	Nd (at.%)	Dy (at.%)	Cu (at.%)	B (at.%)	Fe (at.%)	H_c (kOe)
NdFeB	14.6	0	0	6.1	79.4	3.6
NdFe(Cu)B	14.6	0	0.13	6.1	79.2	13.6

Annealing condition
under magnetic field
140kOe, 550°C × 3 h
quench without magnetic field

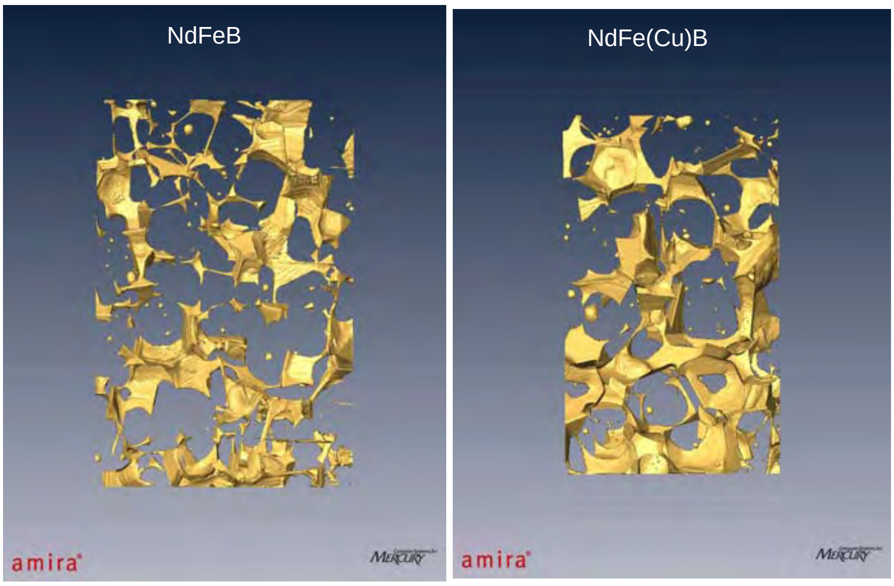


Serial sectioning BSE images

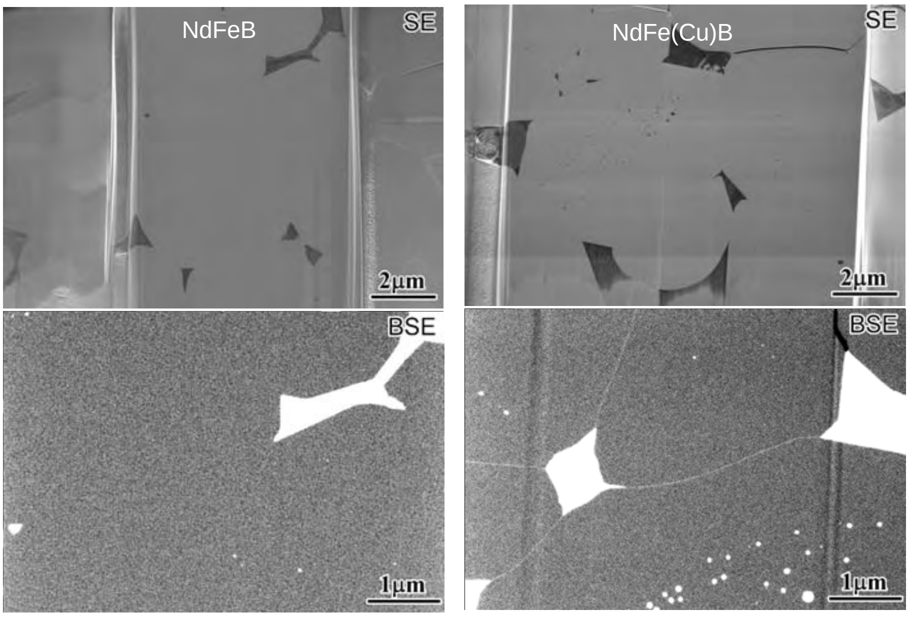
NdFeB 5μm NdFe(Cu)B



3D tomography of Nd-rich phase

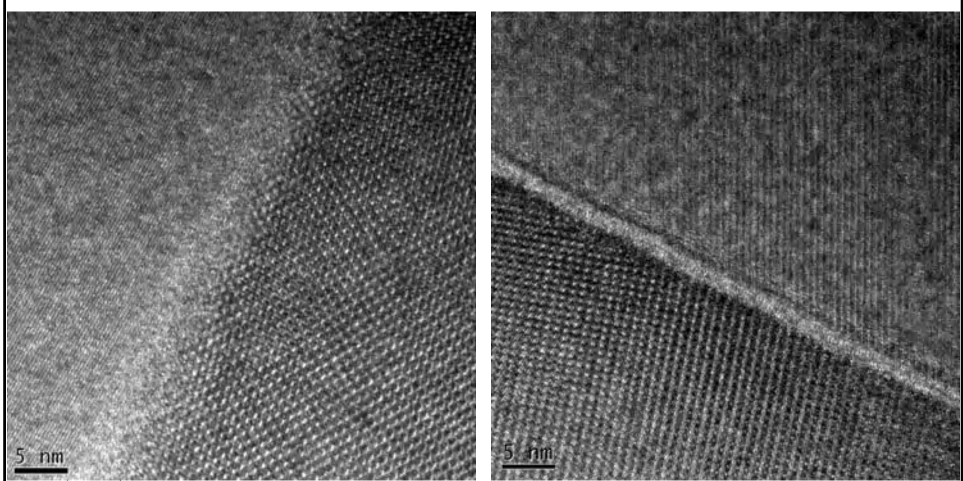


HR BSE images of GBs of Nd-Fe-B magnets



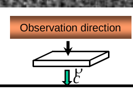
HREM of grain boundaries of Nd-Fe-B magnets

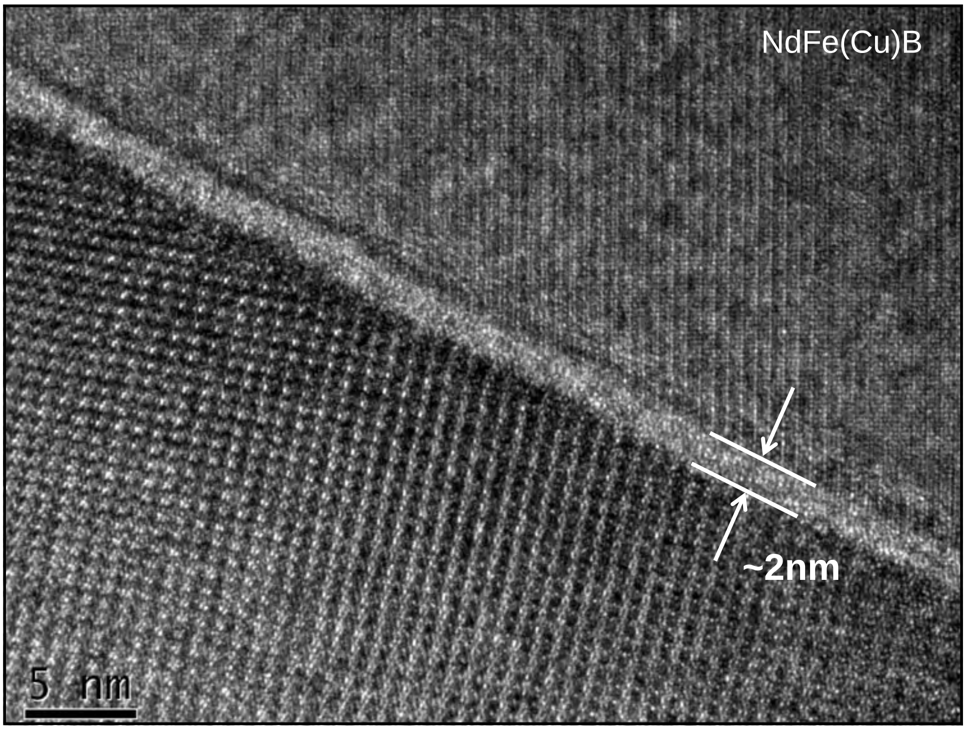
NdFeB NdFe(Cu)B



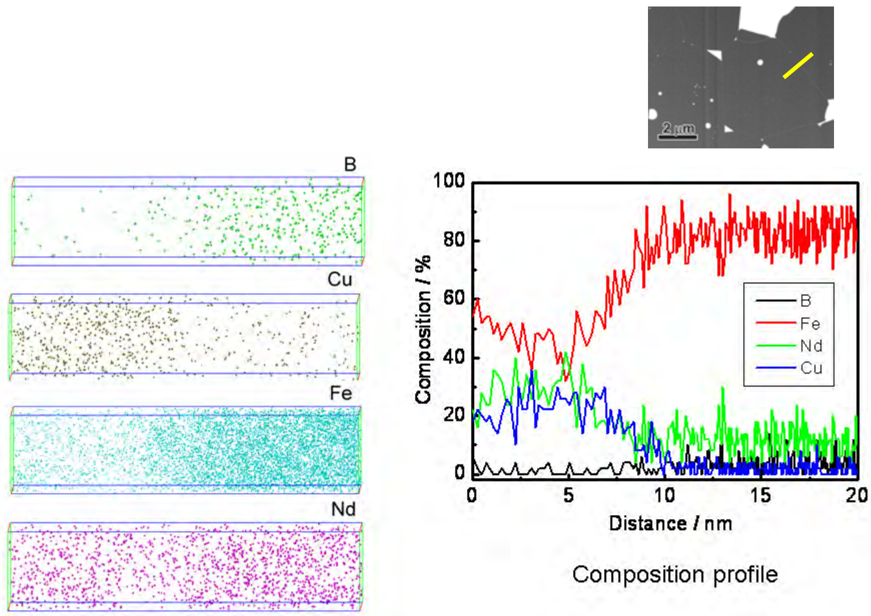
Interface in not clear

Decrease the magnetic anisotropy → H_c is decreased

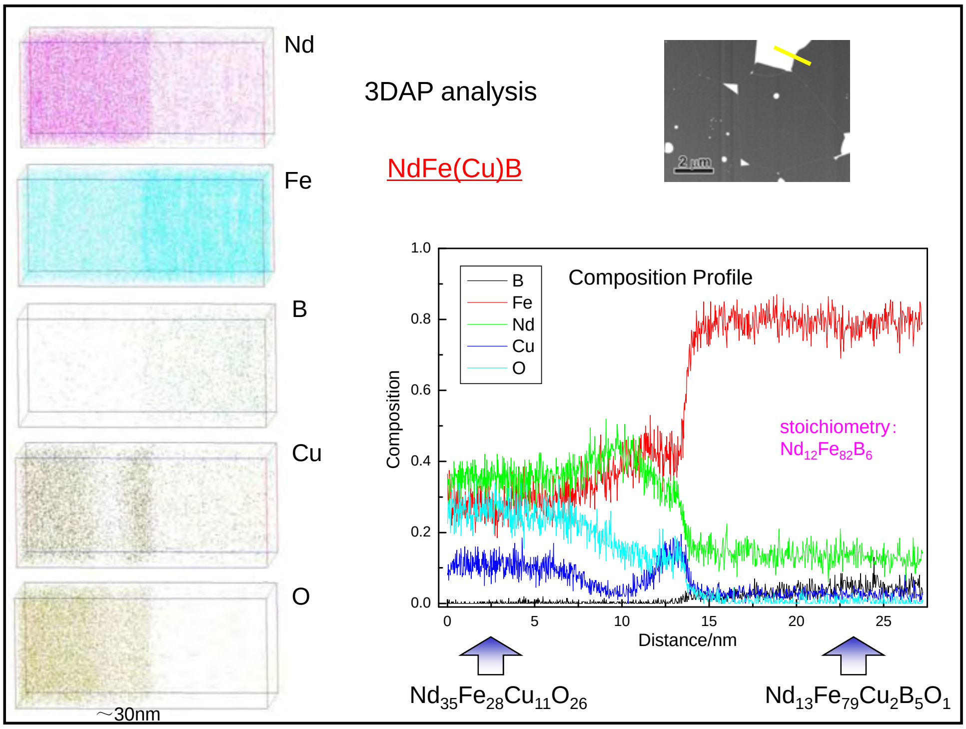




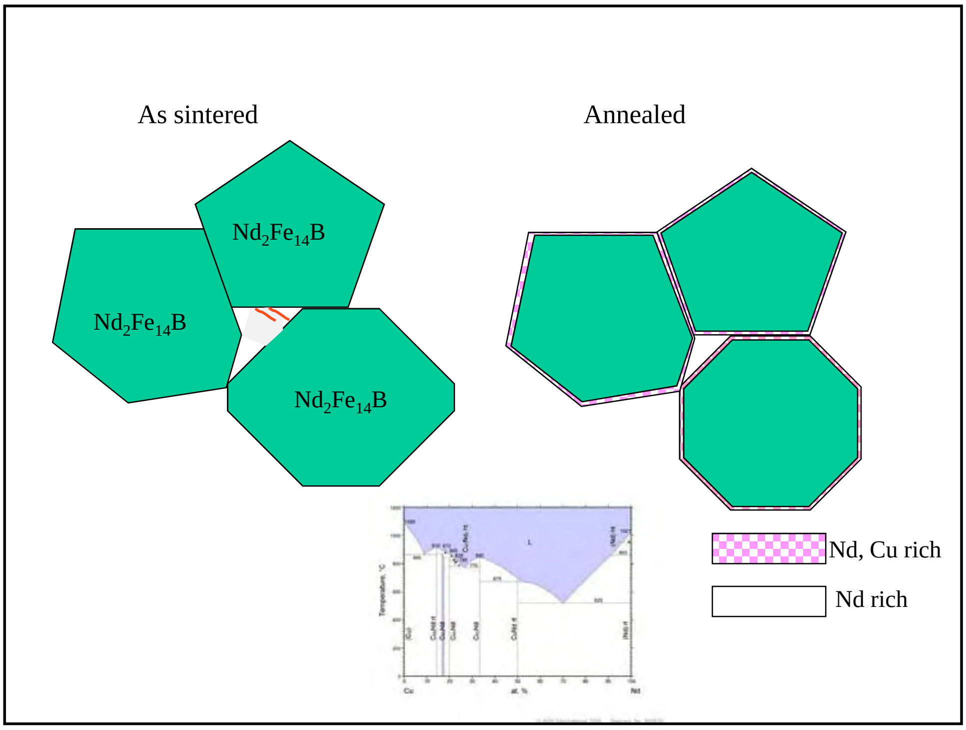
3DAP analysis of grain boundary



Composition profile

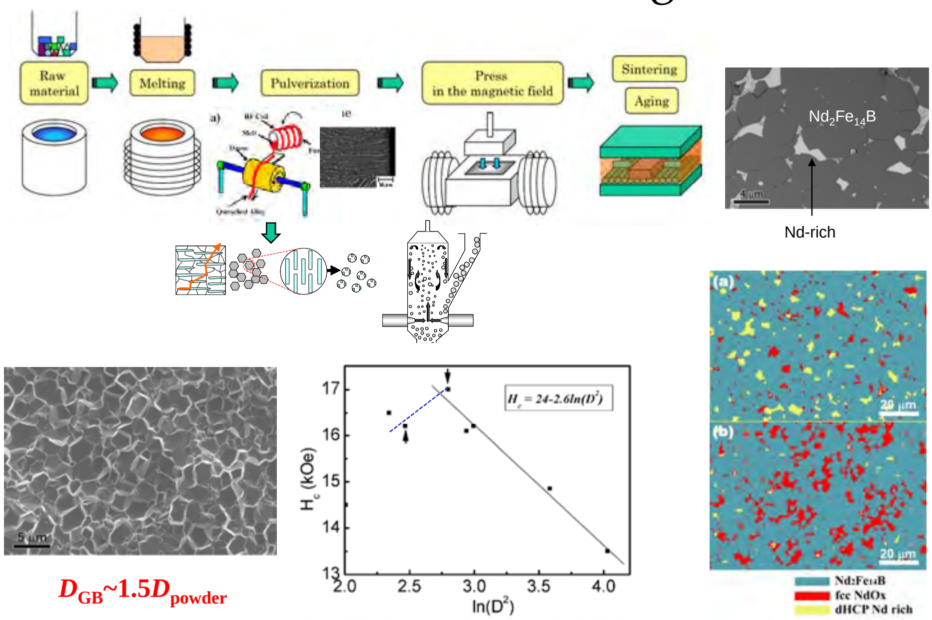


Nd
Fe
B
Cu
O
~30nm
3DAP analysis
NdFe(Cu)B
2 μm
Composition Profile
B
Fe
Nd
Cu
O
Composition
Distance/nm
stoichiometry:
$Nd_{12}Fe_{82}B_6$
$Nd_{35}Fe_{28}Cu_{11}O_{26}$
$Nd_{13}Fe_{79}Cu_2B_5O_1$



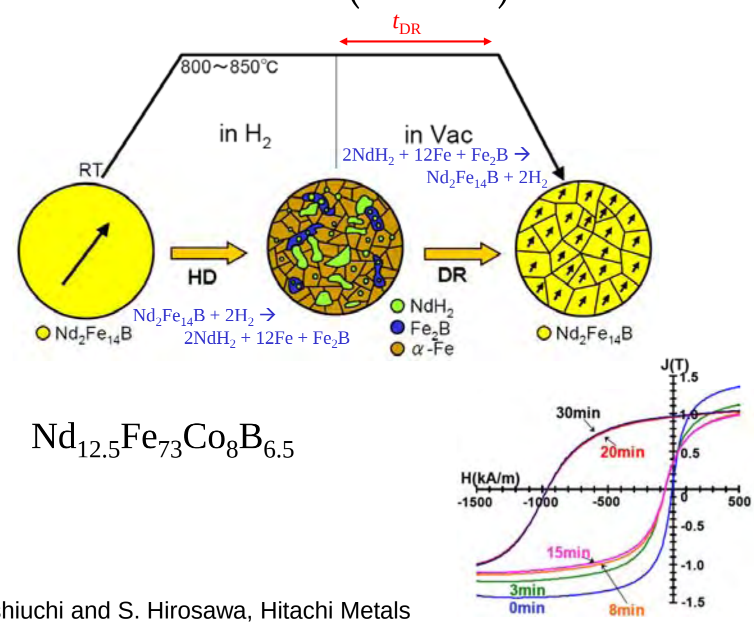
As sintered
Annealed
$Nd_2Fe_{14}B$
$Nd_2Fe_{14}B$
$Nd_2Fe_{14}B$
Nd, Cu rich
Nd rich

Nd-Fe-B sintered magnets

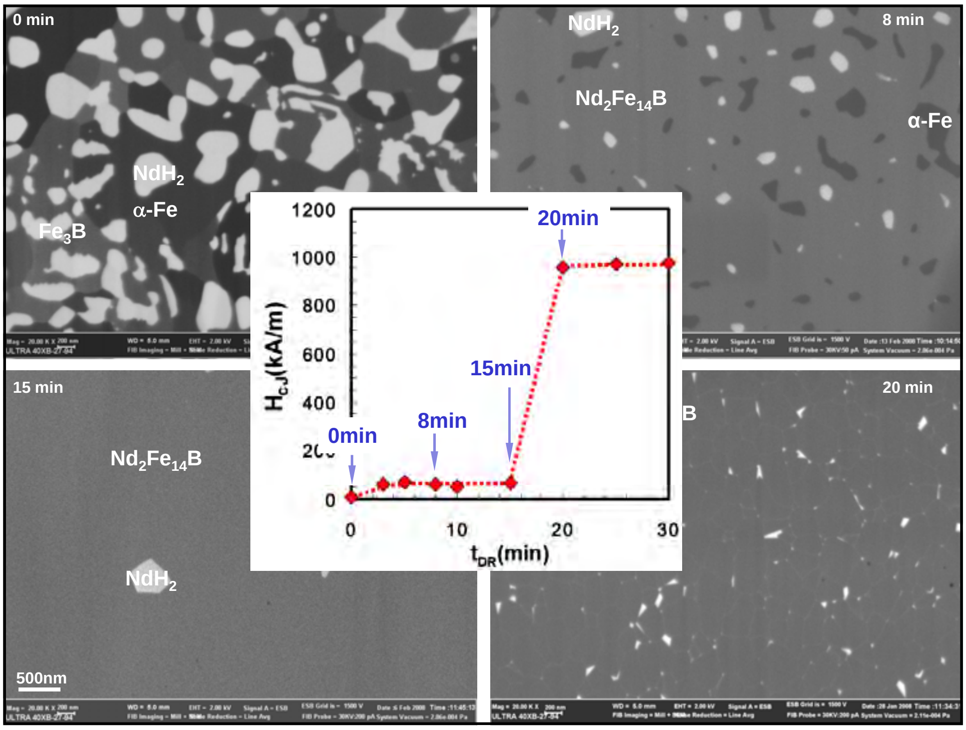


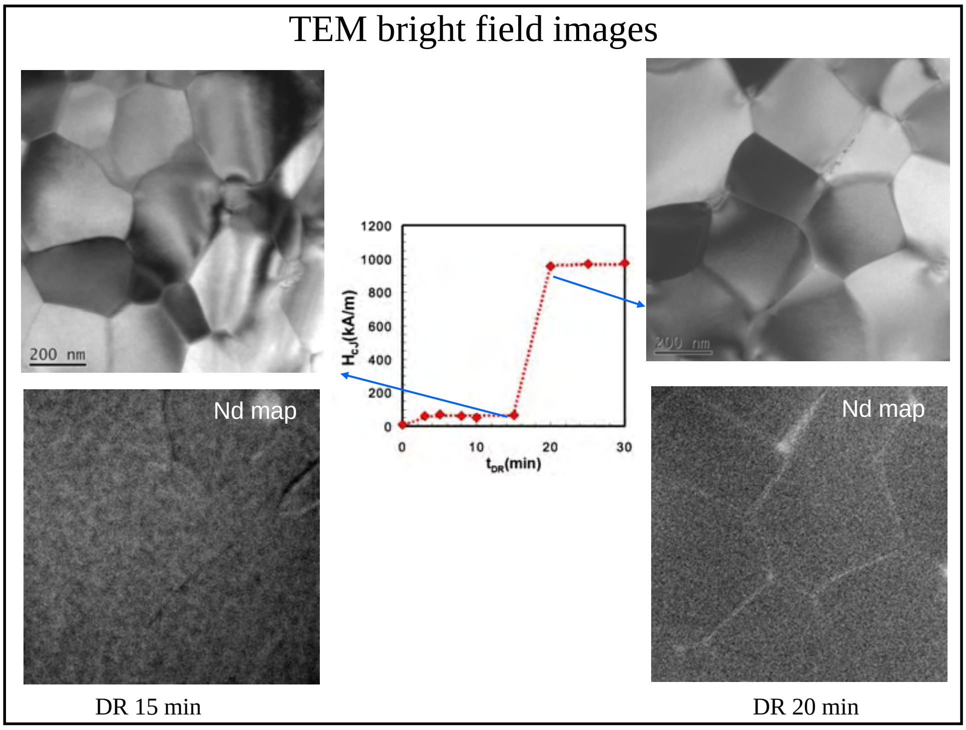
W. F. Li et al. JMMM, (2009) in press.

Hydrogenation Disproportionation Desorption Recombination (HDDR) Process

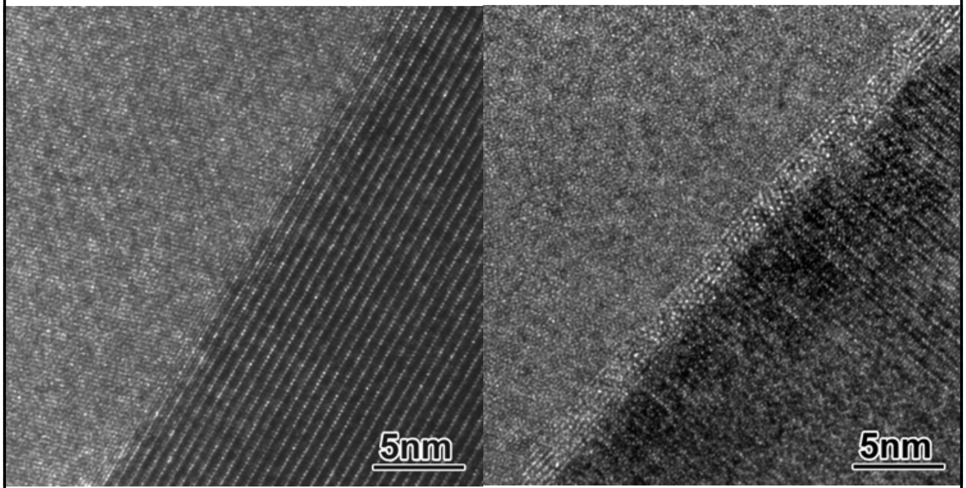


After T. Nishiuchi and S. Hirosawa, Hitachi Metals


0 min
8 min
15 min
20 min
NdH_2
α-Fe
Fe_3B
$Nd_2Fe_{14}B$
500nm
20min
15min
8min
0min
H_{cJ}(kA/m)
t_{DR}(min)


TEM bright field images
200 nm
Nd map
H_{cJ}(kA/m)
t_{DR}(min)
DR 15 min
DR 20 min

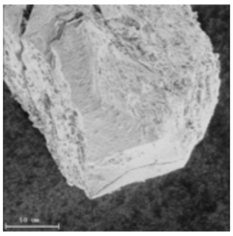
Grain boundaries of HDDR powder



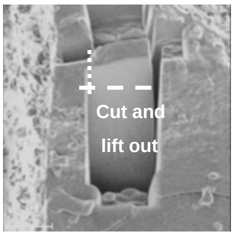
DR 15 min DR 20 min

Specimen preparetion from powder

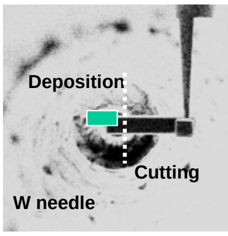
HDDR powder

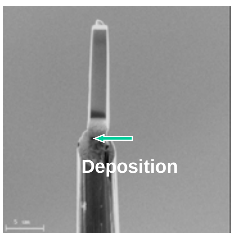


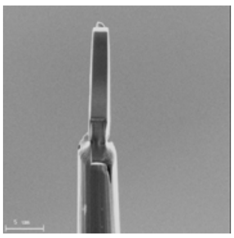
FIB fabrication

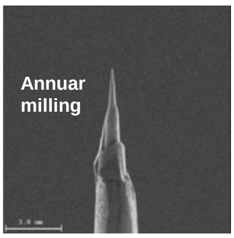


Put sample on the W needle

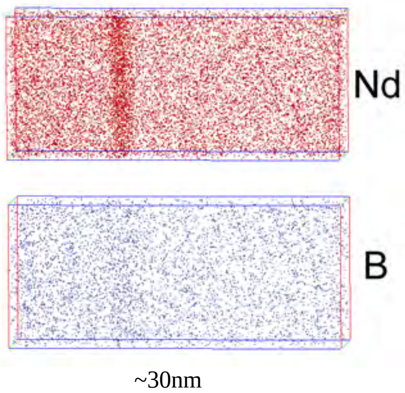


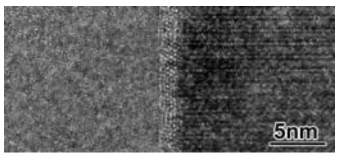


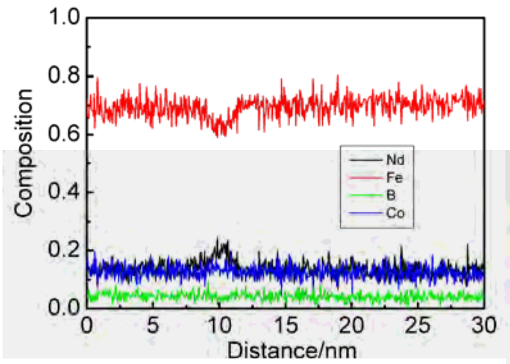


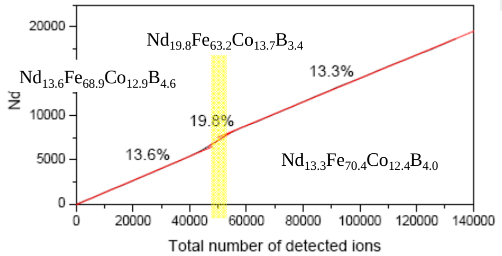


3DAP analysis of HDDR powder



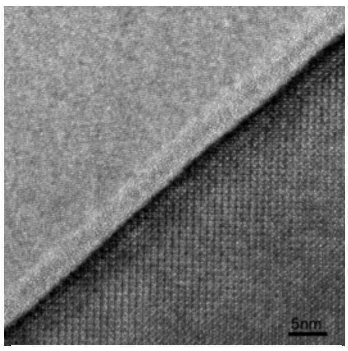




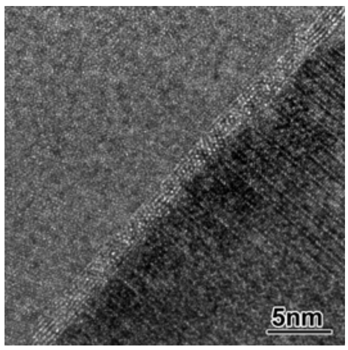


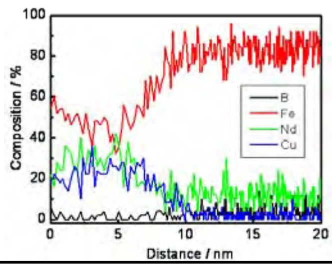
Sintered and HDDR magnets

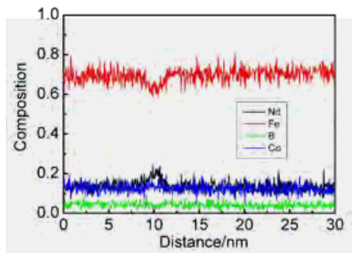
Sintered



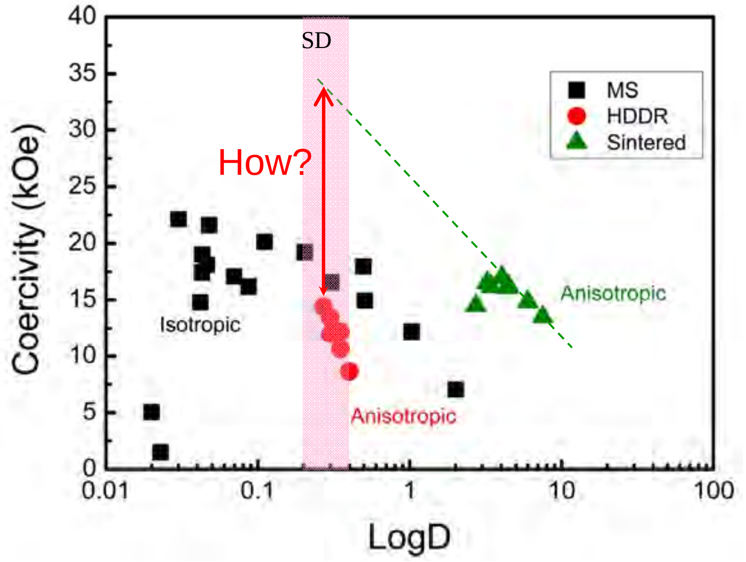
HDDR







H_c increase by optimizing interfacial nanostructure



Summary

- There is a large potential in developing higher performance magnetic materials by controling nanostructures
- Nanostructure characterization by 3DAP/TEM is particularly useful to obtain critical information on designing nanostructured magnetic materilals
- Laser assisted wide angle 3D atom probe expands the application area of 3DAP including semiconductors and their thin film devices

Acknowledgment

NIMS: W.F. Li

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CREST-JST for laser assisted 3D atom probe

TOYOTA and NEDO for permanent magnet research