Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

Fe based bulk amorphous soft magnetic materials

Stefan Roth, Mihai Stoica, Jarmila Degmova*), Uwe Gaitzsch, Jürgen Eckert+), and Ludwig Schultz

IFW Dresden, Institute for Metallic Materials, Dresden, Germany
*) Department of Nuclear Physics and Technology, Faculty of Electrical Engineering and Information Technology, Slovak University of Technology, Bratislava, Slovakia
+) University of Technology Darmstadt, Department of Materials and Earth Sciences, Physical Metallurgy Division, Darmstadt, Germany

Supported by the EU, DFG, VEGA

Bulk amorphous metals for soft magnetic components

Advantage ?

- Shaping (direct casting, low processing temperature)
- magnetic properties ?
- non magnetic properties (mechanical, chemical) ?

melt

RQ-ribbon/flakes
milling **(additives)**
pressing/shaping

casting to final shape,
wall thickness in the
mm - range

component

$Fe_{65.5}Cr_4Mo_4Ga_4P_{12}C_5B_{5.5}$ BMGs: Preparation (cast)

Master Alloy

Metals:

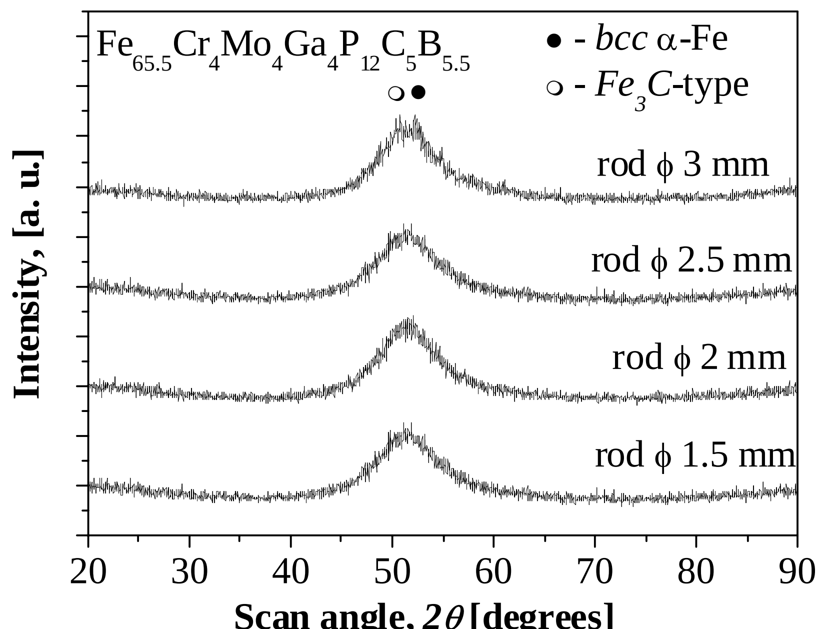
Fe, Cr, Mo

Prealloys:

Fe-Ga, Fe-P, Fe-B, Fe-C

Copper Mold Casting

Cast Samples

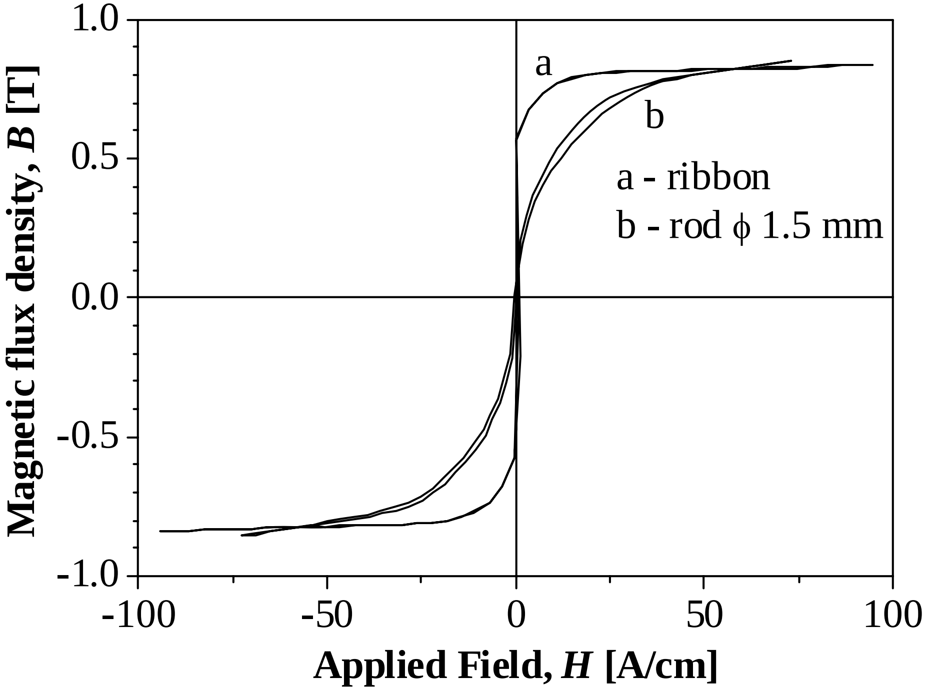


Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

Magnetic properties of as-cast glassy $Fe_{65.5}Cr_4Mo_4Ga_4P_{12}C_5B_{5.5}$

Sample	H_c [A/m]	J_s [T]
Ribbon	1.7	0.82
Rod ϕ 1.5 mm	5	0.81
Rod ϕ 2 mm	3	0.77
Rod ϕ 2.5 mm	9	0.78
Rod ϕ 3 mm	62	0.78
Disc ϕ 10 mm	6	0.80

Coercivity H_c and saturation polarization J_s

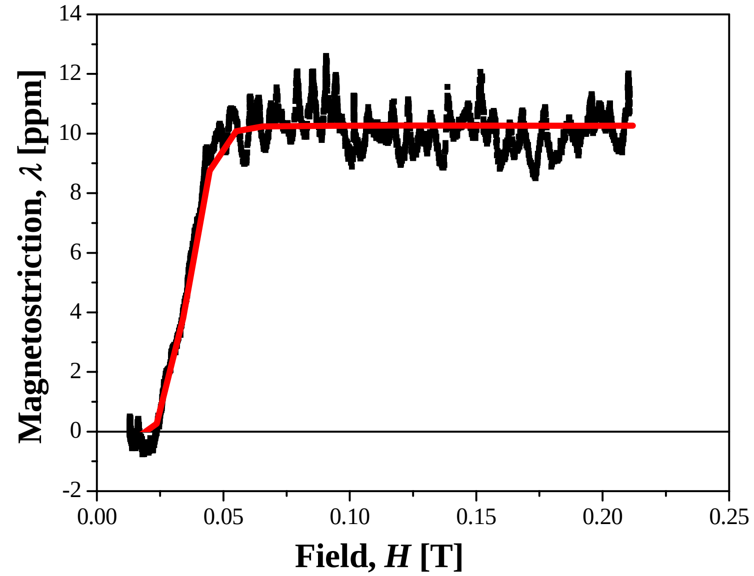


Typical hysteresis loops for ribbon and 1.5 mm rods

Magnetostriction - results

Composition	λ (ppm)
$Fe_{65.5}Cr_4Mo_4Ga_4P_{12}C_5B_{5.5}$	8.9
$Fe_{67.5}Cr_4Mo_4Ga_2P_{12}C_5B_{5.5}$	8.9
$Fe_{69.5}Cr_4Mo_4P_{12}C_5B_{5.5}$	8.5
$Fe_{71.5}Cr_2Mo_2Ga_2P_{12}C_5B_{5.5}$	16.2

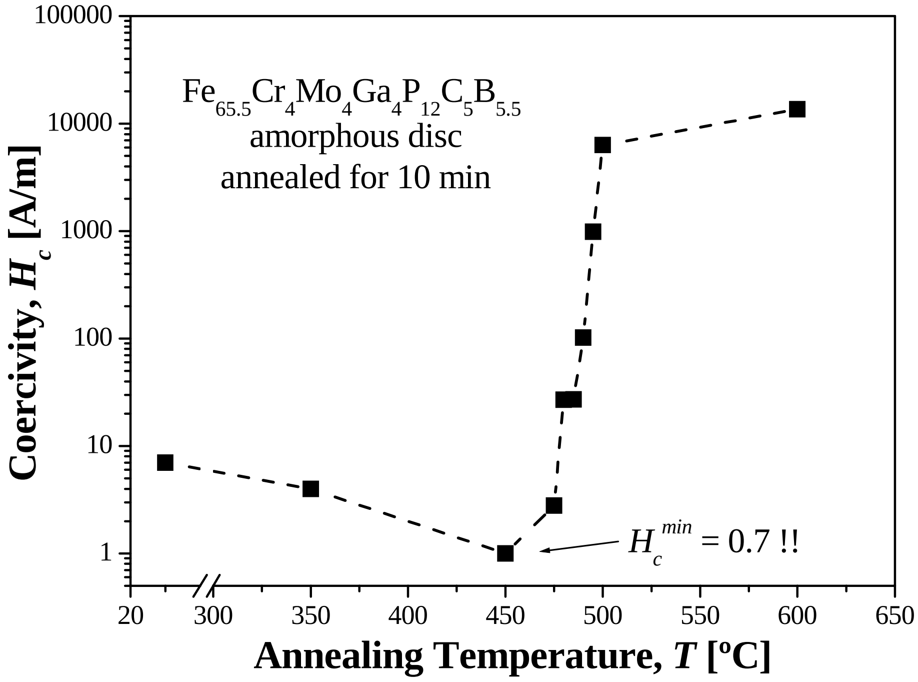
Amorphous ribbons (SAMR)



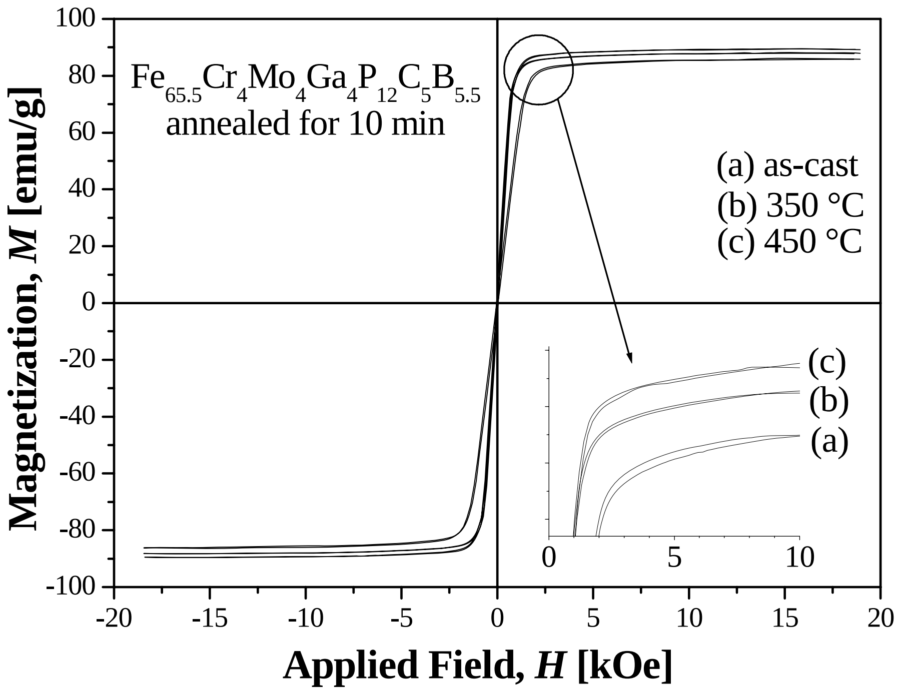
$Fe_{65.5}Cr_4Mo_4Ga_4P_{12}C_5B_{5.5}$
glassy disc (strain gauge)

Influence of annealing

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden



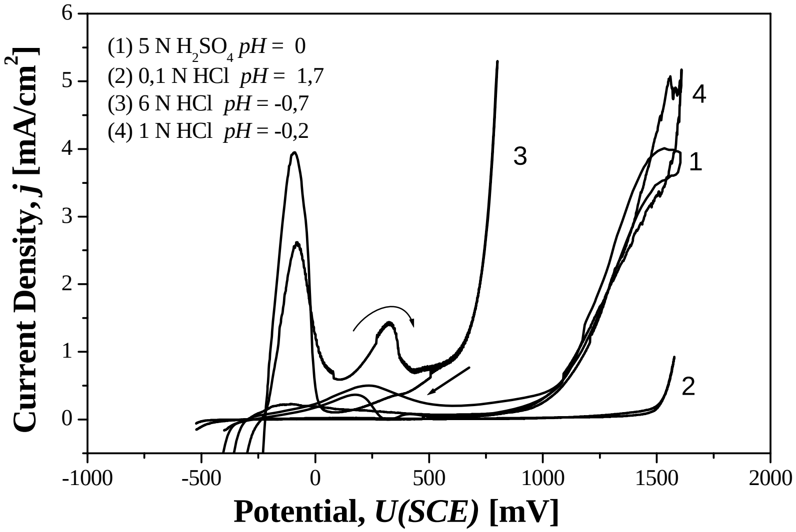
Coercivity H_c as a function of annealing temperature (t_{ann}=10 min)

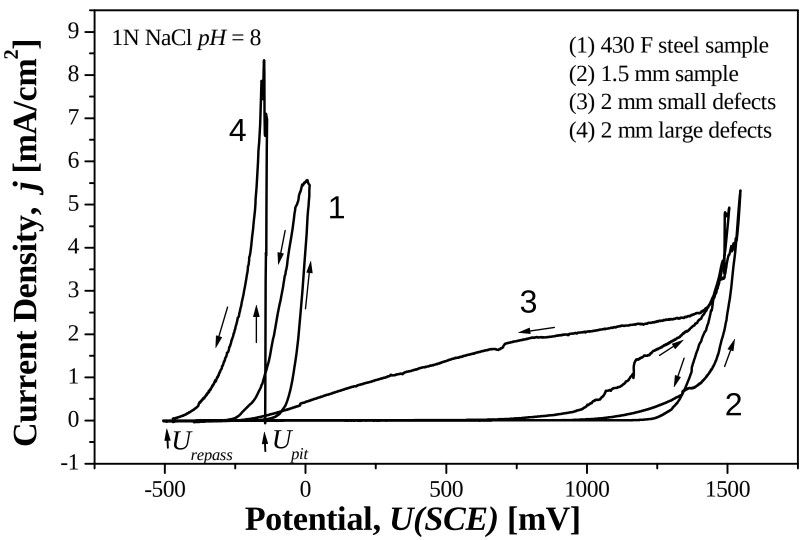


Comparison between hysteresis loops for as-cast and annealed samples

$Fe_{65.5}Cr_4Mo_4Ga_4P_{12}C_5B_{5.5}$ BMGs: electrochemical behavior

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden



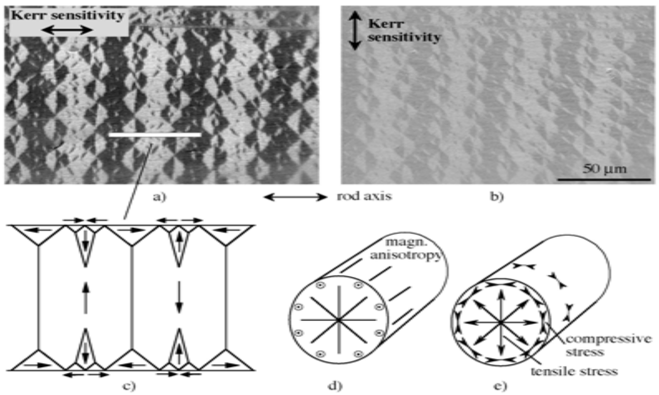


Properties \ Sample	1.5 mm sample		2 mm sample		430 F steel sample	
	pH = 8	*pH* = 0	*pH* = 8	*pH* = 0	*pH* = 8	*pH* = 0
Corrosion current density [µA/cm²]	1	20	1	800	0.2	6000
Max. of active dissolution [mA/cm²]	Not exist.	0.5	Not exist.	5	Not exist.	80
Passive current density [mA/cm²]	0.01 – 0.1	0.2 – 0.4	0.01 – 0.1	1 - 2	0.01 – 0.1	1.5 - 3

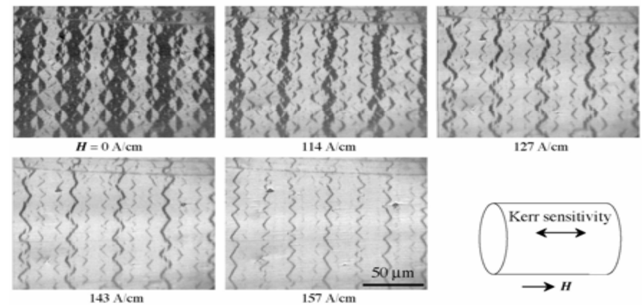
Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

Stress distribution derived from surface domains of an as–cast rod of amorpous $Fe_{77}Al_{2.14}Ga_{0.86}P_{8.4}C_5B_4Si_{2.6}$

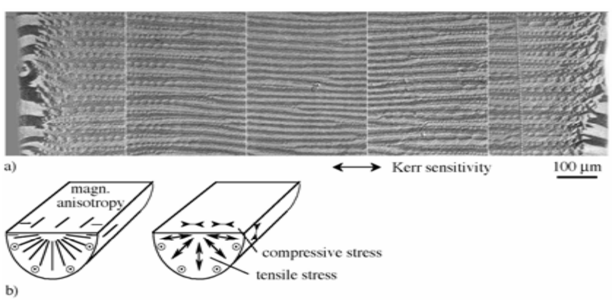
From: H. Grahl, S. Roth, R. Schäfer, J. Eckert, L. Schultz, SMM 16



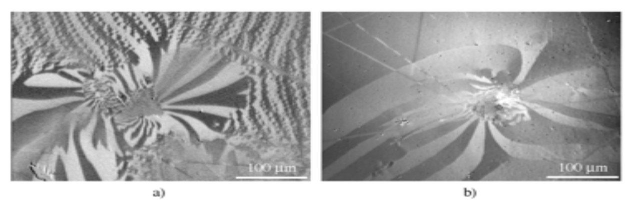
Surface domains of an as-cast rod, observed with a Kerr sensitivity along (a) and orthogonal (b) to the rod axis. The branched closure domain model (c) in the plane of a virtual cut through the rod centre can be derived under the assumption of a stress induced anisotropy as schematically explained in (d) and (e).



Reorganization of surface domains in a magnetic field along the rod axis.



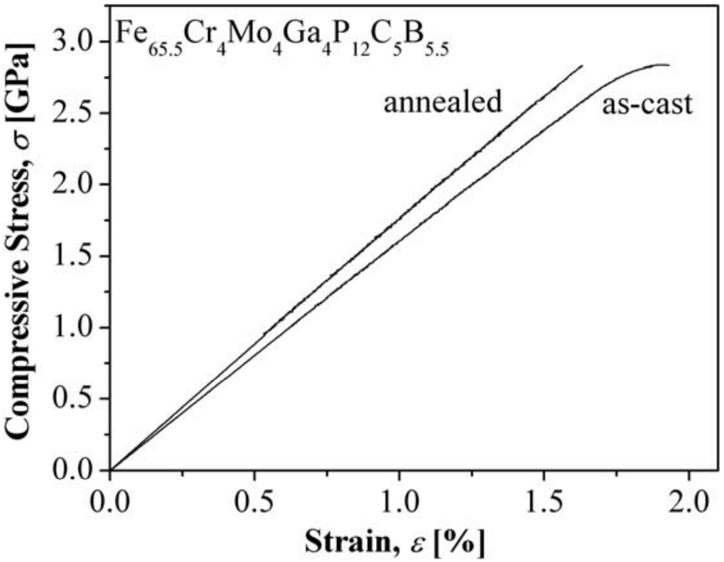
Domains along the rod diameter after cutting the rod in the middle. (b) assumed stress and anisotropy distribution.



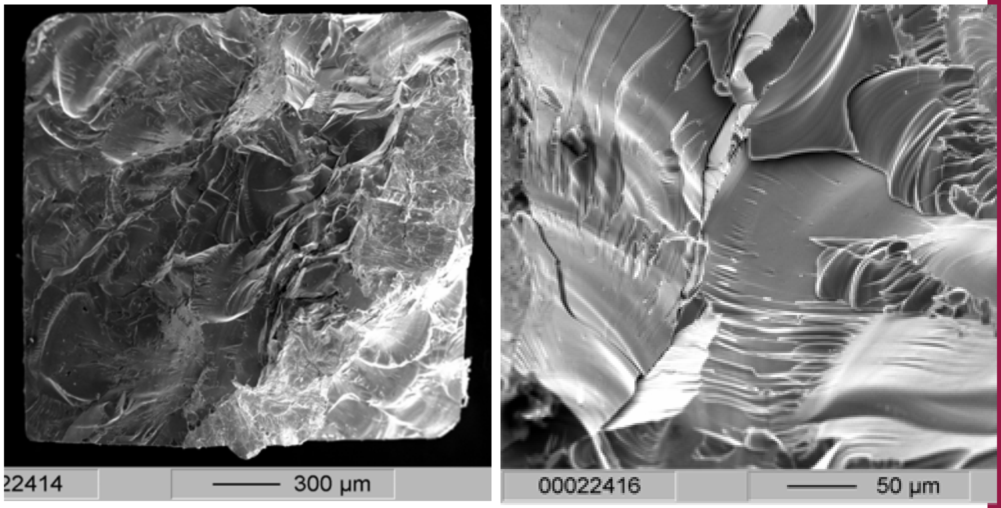
Domains around clusters of fine crystallites, observed somewhere in the center of a rod after cutting (a) and on the surface of an annealed rod (b).

Compression tests at $d\varepsilon/dt = 10^{-4}\ s^{-1}$ as-cast and annealed glassy $Fe_{65.5}Cr_4Mo_4Ga_4P_{12}C_5B_{5.5}$

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden



Fracture surface of as cast samples



Compression tests at $d\varepsilon/dt = 10^{-4}\ s^{-1}$ as-cast and annealed glassy $Fe_{65.5}Cr_4Mo_4Ga_4P_{12}C_5B_{5.5}$

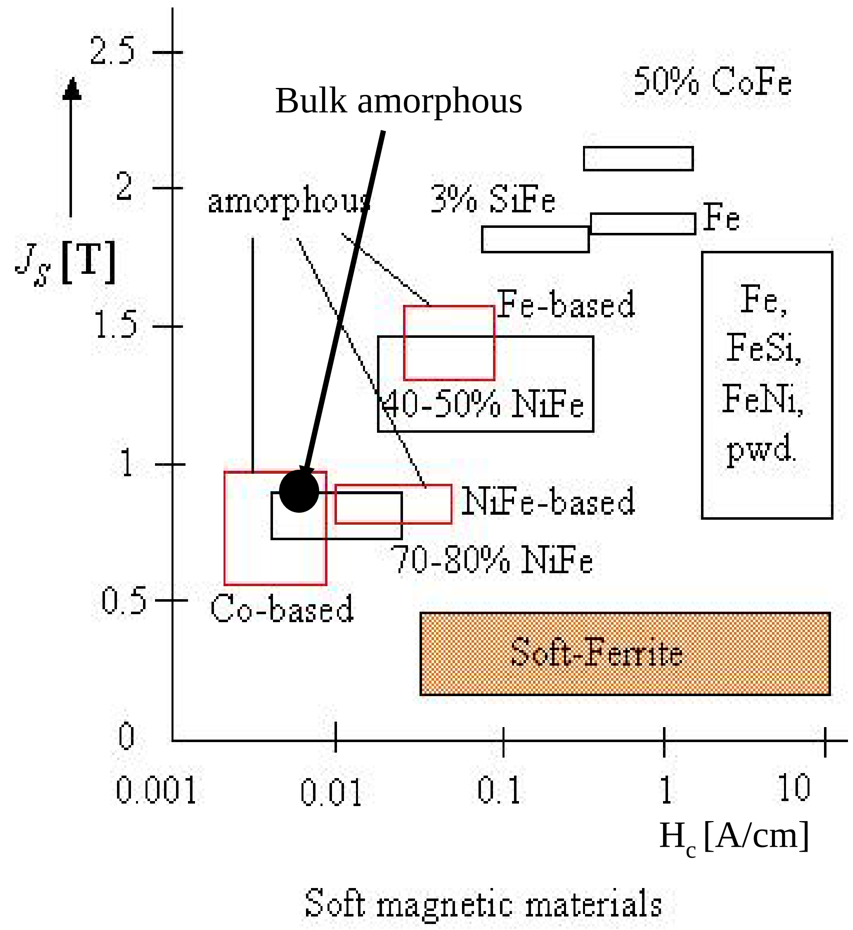
Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden

Sample	*E [GPa]*	σ_y *[GPa]*	σ_f *[GPa]*	ε_e *[%]*	ε_f *[%]*
rod ∅ 2 mm	160	3.19	3.23	2.03	2.33
rod ∅ 2.5 mm	170	3.27	3.27	1.19	2.03
bar 2x2 mm^2	161	2.82	2.84	1.76	1.91
bar 2x2 mm^2 annealed	177	2.84	2.84	1.60	1.63

Comparison to other high strengh alloys	Source	σ_f *(GPa)*	ε_f *(%)*	*E (GPa)*
FC20	Inoue et al. 2000	0.2~0.3	~ 0	(200)
Fe-based BMG	Inoue et al. 2003	3~4	~ 2	170
Zr-based BMG	Inoue et al. 1995	1.75	1.8	100
Zr BMG + *bcc*	Kühn et al. 2005	1.4	8	100

Bulk amorphous $Fe_{65.5}Cr_4Mo_4Ga_4P_{12}C_5B_{5.5}$ compared with other soft magnetic materials

Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden



Bulk amorphous $Fe_{65.5}Cr_4Mo_4Ga_4P_{12}C_5B_{5.5}$

- **Magnetic**
  J_s:0.8 T, H_c: 0.7... 5 A/m
- **Mechanic**
  ε: 1.5 - 2%, σ_y: ca. 3 Gpa
  Permalloy: σ_y: ca: 0.15 Gpa
  precipitation-hardend Py. σ_y: ca: 0.54 GPa
- **Corrosion resistance**
  better than type 430 stainless steel (16-17% Cr)
- **electrical resitivity**
  ρ: ca. 250 µΩcm
  Permalloy ρ: ca. 60 µΩcm

Bulk amorphous metals
for soft magnetic components

Advantage ?

- Shaping (direct casting, low processing temperature)
- magnetic properties
- non magnetic properties (mechanical, chemical) !

melt

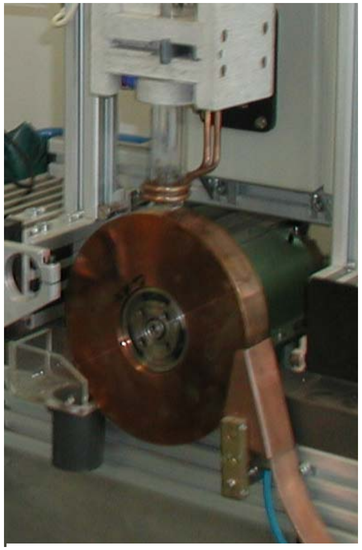
RQ-ribbon/flakes
milling (additives)
pressing/shaping

casting to final shape,
wall thickness in the
mm - range

component

Powdermetallurgical route

Master Alloy



Melt Spinning

Planetary Ball Mill

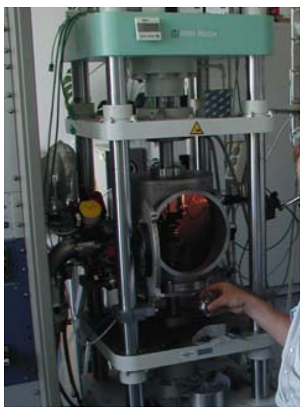
Hot Pressing

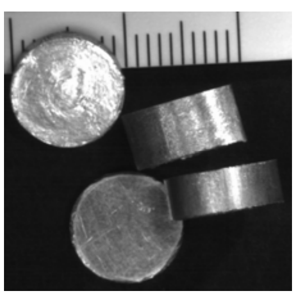
Sample for magnetic characterization

Pressed Samples

IFW
Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden

Experimental, Powder processing





Melt-spun $Fe_{77}Al_{2.14}P_{8.4}C_5B_4Ga_{0.86}Si_{2.6}$ ribbons cut and ball-milled:

- Retsch PM4000 planetary ball mill;
- under argon atmosphere;
- rotation velocities of the mill were 150, 200 and 250 rpm;
- milling time was 1 h.

Compaction to the bulk disc shaped bulk samples (diameter 10 mm and 3 mm high):

- in unaxial hot press;
- during 2 minutes;
- at the pressure of 900 MPa;
- temperatures were chosen from the range of the ΔT_x (=T_x-T_g) region.

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

Milling Induced Changes

The process of ball milling induces internal mechanical stress in the material and produces surface roughness.

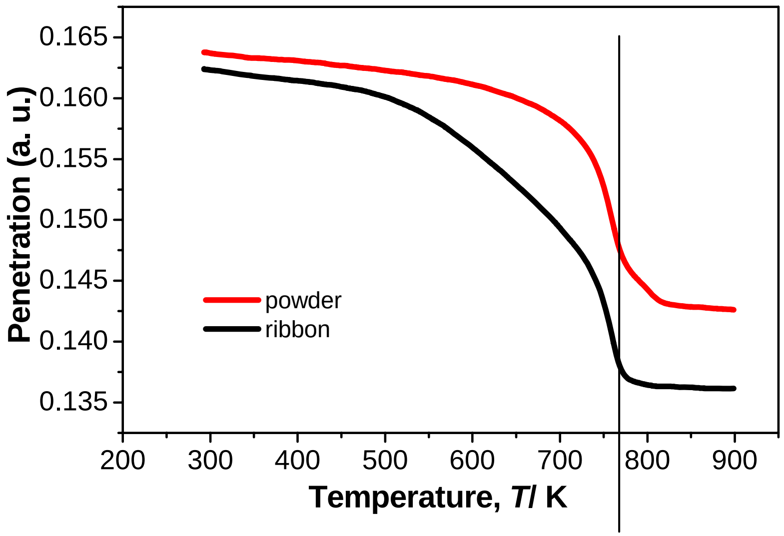
Accordingly, the thermal, structural and magnetic properties of the resulting powder are different from those of the initial melt-spun ribbon.

After ball milling for 1 h the oxygen content doubled
=> melt spun ribbons 0.04 ± 0.01 at. %
=> ball- milled powders 0.08± 0.01 at. %

The width of the supercooled liquid region (ΔT_x) is influenced by ball-milling because the oxygen impurities enhance crystallization.

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

Milling Induced Changes



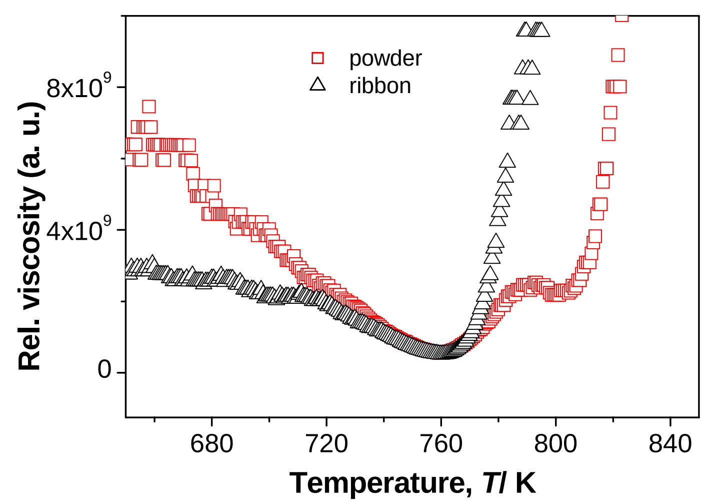
The limiting values of the ΔT_x temperature region are important for compaction because of the material softening which starts at temperatures above T_g

Above T_g :

=> distinct softening of the sample (i.e. penetration of the probe)

=> decrease of the viscosity

easy deformation and working



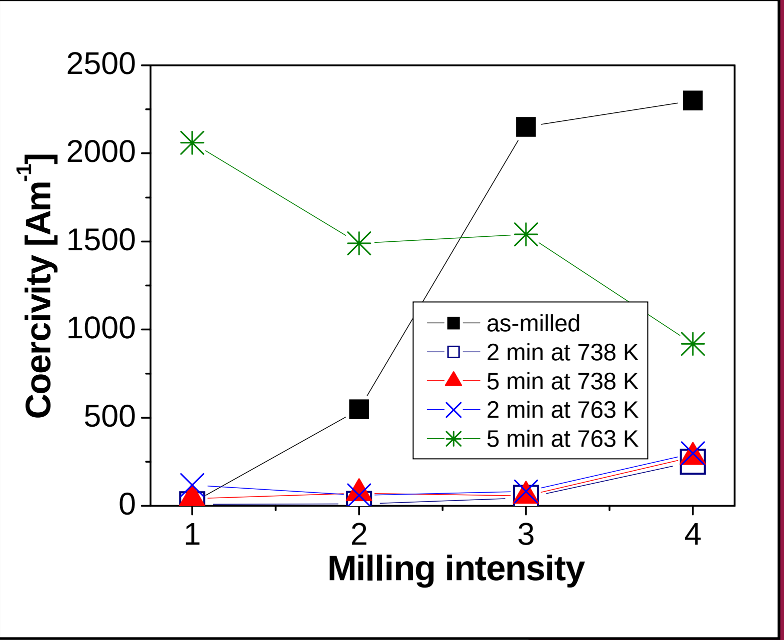
IFW
Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

Annealing behaviour

	Hc [A/m]				
Sample	As-cut As-milled	2 min at 738 K	5 min at 738 K	2 min at 763 K	5 min at 763 K
Ribbon (1)	16	8.5	40	118	2060
150 rpm (2)	547	9.5	71	59	1490
200 rpm (3)	2150	44.3	56	82	1540
250 rpm (4)	2300	250	277	297	919

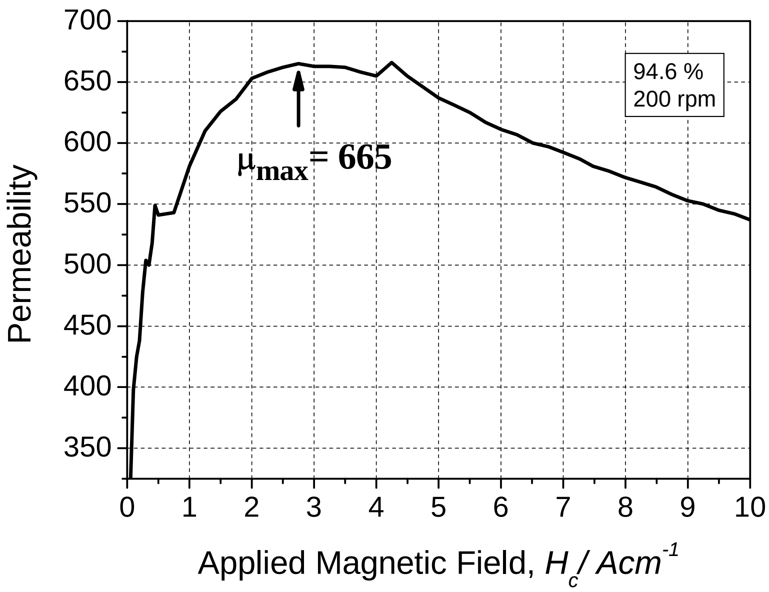
Melt-spun glassy ribbon: $H_{c\ \text{as prepared}} \approx 7\ Am^{-1}$

Ball milling-induced stress causes significant differences in coercivity (H_c) between the melt-spun ribbon and the ball-milled powder.



Measurement of hysteresis loops and permeability

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden



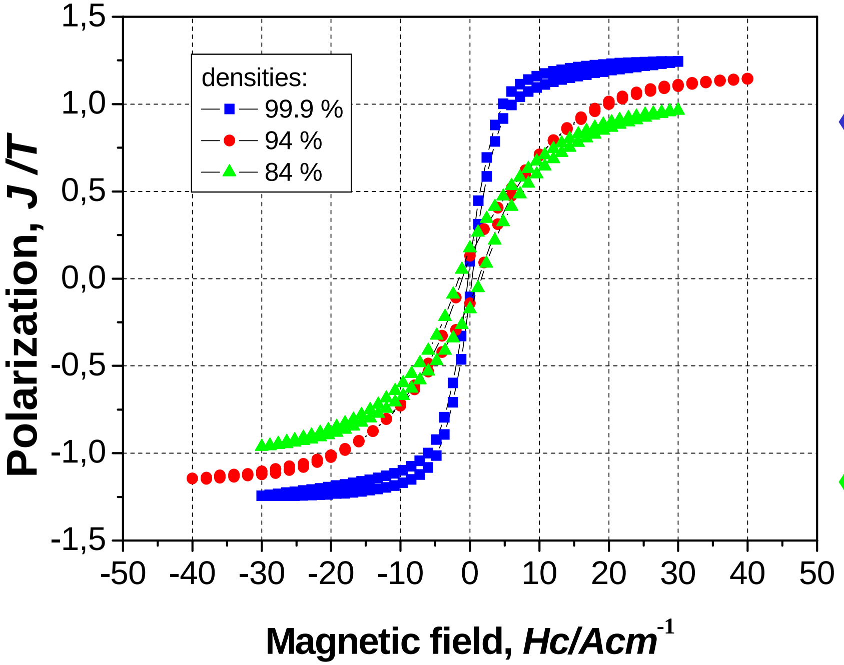
Maximum permeability of FeAlGaPCBSi bulk samples increases with an increase of their relative density.

For the sample with:

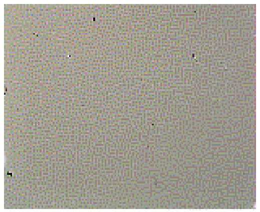
- 84 % of relative density μ = 630 ;
- 94.6 % of relative density μ = 665 ;
- 99.9% of relative density μ = 2530.

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

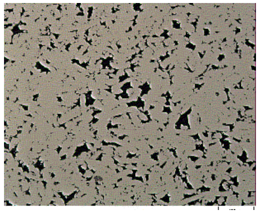
Measurement of hysteresis loops and permeability



J_s = 1.36 T



J_s = 1.17 T



Bulk amorphous metals for soft magnetic components

Advantage ?

- Shaping (direct casting, low processing temperature)
- magnetic properties
- non magnetic properties (mechanical, chemical) !

melt

RQ-ribbon/flakes
milling **(additives)**
pressing/shaping

casting to final shape,
wall thickness in the
mm - range

component

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

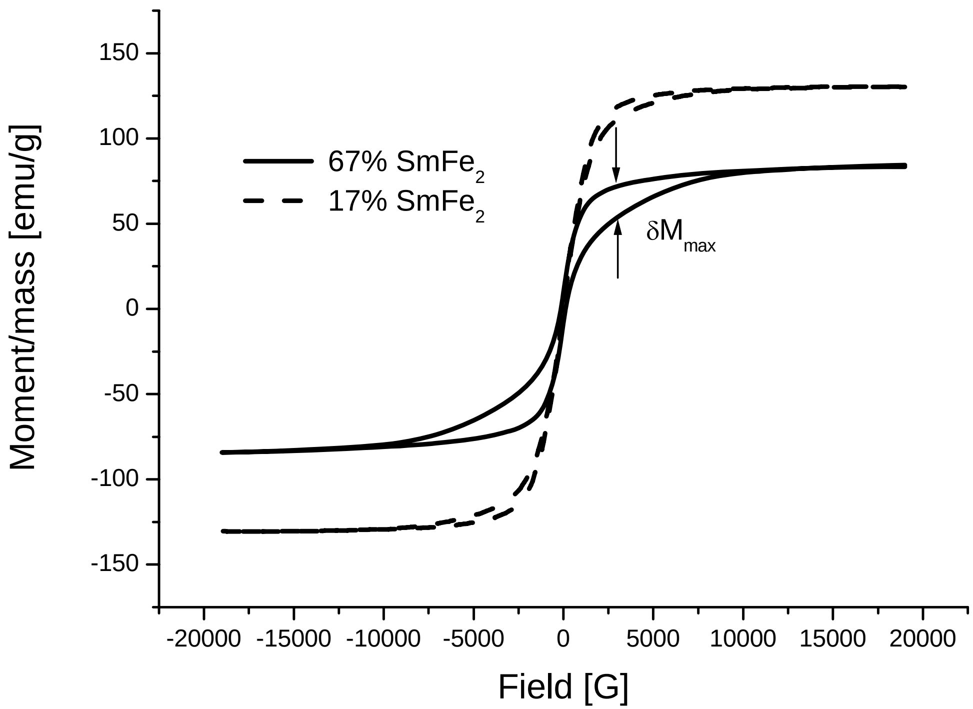
Composite

$(Fe_{77}Al_{2.14}P_{8.4}C_5B_4Ga_{0.86}Si_{2.6})$ - (Fe_2Sm)

FeAlPCBGaSi:	bulk morphous metal processable by powder technology high mechanical strength good soft magnetic properties
Fe_2Sm:	high magnetostriction
Preparation:	mixing of **S**eparately milled powders milling of powders **T**ogether

$(Fe_{77}Al_{2.14}P_{8.4}C_5B_4Ga_{0.86}Si_{2.6})$ - (Fe_2Sm) – composite

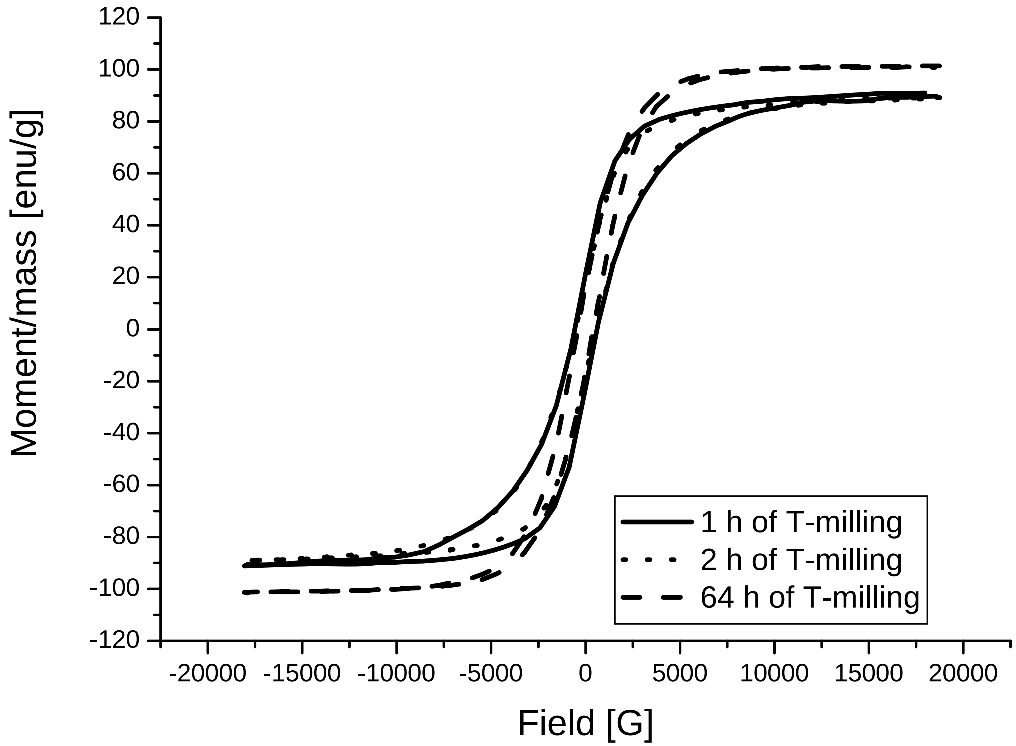
(2h S-milling)



Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

$(Fe_{77}Al_{2.14}P_{8.4}C_5B_4Ga_{0.86}Si_{2.6})$ - (Fe_2Sm) – composite

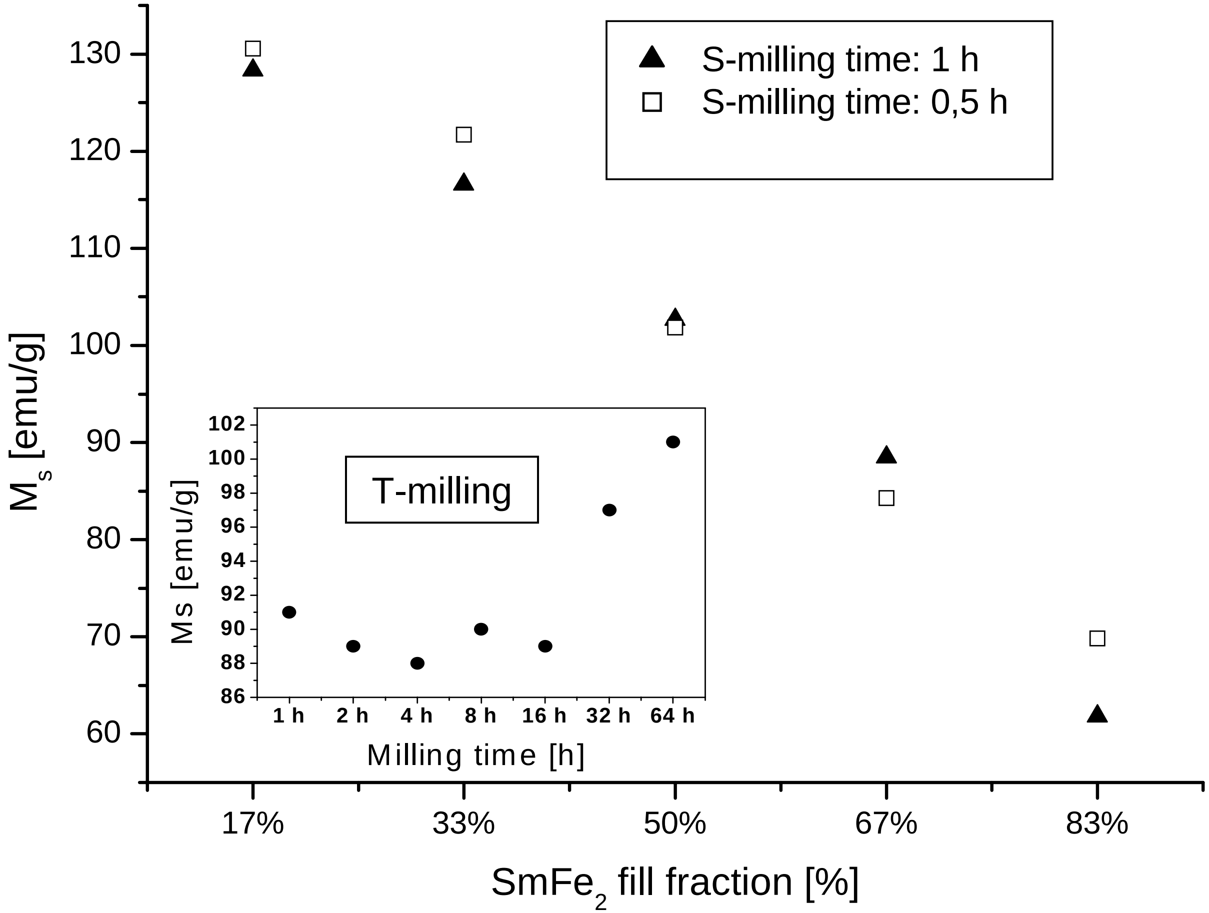
67% $SmFe_2$ (T-milling)



Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

Magnetisation, for various treatments

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

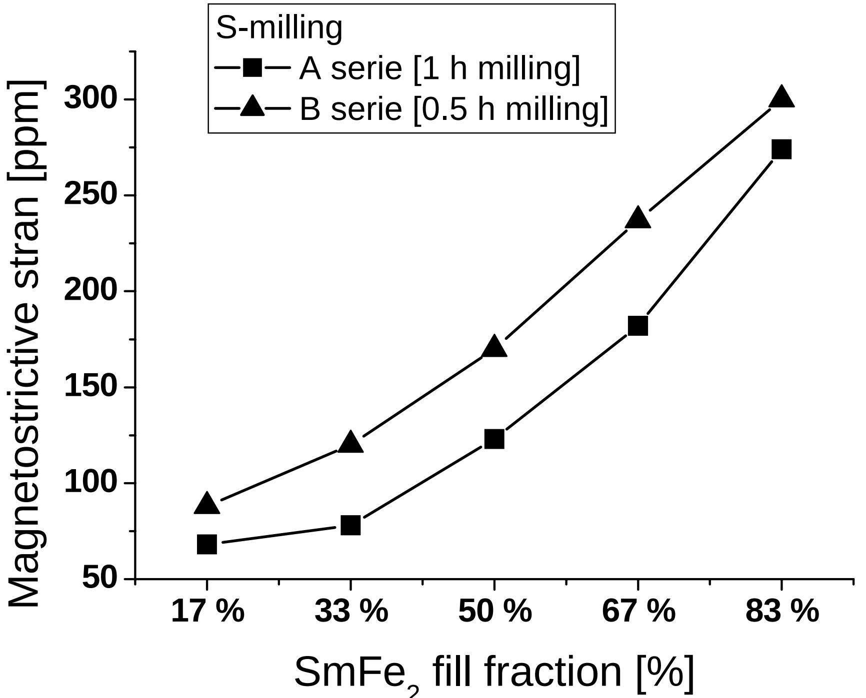


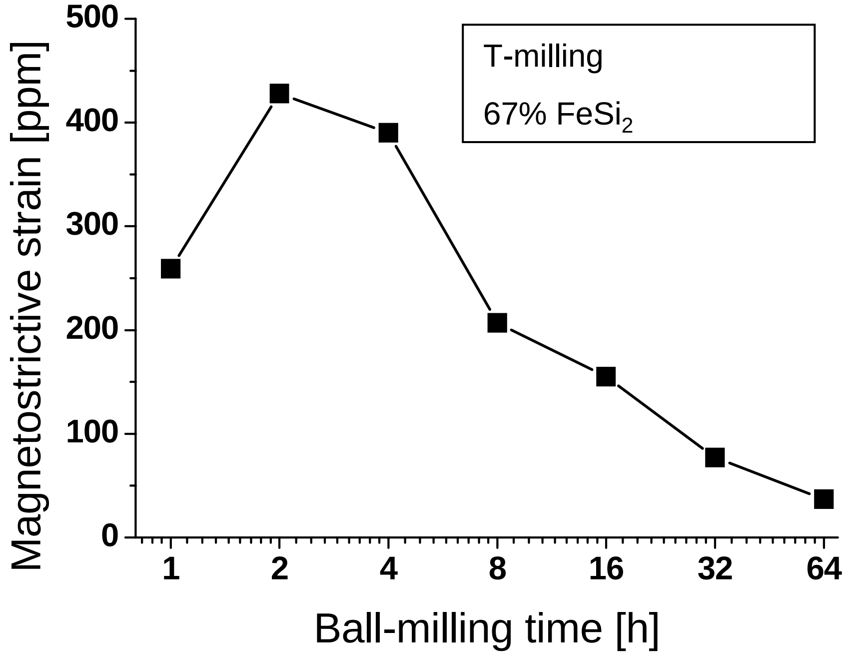
Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

Bulk amorphous Fe-base alloy – $SmFe_2$ composites
Magnetostriction, for various treatments

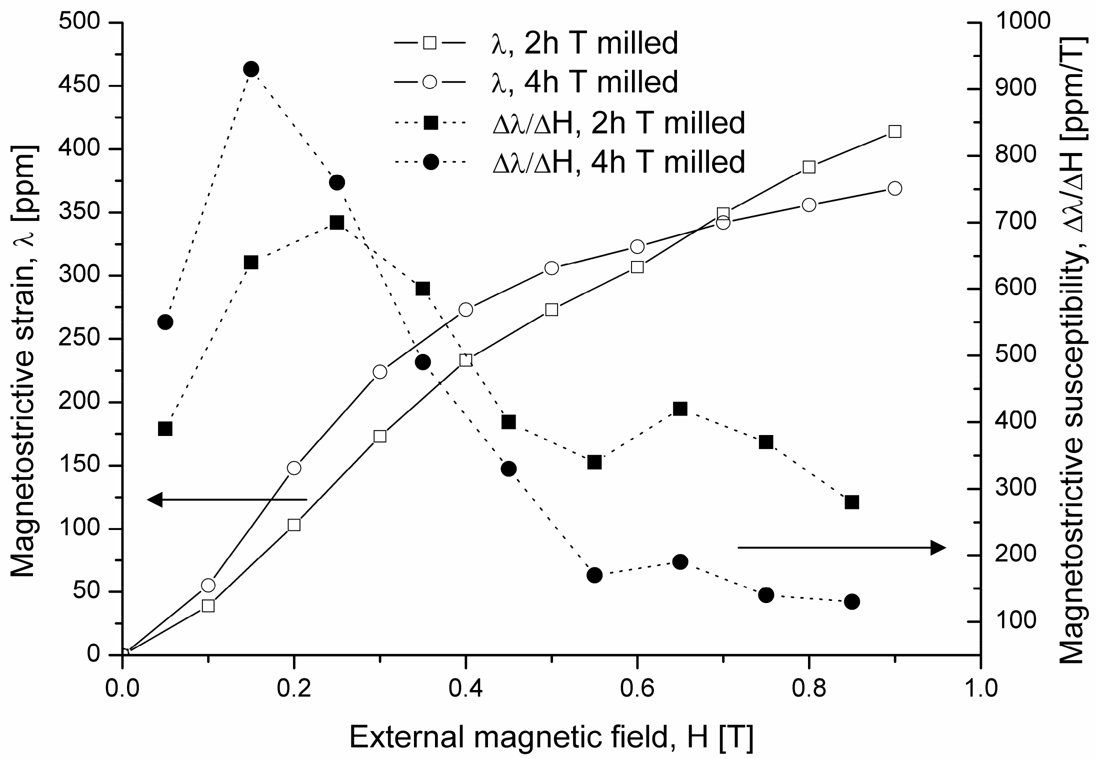
separate-milling, vs. amount of $FeSi_2$

together-milling, 67% $FeSi_2$ vs. milling time





Bulk amorphous Fe-base alloy – 67%$SmFe_2$ composites
Magnetostriction, magnetostrictive susceptibility vs. field



Δλ/ΔH ~920 ppm/T @ 0.1T (4h T- milling)

~700 ppm/T @ 0.2T (2h T - milling)

Bulk amorphous Fe-base alloy – $SmFe_2$ composites
Comparison with Fe – $SmFe_2$ composites

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

J. Appl. Phys., Vol. 83, No. 11, 1 June 1998 Pinkerton *et al.* 7252

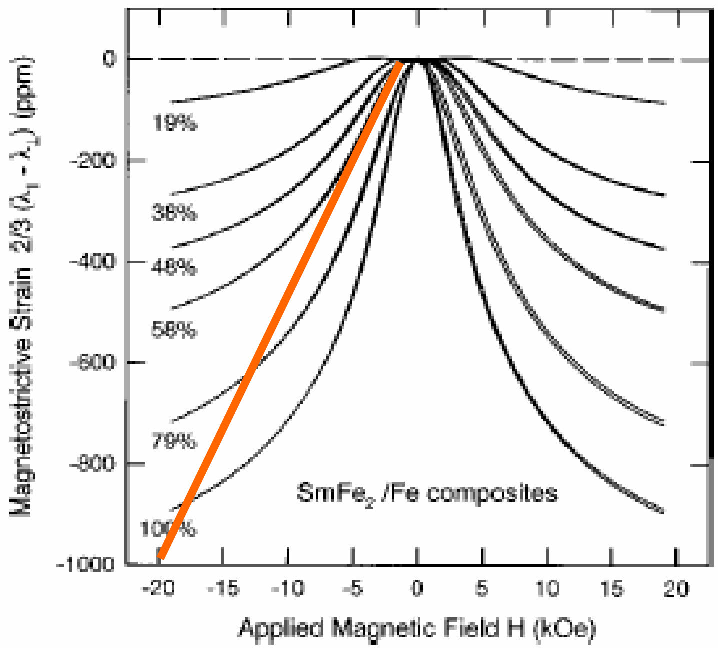


FIG. 1. Magnetostriction $2/3(\lambda_{\parallel}-\lambda_{\perp})$ as a function of applied mag field H for hot pressed $SmFe_2$/Fe composites made at various $SmFe_2$ fractions.

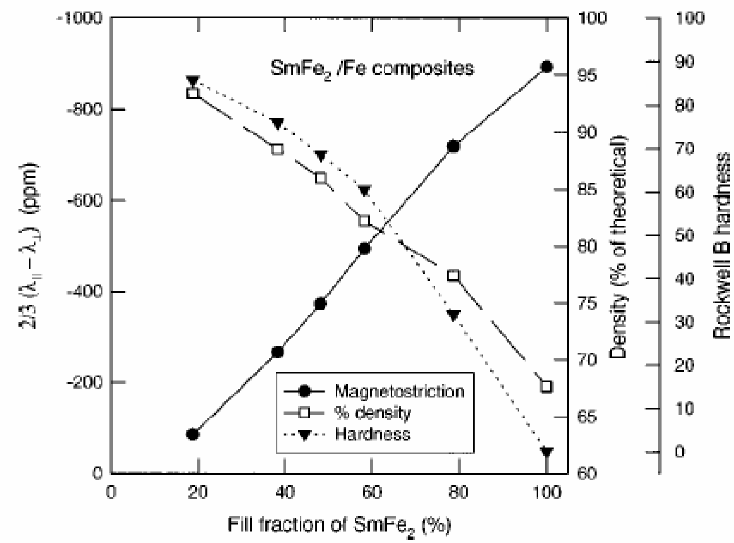
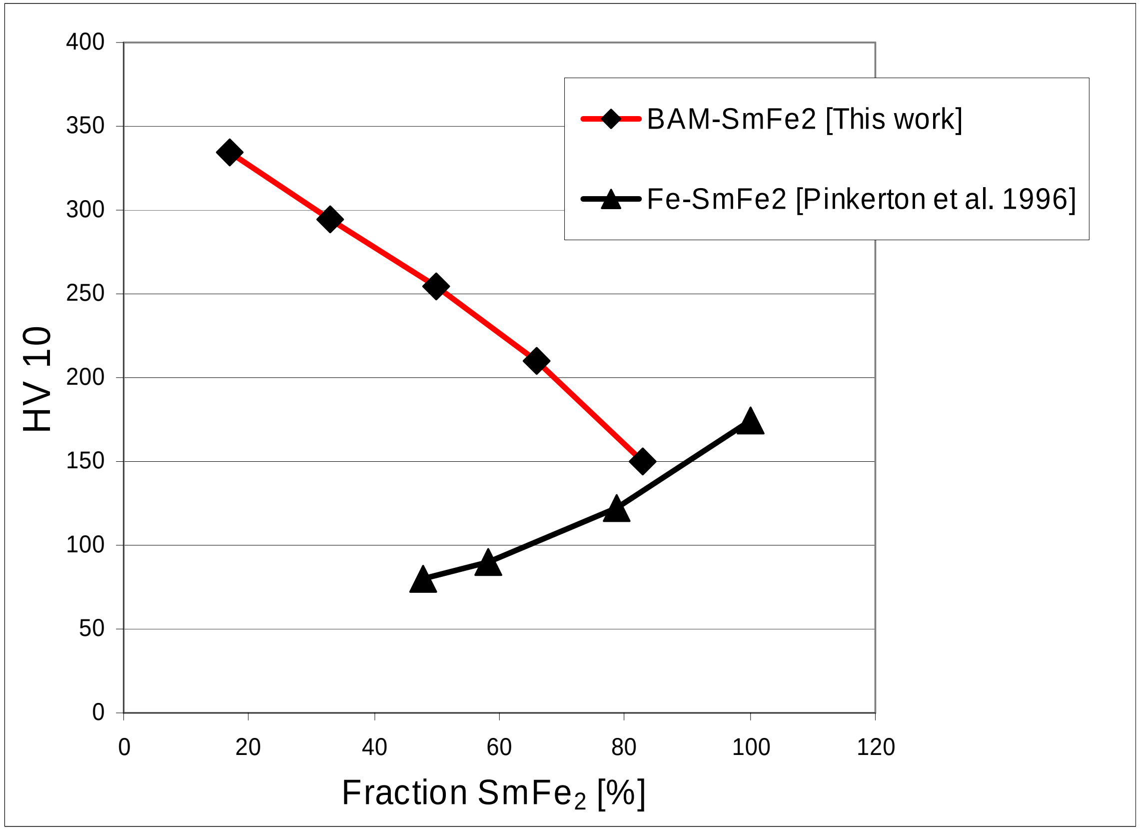


FIG. 3. Dependence of the magnetostriction $2/3(\lambda_{\parallel}-\lambda_{\perp})$ at $H=19$ kOe (●), the density ρ normalized to the theoretical density $\rho_{theoretical}$ (□), and the hardness (▼) on the volume fraction of $SmFe_2$ in hot pressed composites made with an Fe matrix.

$\Delta\lambda/\Delta H$ ~ 500 ppm/T @ 0.5T

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

Hardness of $SmFe_2$ - Fe/BMG - composites



Data of Pinkerton et al. (1998) are transformed from HRB to HV

Leibniz-Institut
für Festkörper- und
Werkstoffforschung
Dresden

Conclusions

Bulk metallic glasses on iron base are well suited for driving magnets, moving magnets or magnetic yokes for service in rough environment, e.g.:

- magnetic valves (pneumatic, hydraulic)
- clutches
- locking bolts
- actors
- switches

Improve manufacturing