Texture development in Nd-Fe-B and Nd-Fe-V alloys by hot forging in view of improving permanent Magnet properties

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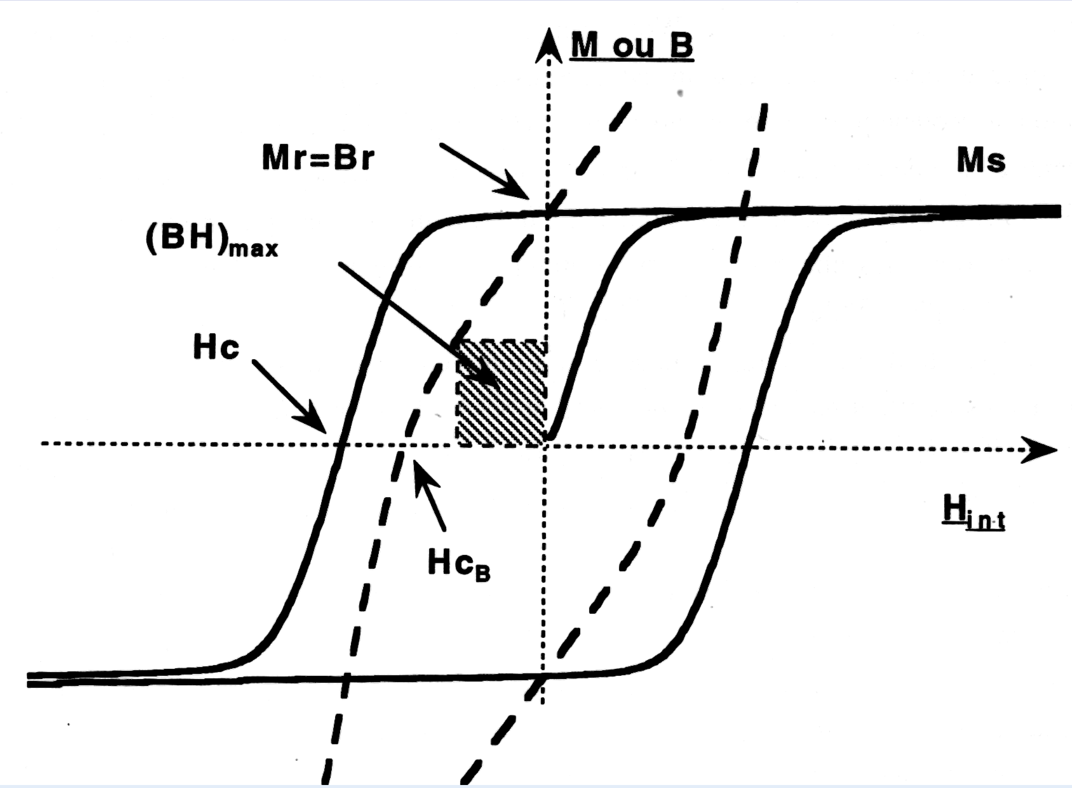
- Some intrinsic magnetic and extrinsic magnet properties
- QTA and anisotropic magnetisation curves: $ErMn_4Fe_8C$ case
- Nd-Fe-B-Cu, Nd-Fe-V alloys
- conclusions

Permanent magnet characteristics



Intrinsic properties

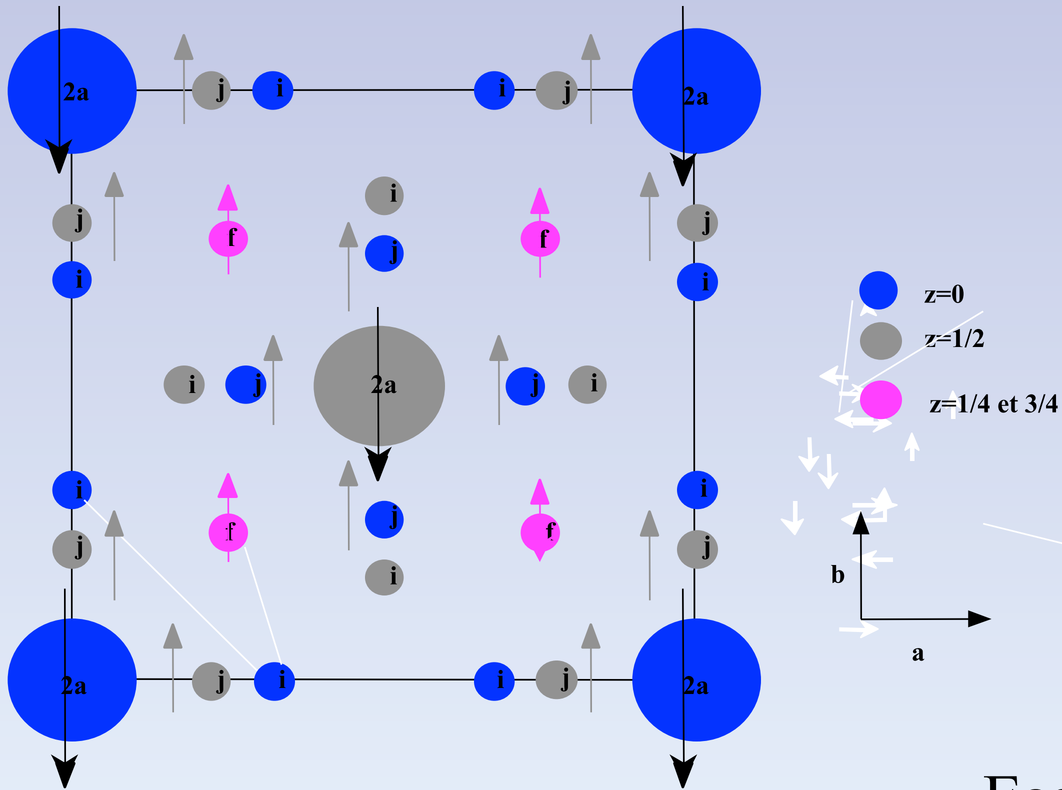
Hard magnetic phase

- Saturation magnetisation, M_s
- Magnetocrystalline anisotropy
- Curie temperature
- Anisotropy field

Extrinsic properties

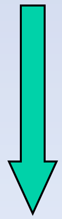
- Remanence, M_r
- Coercive field, H_c
- Maximum energy product, BH_{max}

$ErMn_4Fe_8C$



Structural determination:

M. Morales et al.: J. Magn. And Magn. Mat. ***196*** *(1999) 703*

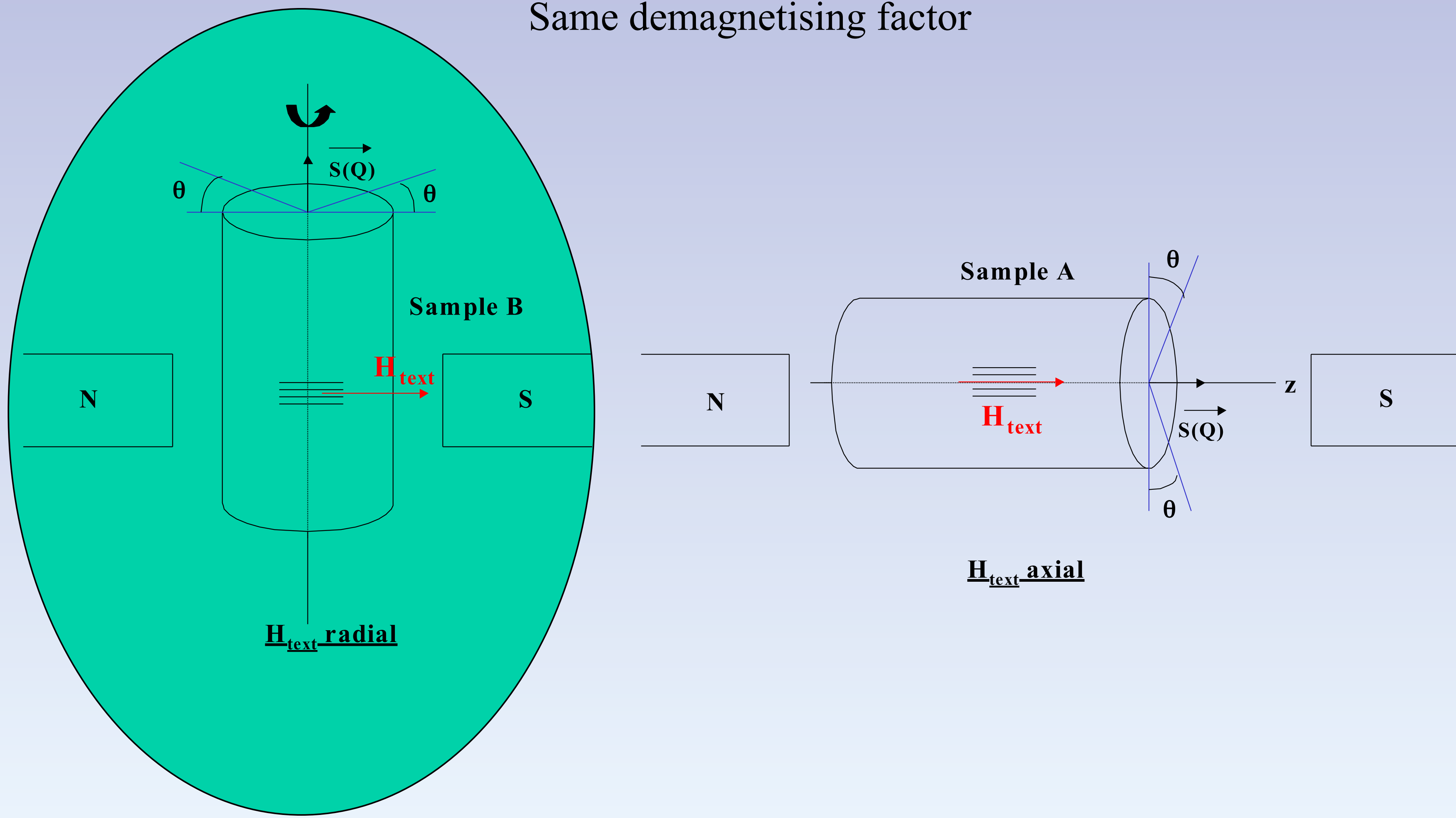


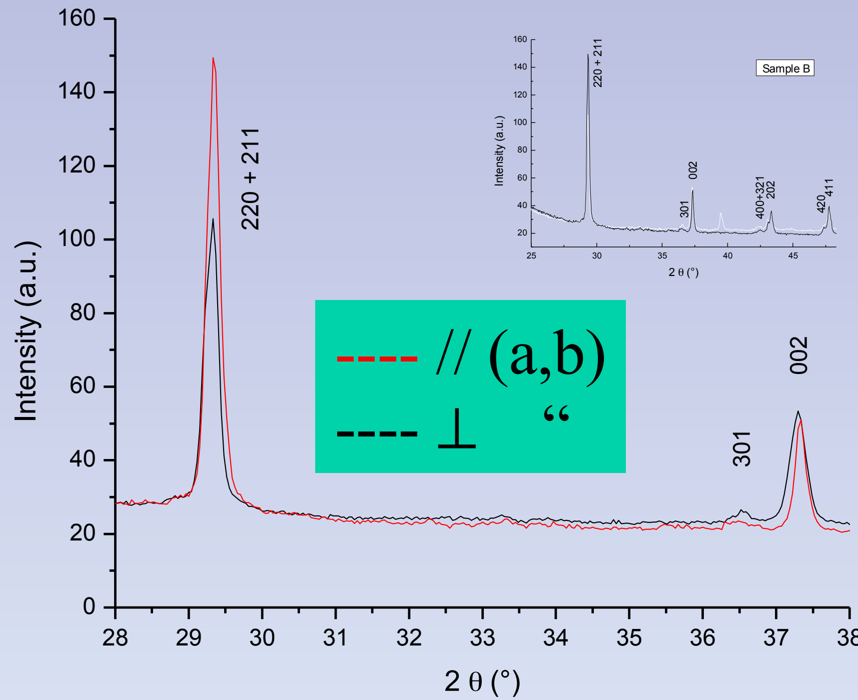
Easy-plane tetragonal phase

magnetic moments in the (a,b) planes

⊥ (a,b) ⟵ H_{meas} // z ⟶ // (a,b)

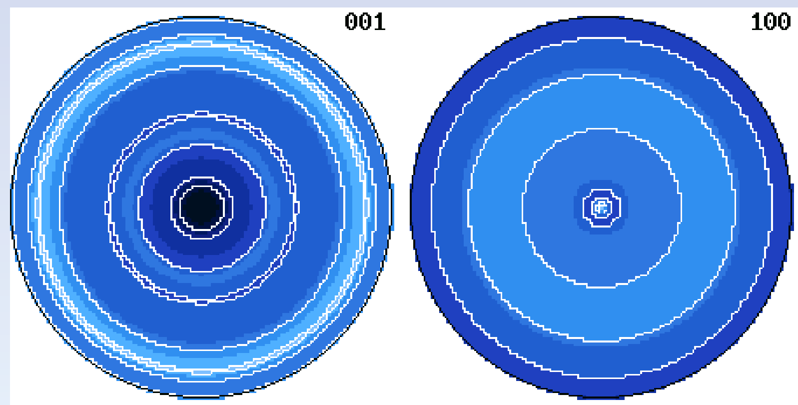
Same demagnetising factor





Quantitative Texture Analysis

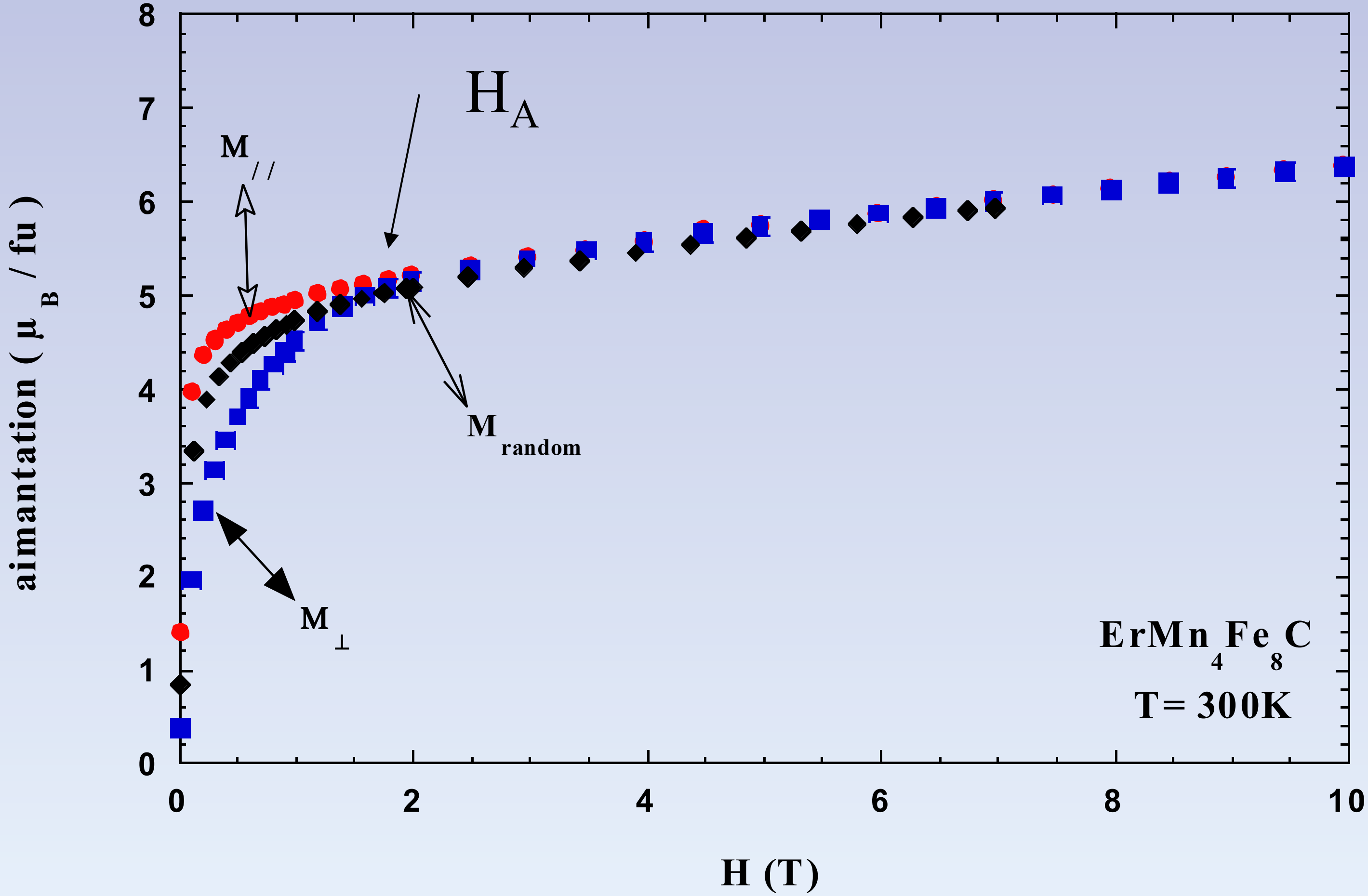
$RP_0 = 1.2$ %
$F_2 = 1.3$ mrd^2
$S = -0.13$



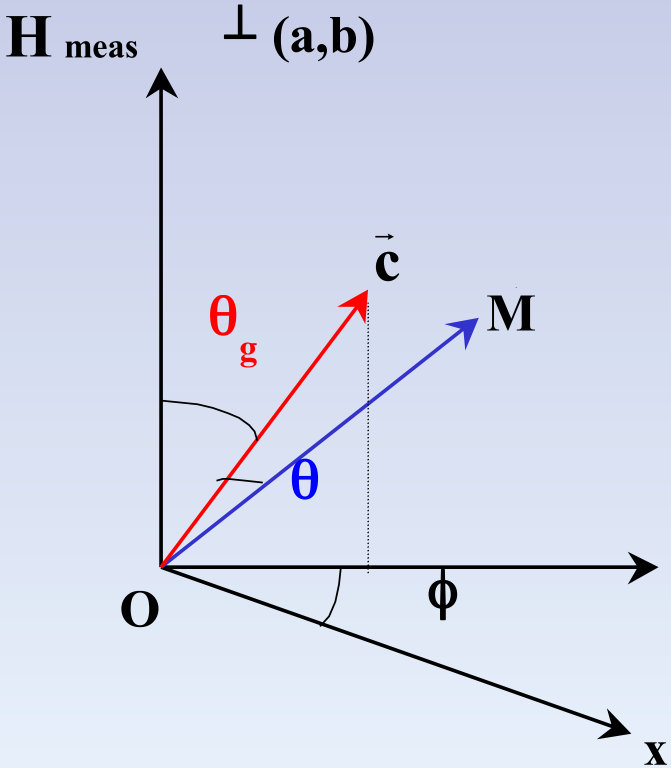
max {001}: 3.9 mrd
min: 0.5 mrd

{001} radial distribution: ρ_0 (0.5 mrd) + PV (HWHM = 12°)

Anisotropic magnetisation curves



Model for $M_\perp$:



$$M(H_{meas}) = M_S \cos(\theta_g - \theta)$$

$$E(H_{meas}) = K_1 \sin^2\theta - H\, M_S \cos(\theta_g - \theta)$$

anisotropy energy — Zeeman energy

$$\frac{dE}{d\theta} = 0 \Rightarrow H_{meas} = \frac{2K_1 \sin\theta \cos\theta}{M_S \sin(\theta_g - \theta)}$$

$H_A = 2K_1/M_S$ $\quad$ $M_S = 5.24\ \mu_B/fu$

$$\frac{H_{meas}}{H_A} = \frac{\sin\theta \cos\theta}{\sin(\theta_g - \theta)}$$

Normalised Probability function F, to find c-axes in d**y**:

$$\int_{\theta_g=0}^{\frac{\pi}{2}} \int_{\varphi=0}^{2\pi} F(\theta_g, \varphi) \sin\theta_g d\theta_g d\varphi = 1$$

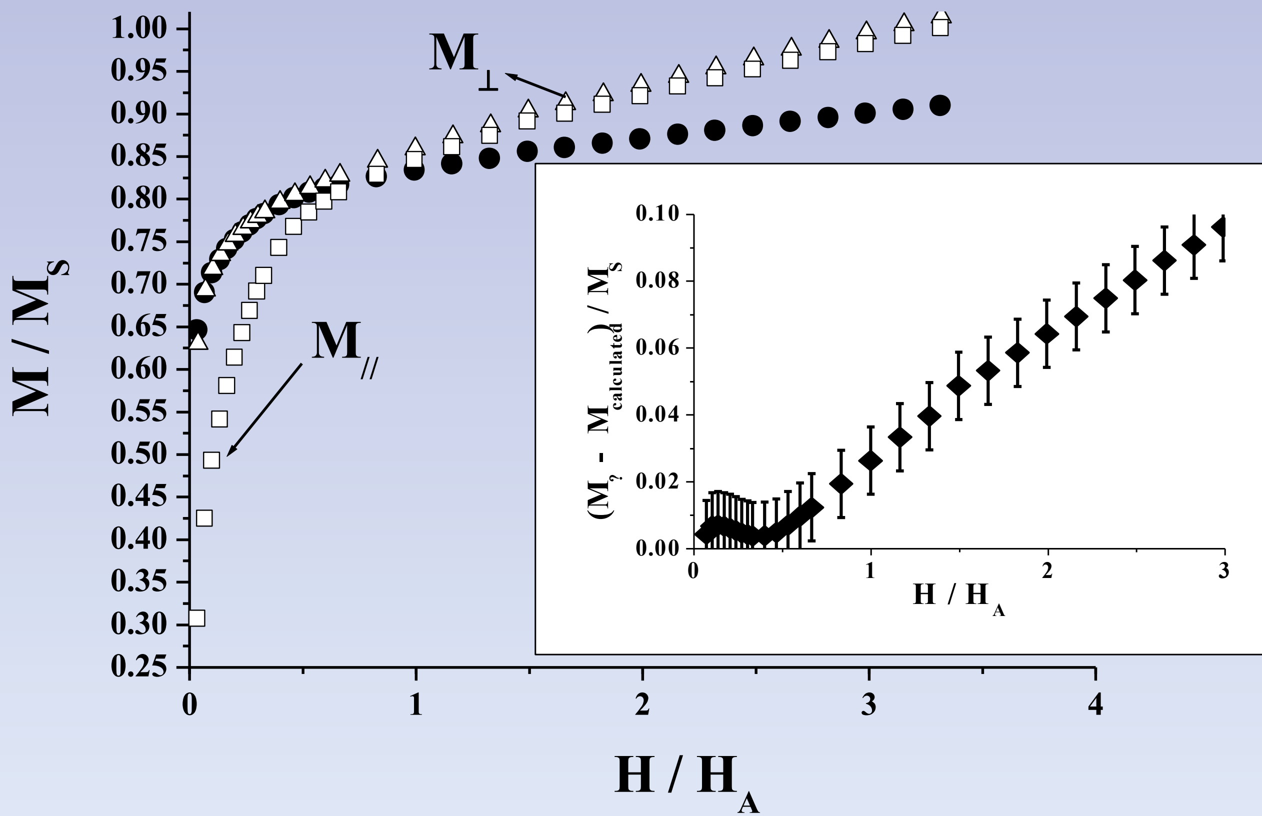
Fibre texture:

$$2\pi \int_{\theta_g=0}^{\frac{\pi}{2}} G(\theta_g) \sin\theta_g d\theta_g = 1$$

$$G(\theta_{\mathbf{g}}) = \left(1 - \rho_0\right) PV(\theta_{\mathbf{g}})$$

Finally:

$$\frac{M_{\perp}}{M_S} = 2\pi \int_0^{\frac{\pi}{2}} \left(1 - \rho_0\right) PV(\theta_g) \sin\theta_g \cos(\theta_g - \theta)\, d\theta_g + \rho_0 M_{random}$$

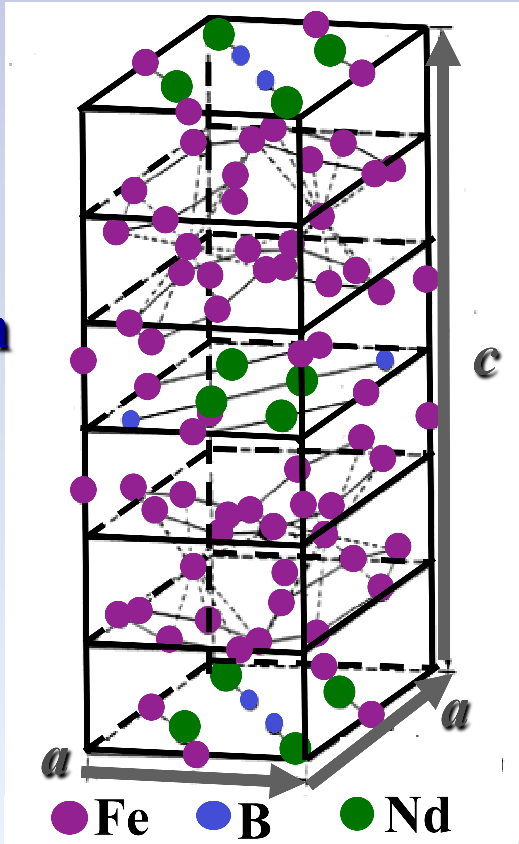


Morales, Chateigner, Fruchart: J. Magn. Mag. Mat. 2003

$Nd_2Fe_{14}B$

Crystallographic structure

$P4_2/mnm$



Intrinsic magnetic properties:

hard magnetic phase

-Uniaxial anisotropy → c-axis = easy magnetisation axis

- Ha= 7T

- M_s= 1.61 T

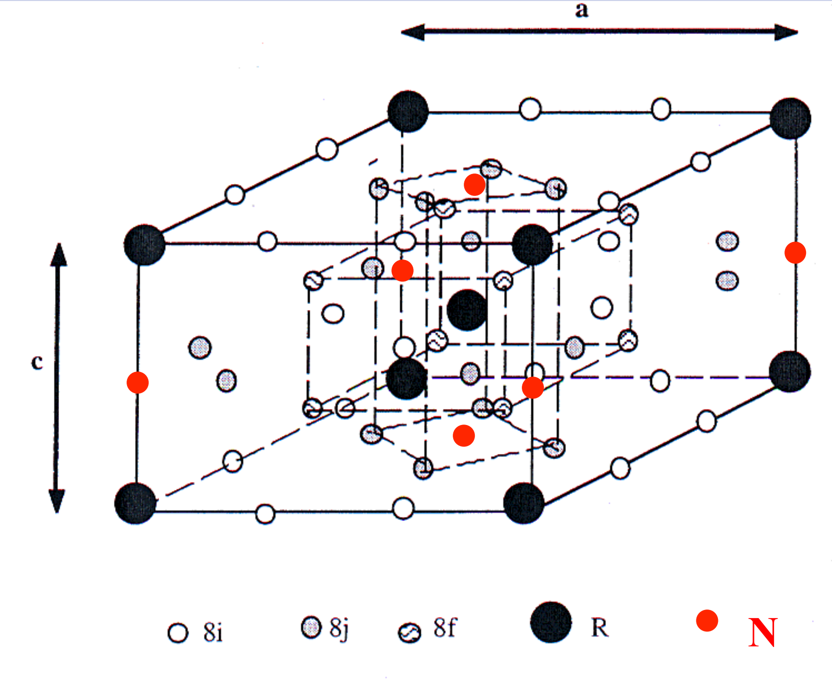
- T_c= 315°C

$NdFe_{12-x}V_xN$ compounds

Crystallographic structure

- space group I4/mmm

Nitrogenation → **N atoms in 2b sites**



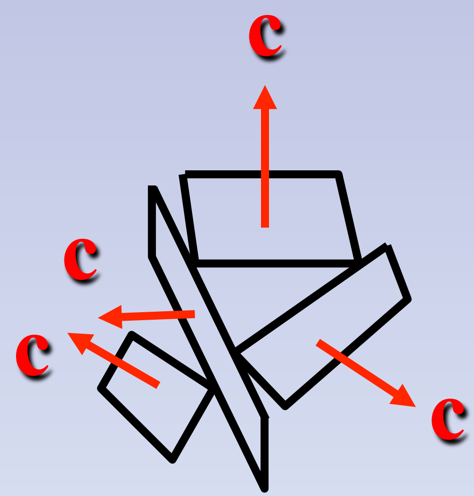
Intrinsic magnetic properties:
hard magnetic phase

- **c-axis = easy magnetisation axis**
- **Ha= 1.5T → 11 T**
- **M_s= 1.13 T → 1.37 T**
- **T_c= 340°C → 510°C**

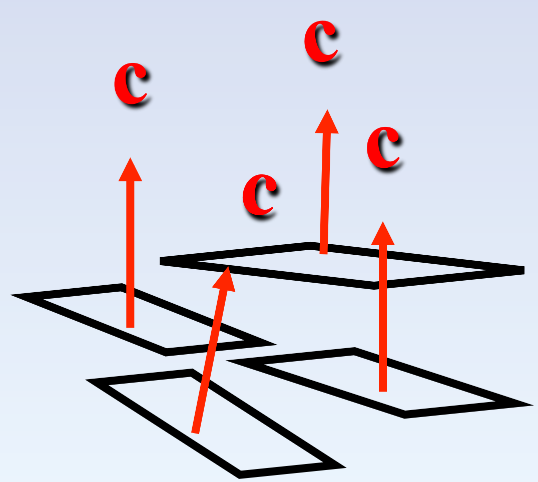
Permanent magnet extrinsic properties: anisotropy

ANISOTROPY

$$\frac{M_r^{//}}{M_r^{\perp}}$$



common orientation of the easy magnetisation axes of the crystallites



Orientation

of magnetic particles in a non magnetic matrix

Permanent magnet extrinsic properties: coercivity

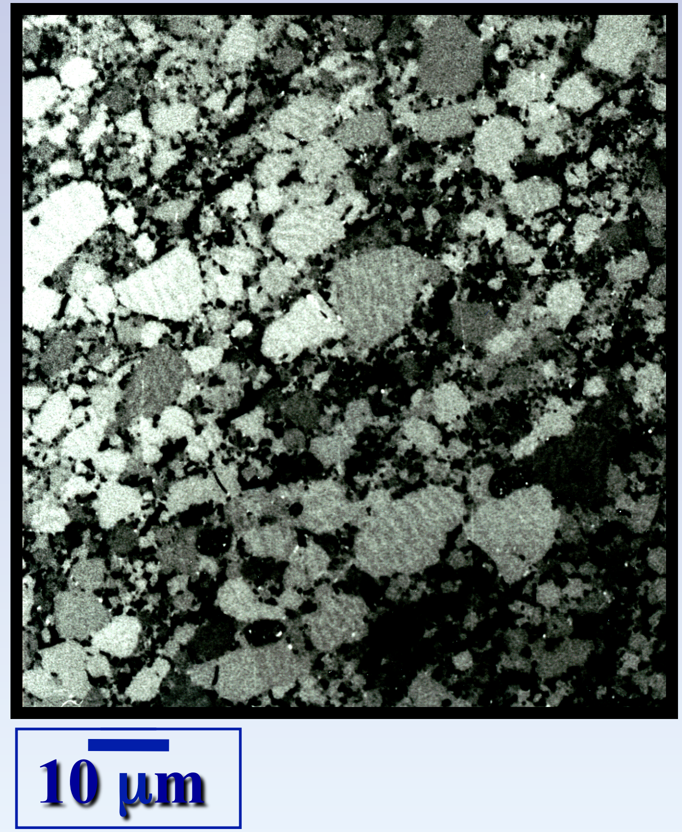
COERCIVITY

H_C

Grain size control of the hard magnetic phase

Intergranular phase distribution → Magnetic decoupling

Secondary phases



Preparation route

Induction Melting and Casting

+

Hot forging

+

Powdering (HD Process)

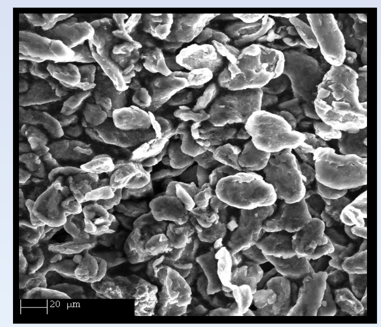
+

Nitrogenation (Nd-Fe-V)

Magnet Powder

Bulk Magnet

Anisotropy + Coercivity

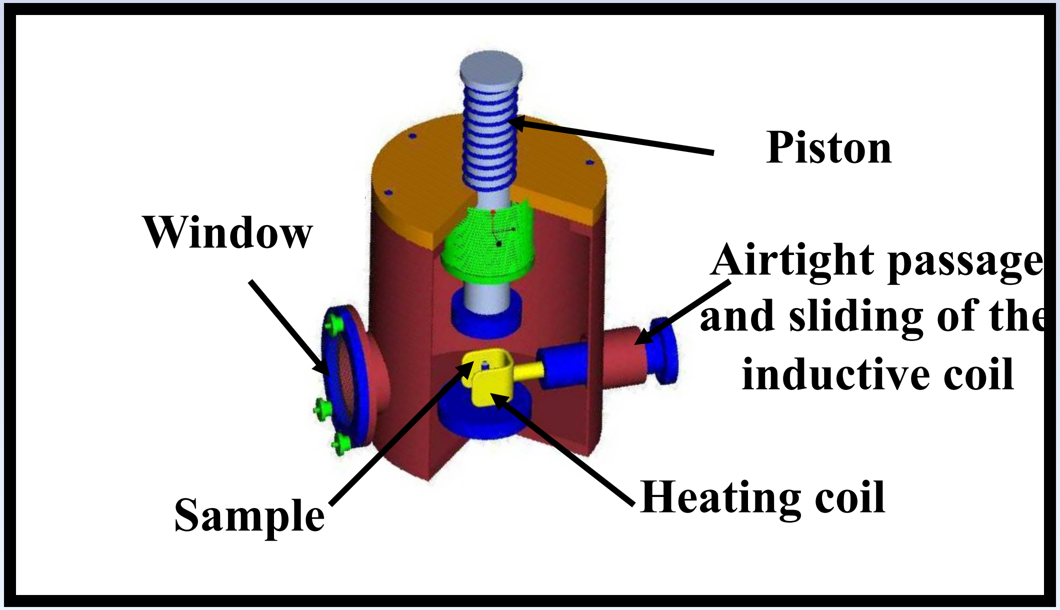


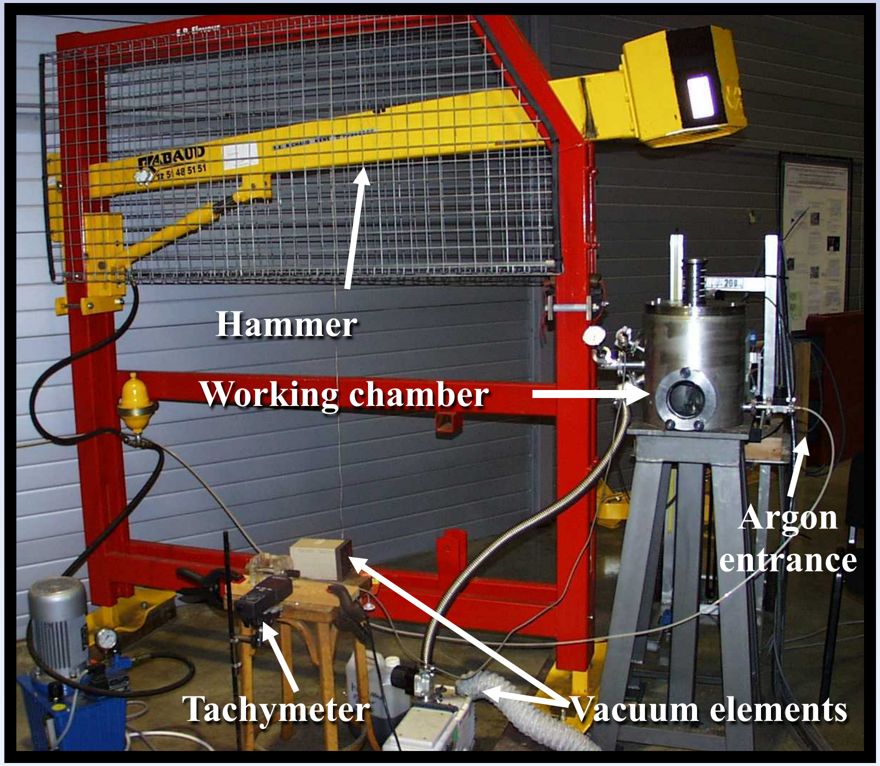
High-speed hot forging

$\dot{\varepsilon} = 125\ s^{-1}$

Thermomechanical treatment of the as-cast alloy to induce permanent magnet properties

EXPERIMENTAL DEVICE

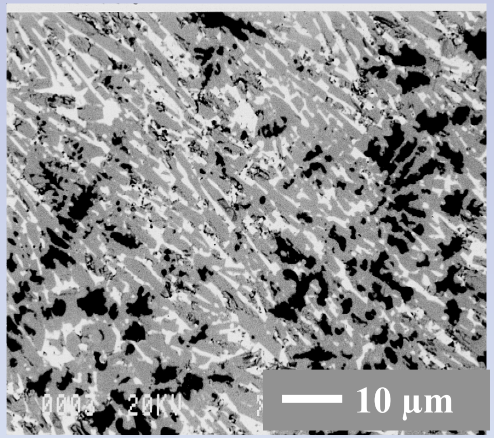


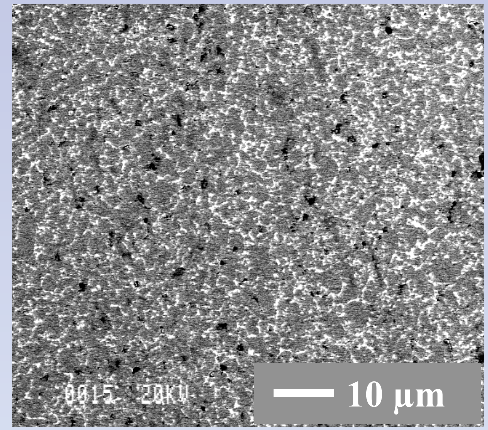


Microstructures

as-cast alloy | *forged sample*

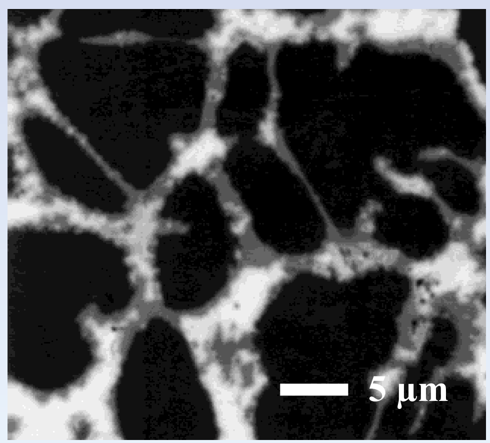
$NdFe_{14}B$

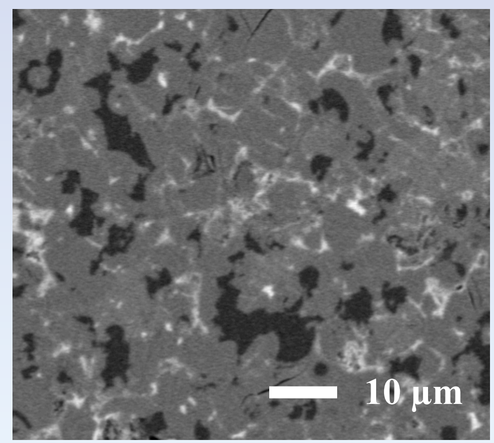




Black: iron
White intergranular phase
Grey: hard magnetic phase

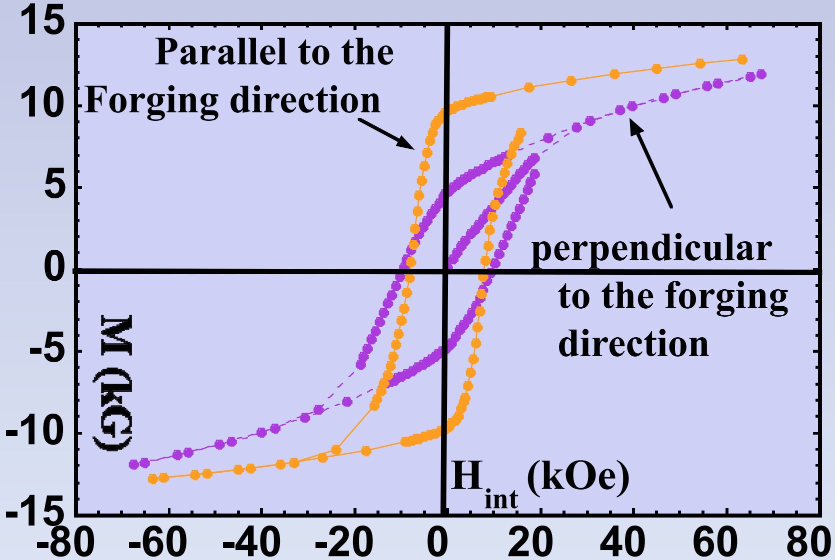
$NdFe_{10.5}V_{1.5}$





Permanent magnet properties

$Nd_{15,5}Fe_{78}B_5Cu_{1,5}$ alloys

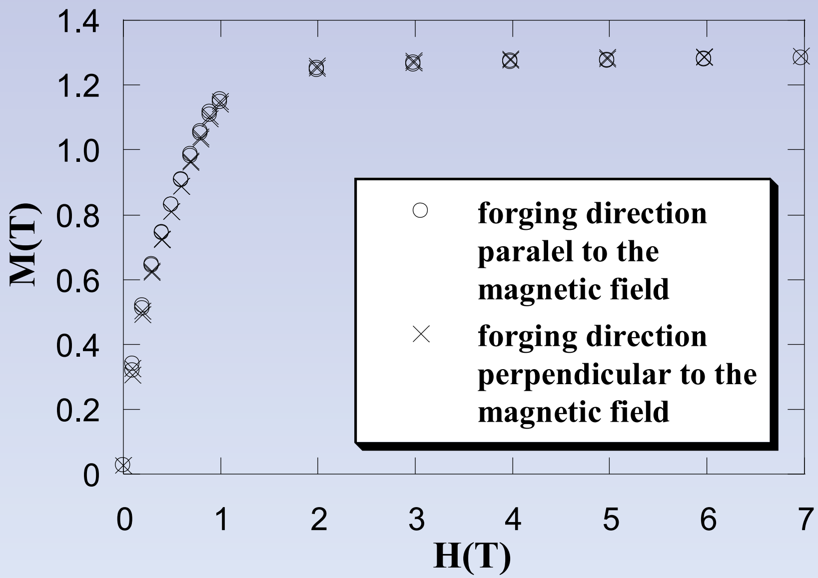


H_c = 10 kOe (795 kA/m)

B_r = 10 kG (1 T)

BH_{max} = 24 MGOe (191 kJ/m^3)

$NdFe_{10,5}V_{1,5}N$+10% Nd alloys

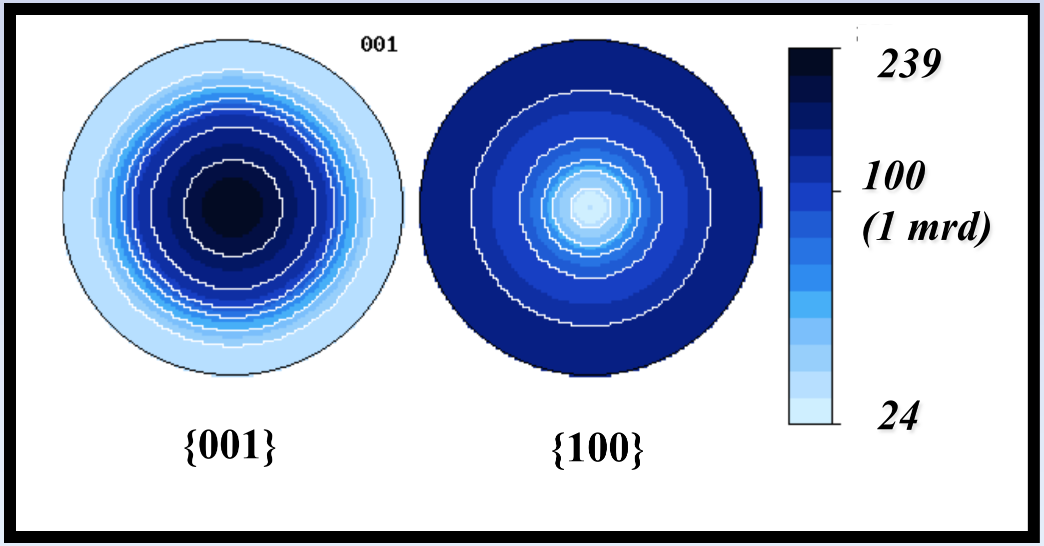


Isotropic magnet

Texture development in the $Nd_2Fe_{14}B$ phase

ILL, D1B

⇒ **Fibre texture of $Nd_2Fe_{14}B$**
⇒**c-axes // forging direction Y**



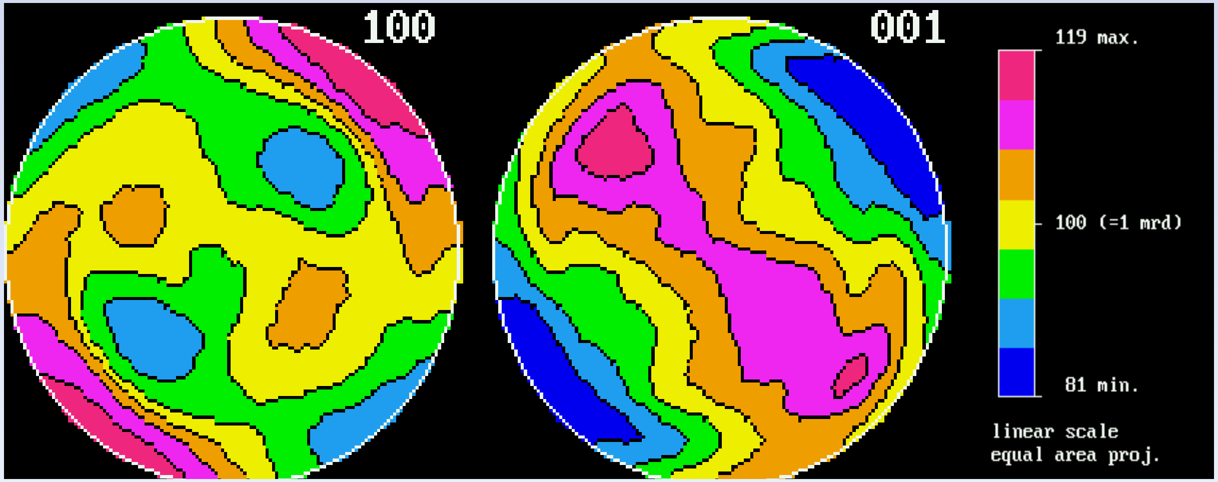
forging direction Y ⊙

Cristallographic texture ⇒ extrinsic anisotropy

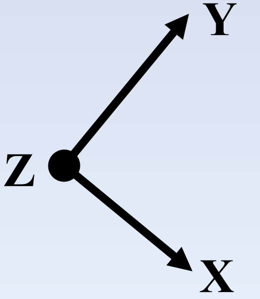
Texture investigation of the $NdFe_{10,5}V_{1,5}$ phase

ILL, D20

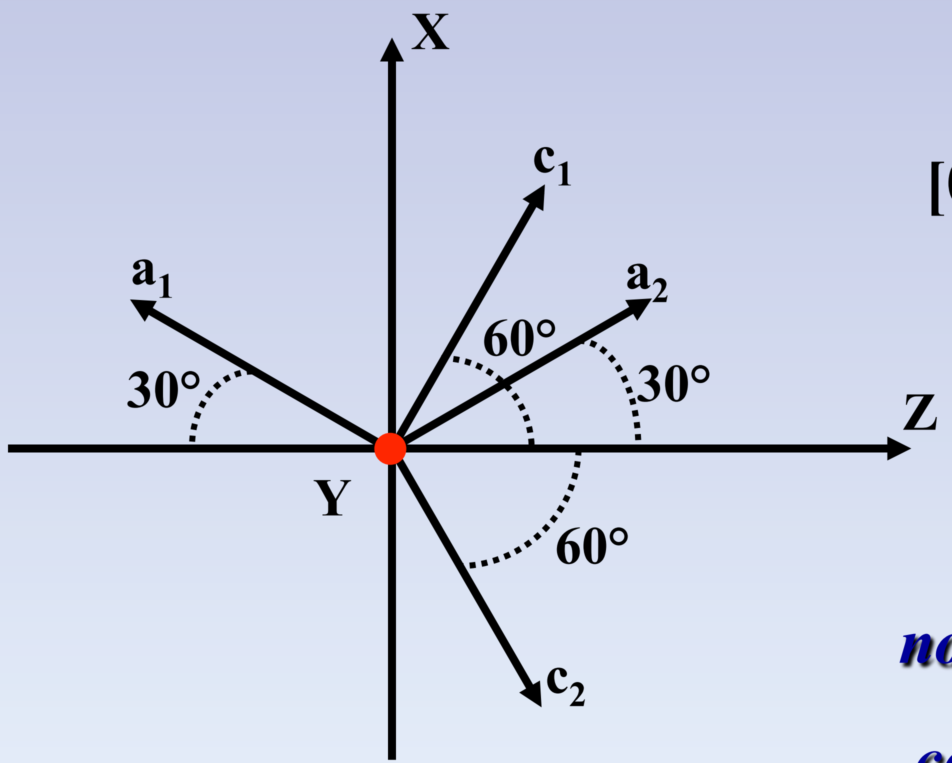
⇨ **texture with <100> axes // forging direction Y**
2 components, ≈ fibre



forging direction



Texture development of the $NdFe_{10,5}V_{1,5}$ phase

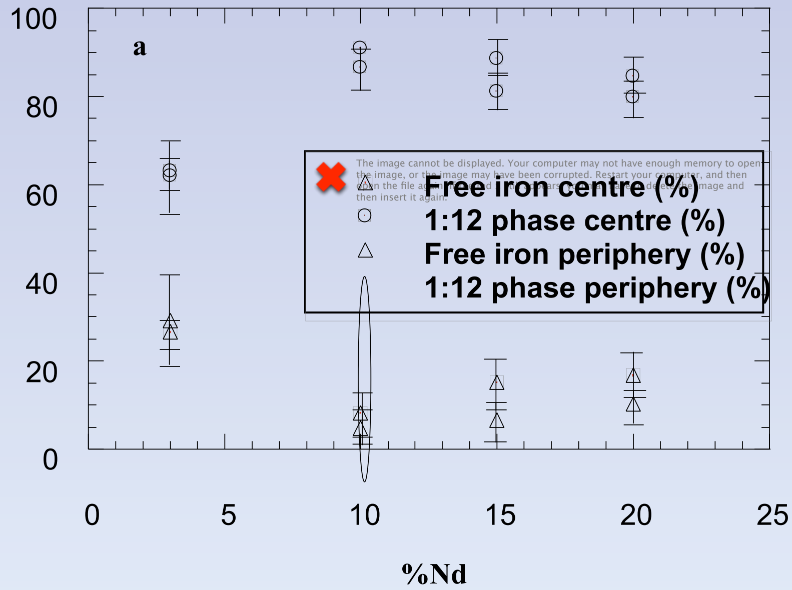


[010] // Y ⟺

[001] in (X,Z) plane ⊥ forging direction Y

no extrinsic magnetic anisotropy can be observed on forged bulks

Influence of primary iron on the $NdFe_{10,5}V_{1,5}$ phase stabilisation and texturing

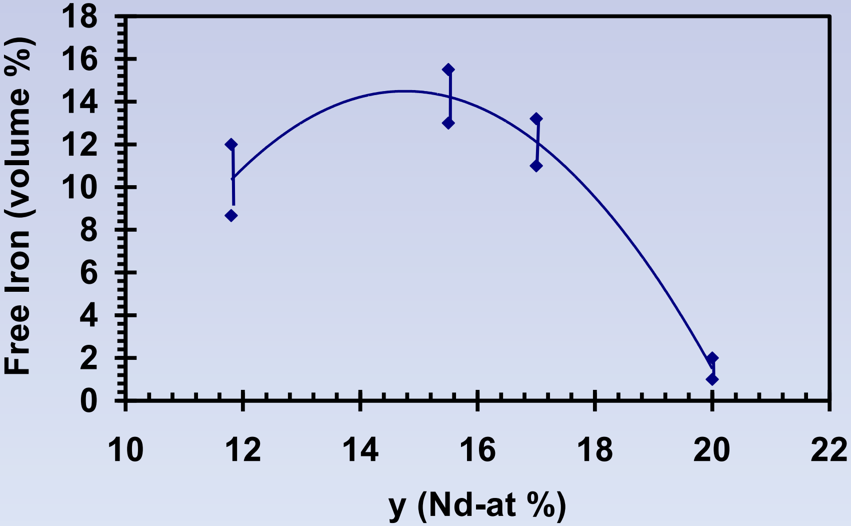


NdFeV alloys

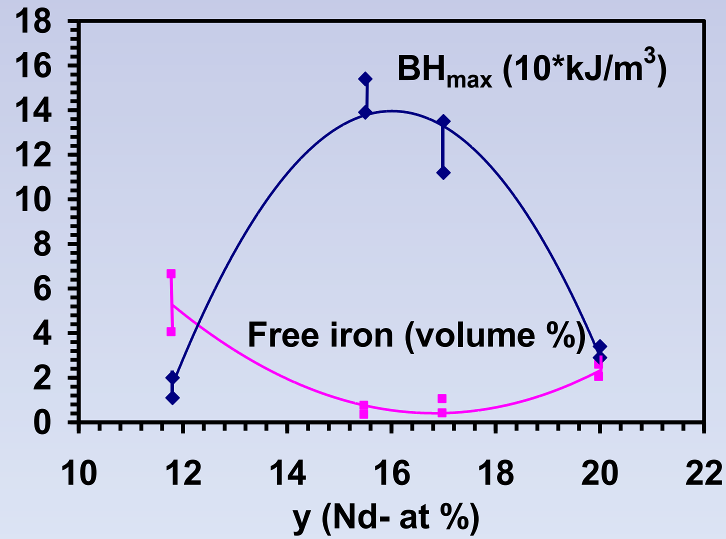
Nd-rich phase+ (Fe,V) → $NdFe_{10,5}V_{1,5}$

$NdFe_{10,5}V_{1,5}$ phase stabilisation: optimised microstructure for Nd=10%

Influence of primary iron on the $Nd_2Fe_{14}B$ phase texturing



NdFeBCu as cast alloys

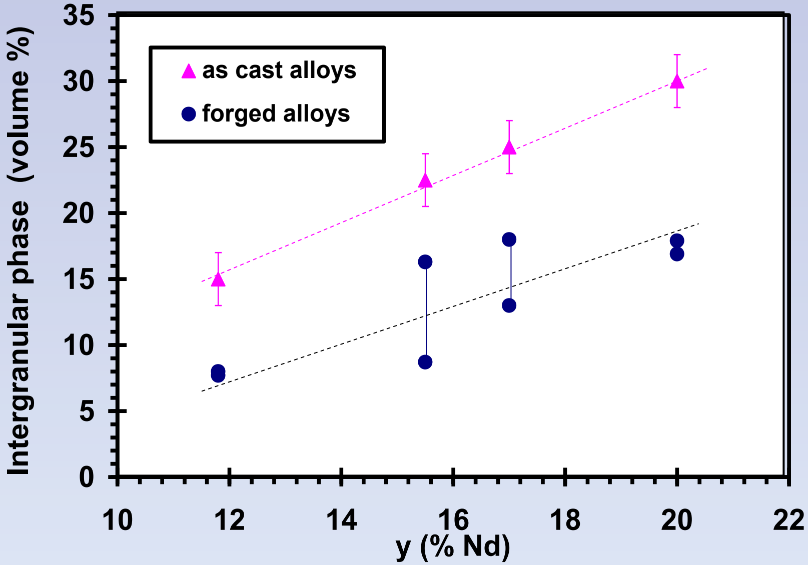


NdFeBCu forged alloys

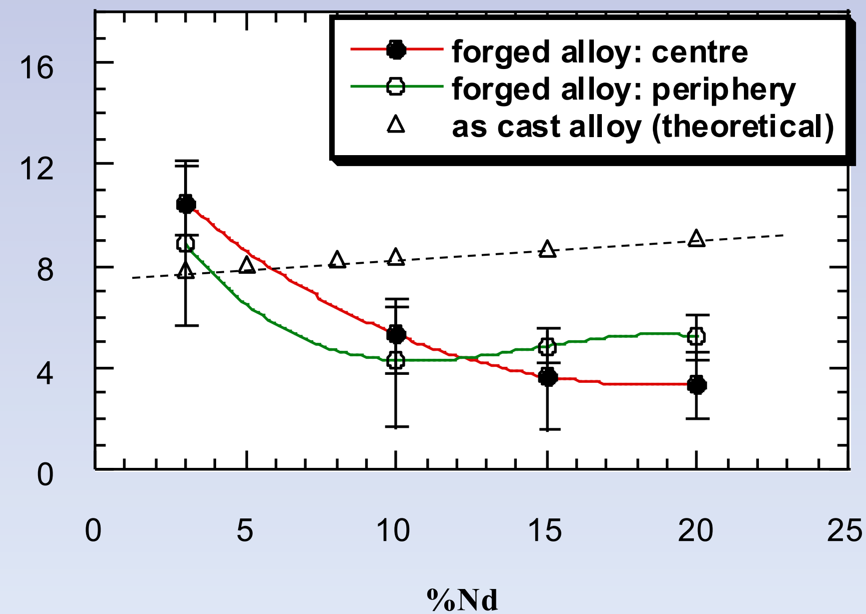
Optimised microstructure and magnetic properties for Nd=15,5%

Influence of the intergranular volume fraction on texturing

NdFeBCu alloys



NdFeV alloys



volume of intergranular phase = volume of liquid at T_f

NdFeBCu alloys

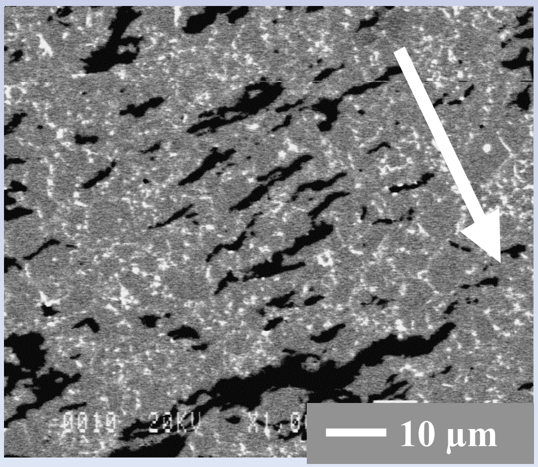
volume fraction ↑ with %Nd

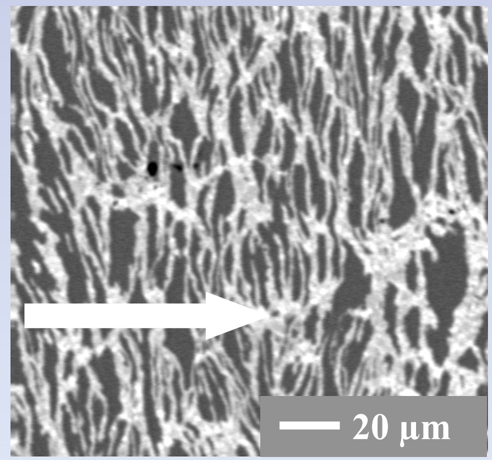
NdFeV alloys

%Nd>10%: liquid phase segregation

Effect of iron on the hot deformation process

$Nd_{15,5}Fe_{78}B_5Cu_{1,5}$ **$NdFe_{10.5}V_{1.5}$ + 10%Nd**

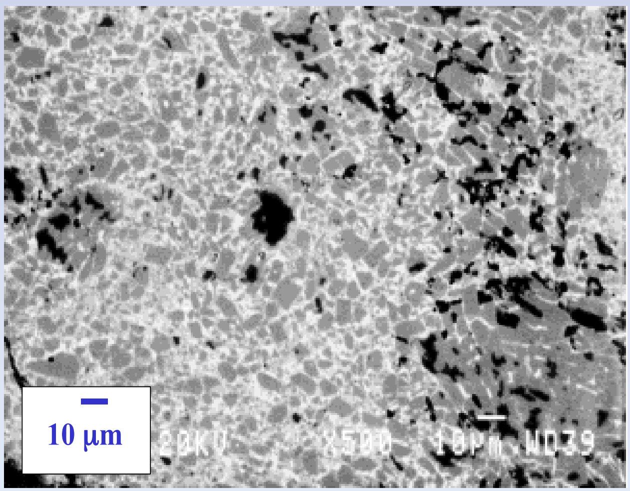




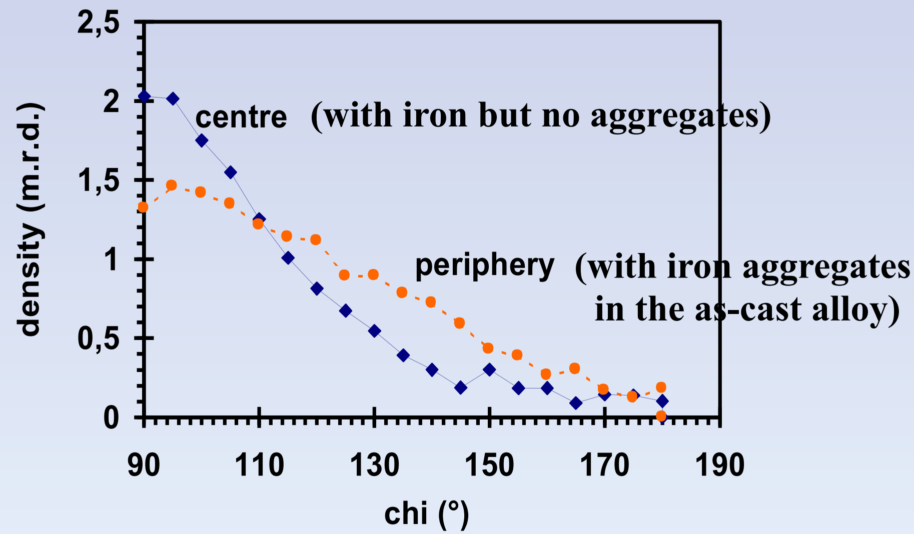
Forging energy mainly used for the plastic deformation of iron

Effect of iron on the hot deformation process

Inhomogeneous microstructure with non deformed areas in the periphery of the $Nd_{15,5}Fe_{78}B_5Cu_{1,5}$ alloy



weaker $Nd_2Fe_{14}B$ texture in the presence of iron aggregates



Large amount of iron or non homogeneous distribution detrimental to the alloy deformation process

On the texturing mechanism of the hard magnetic phase…

Some similitudes of the Nd-Fe-V and Nd-Fe-B alloys :

- TM-RE alloys
- Similar microstructures: nature of phases, proportions, distribution…
- At T_f: solid main phase, liquid intergranular phase
- At Tf: solid main phase in the brittle state

Uniaxial hot forging → Fibre texture in both cases

On the texturing mechanism of the hard magnetic phase…

… But different texturing mechanisms of the Nd-Fe-V and Nd-Fe-B alloys

NdFeBCu

-At T_f, deformation behaviour of an alloy in the semi-solid state

⇩

Fiber texture with <001> axis parallel to the forging direction

NdFeV

-At T_f, low liquid volume fraction → liquid not responsible for the main deformation process

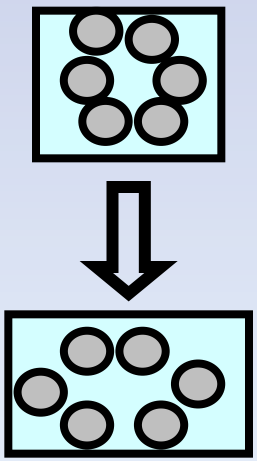
⇩

Fiber texture with <010> axis parallel to the forging direction Y + 2 three-dimensional components

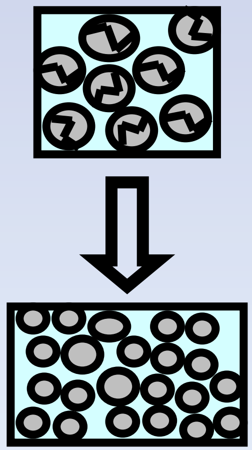
On the texturing mechanism of the hard magnetic phase…

… and different deformation behaviours

NdFeBCu

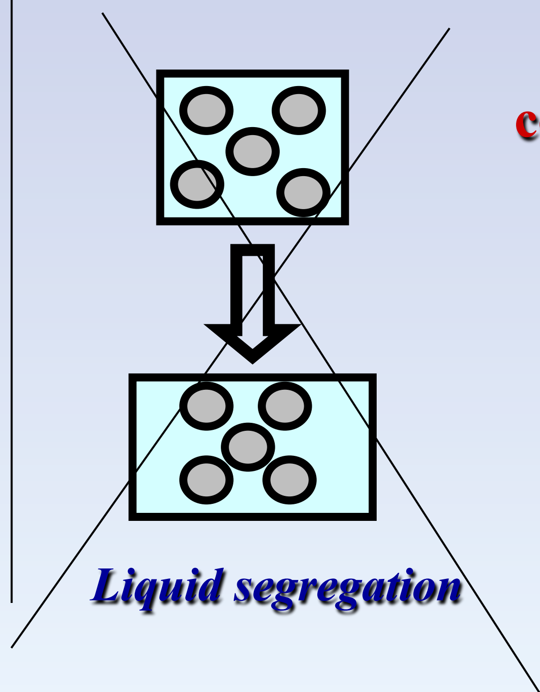


Homogeneous Flow
+
Rotation



Grain Cracking

NdFeV



Liquid segregation

complex mechanism…

$NdFe_{10,5}V_{1,5}$ crystallisation
+
crystallites orientation

Conclusion

NdFeBCu

- T_f= 930°C, Nd=15,5% (fl= 10%)

$\dot{\varepsilon} = 125\ s^{-1}$

Fiber texture with <001> axis parallel to the forging direction

-Coercive and anisotropic powder (patented process)

BH_{max} = 24 MGOe (191 kJ/m^3)

NdFeV

- T_f= 980°C, Nd-excess=10% (fl<5%)

$\dot{\varepsilon} = 125\ s^{-1}$

Fiber texture with <010> axis parallel to the forging direction Y + 2 three-dimensional components

-Magnetic properties still under optimisation