Nanostructure and properties of magnetic materials

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Development of Nanostructured Materials
-understanding property-structure relationships
-understanding the roles of alloying elements

Process
• sputtering
• rapid solidification
• mechanical milling
• sintering
• thermomechanical
• phase transformation

Properties
• magnetic
• spintronics
• mechanical

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

Process tuning
structure & properties
multiscale characterization
Chemical analysis by TEM

TEM image – 2D projection

3D atom probe

\[ m/n = 2eV(t/l)^2 \]

\[ \text{Nd}_{4.5}\text{Fe}_{77}\text{B}_{18}\text{Cu}_{0.2} \]
(Fe$_{0.85}$B$_{0.15}$)$_{100-x}$Cu$_x$ nanocrystalline softmagnets

$x=1.0$

$x=1.5$

Site specific specimen preparation for 3DAP

W needle

Ga$^+$ annular beam

AP specimen
Laser assisted wide angle 3DAP

FIM tip

$V_{dc} \sim 5 \text{kV}$

HV

Position sensitive detector

$r = E_{dc} + E_p$

Position $(x_i,y_i)$

Laser assisted wide angle 3DAP

Dilute ferromagnetic semiconductor thin films

$(Ga_{0.95}Mn_{0.05})_{0.5}As_{0.5}$

Substrate: Zn doped p$^+$

Ga$_{0.5}$As$_{0.5}$ (001)


Nanostructure analysis of GaInN laser diodes for blue-ray devices

- 6 MQW (In~0.25, 0.15, 0.08)
- InGaN Well ~3nm
- GaN Barrier ~14nm
- Ni ~500nm
- GaN ~5nm
- In0.25GaGan
- GaN
- In0.15GaGan
- GaN
- In0.08GaGan
- GaN
- In0.25GaGan
- GaN
- In0.15GaGan
- GaN
- In0.08GaGan
- GaN ~0.5um
- Sapphire sub

After Tomiya @ SONY

Demand of permanent magnets for HV&EV

Hybrid vehicle, electric vehicle

Operation temperature: 200°C

Year

Demand of rare earths - Dy

Year

5% increase

Others
Nd-Fe-B sintered magnets

K_{1(Dy)} >> K_{1(Nd)}

Magnetization reversal by the nucleation mechanism

H_c ~ 0.15H_A
FIM specimen preparation using FIB

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

FIB cutting NdFeB tip

FIB cutting

NdFeB tip

Grain boundary

Grain boundary

Question #1
Why Hc increase by trace Cu addition?

<table>
<thead>
<tr>
<th>Sample</th>
<th>Nd (at.%)</th>
<th>Dy (at.%)</th>
<th>Cu (at.%)</th>
<th>B (at.%)</th>
<th>Fe (at.%)</th>
<th>Hc (kOe)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NdFeB</td>
<td>14.6</td>
<td>0</td>
<td>0</td>
<td>6.1</td>
<td>79.4</td>
<td>3.6</td>
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<tr>
<td>NdFe(Cu)B</td>
<td>14.6</td>
<td>0</td>
<td>0.13</td>
<td>6.1</td>
<td>79.2</td>
<td>13.6</td>
</tr>
</tbody>
</table>

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

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

Interface in not clear
Decrease the magnetic anisotropy $\rightarrow$ $H_c$ is decreased
3DAP analysis of grain boundary

Composition profile
3DAP analysis

\( \text{NdFe(Cu)B} \)

Composition Profile

stoichiometry: \( \text{Nd}_{12}\text{Fe}_{82}\text{B}_6 \)

As sintered

Nd, Cu rich

Nd rich

Annealed
Nd-Fe-B sintered magnets

$D_{\text{GB}} = 1.5D_{\text{powder}}$


Hydrogenation Disproportionation Desorption Recombination (HDDR) Process

$\text{Nd}_{12.5}\text{Fe}_{73}\text{Co}_{8}\text{B}_{6.5}$

After T. Nishiuchi and S. Hirosawa, Hitachi Metals
TEM bright field images
Grain boundaries of HDDR powder

Specimen preparation from powder

1. HDDR powder
2. FIB fabrication
3. Put sample on the W needle
4. Cutting
5. Annular milling
3DAP analysis of HDDR powder

Sintered and HDDR magnets
Summary

• There is a large potential in developing higher performance magnetic materials by controlling nanostructures

• Nanostructure characterization by 3DAP/TEM is particularly useful to obtain critical information on designing nanostructured magnetic materials

• Laser assisted wide angle 3D atom probe expands the application area of 3DAP including semiconductors and their thin film devices
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