National Academy of Sciences of Ukraine

Institute of Magnetism

# Thin Films of Ni-Mn-Ga


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# In collaboration with

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- Mullner P., Boise University, Boise, USA
- Kohl M., IMT, Forschungszentrum Karlsruhe, Germany
- L'vov V.A., Taras Shevchenko University, Kiev, Ukraine
- Ohtsuka M., IMRAM, Tohoku University, Sendai, Japan

# PLAN

1 **Introduction**

2 Submicron Ni-Mn-Ga/substrate thin films ferromagnetic martensites:
----Fabrication by magnetron sputtering
---- Transformation behavior
---- Structure and substructure
---- Magnetic properties

Generally, all properties are found to be dependent regularly on film thickness and substrate nature

3 Actuation ability
Current work

# Development of thin film technologies of FSMA's

## (1) Free-standing NiMnGa films

They are implemented and characterized as actuators: optical microscanner

## (2) ***submicron*** Ni-Mn-Ga/substrate thin film composites

Possible applications: magnetic MEMS, microsensors, memory storage etc.

**Fabrication methods used**: molecular beam epitaxy, magnetron sputtering, evaporation technique, laser ablation and electron beam deposition

# Fabrication of Ni-Mn-Ga/substrate Thin Film Composites

RF magnetron sputtering:

- Target: Hot pressing of Ni, Mn and $Mn_{30}Ga_{70}$ mixed powders

  → **nominal compositions $Ni_{49.5}Mn_{28.0}Ga_{22.5}$, $Ni_{52}Mn_{24}Ga_{24}$**
- **Substrates: $Al_2O_3$ ceramic, Mo foil (5 and 10 μm), MgO(100), [Si(100)+$SiN_x$ 500nm) wafers**

Sputtering conditions:

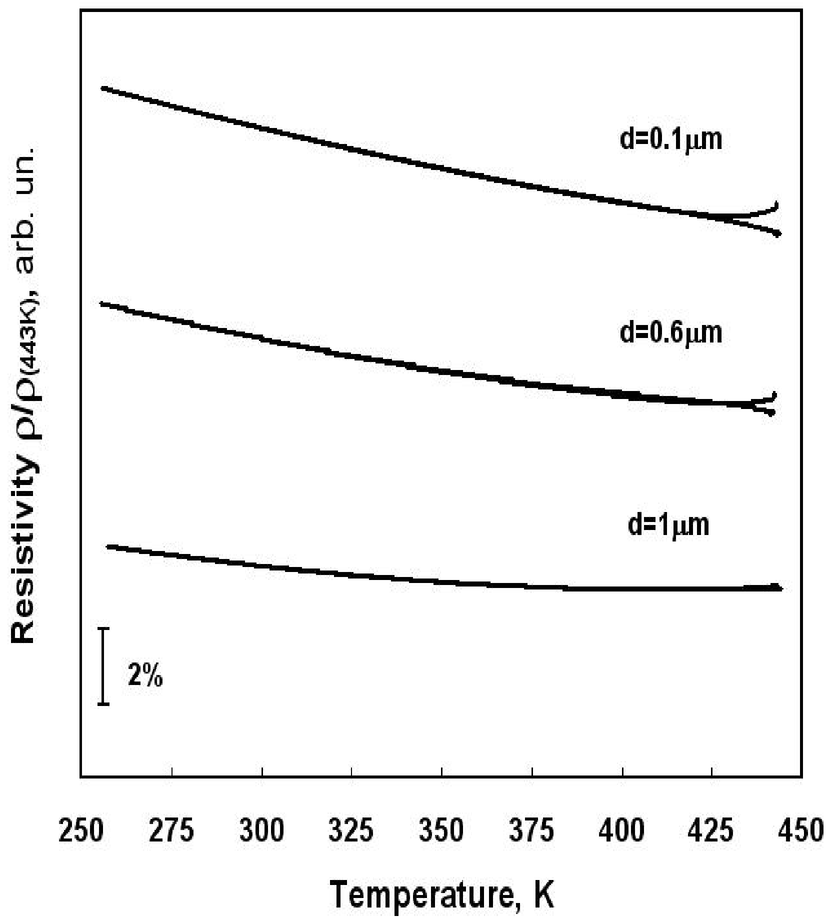
- RF power: 200 W
- Substrate temperature: 323 K
- Ar gas flow rate: 230 $mm^3s^{-1}$
- **Film thicknesses: 0.1 - 1 μm, 5 μm (reference)**

Heat treatment:

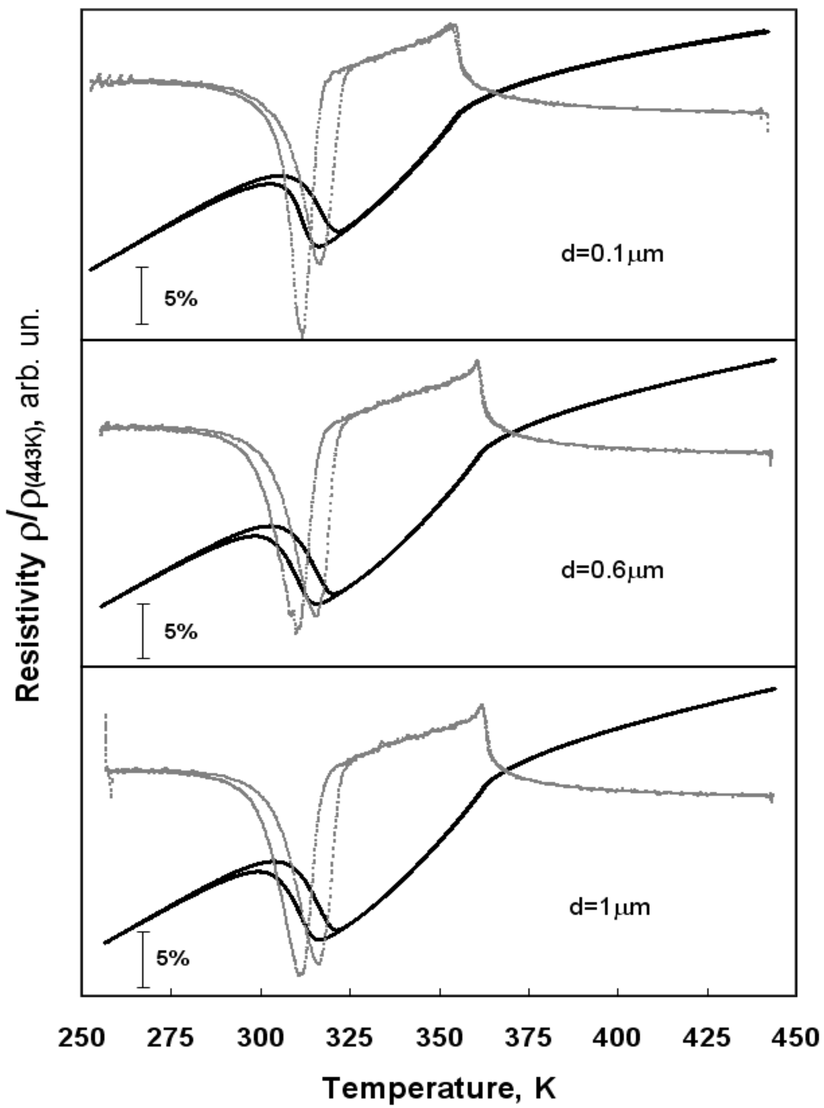
- Temperature: **1073 K**
- Time: **1h.**
- Pressure: ~ $2{\cdot}10^{-4}$ Pa

# Transformation behavior of **Ni49/MgO(100)**

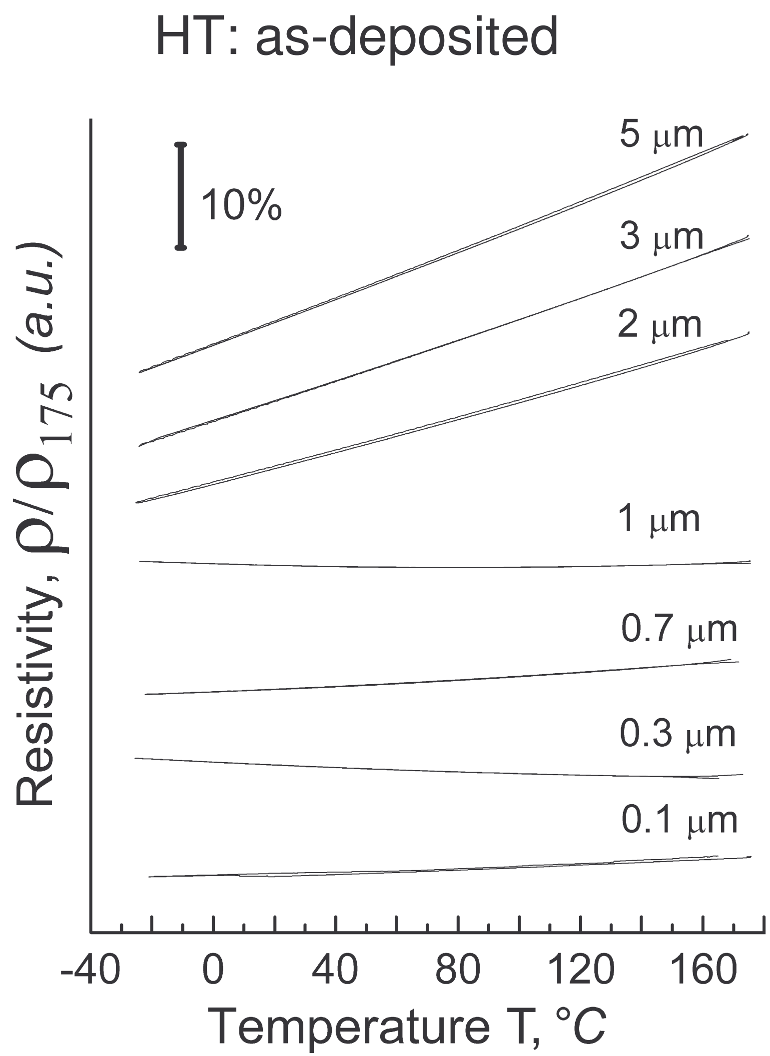
HT: as-deposited

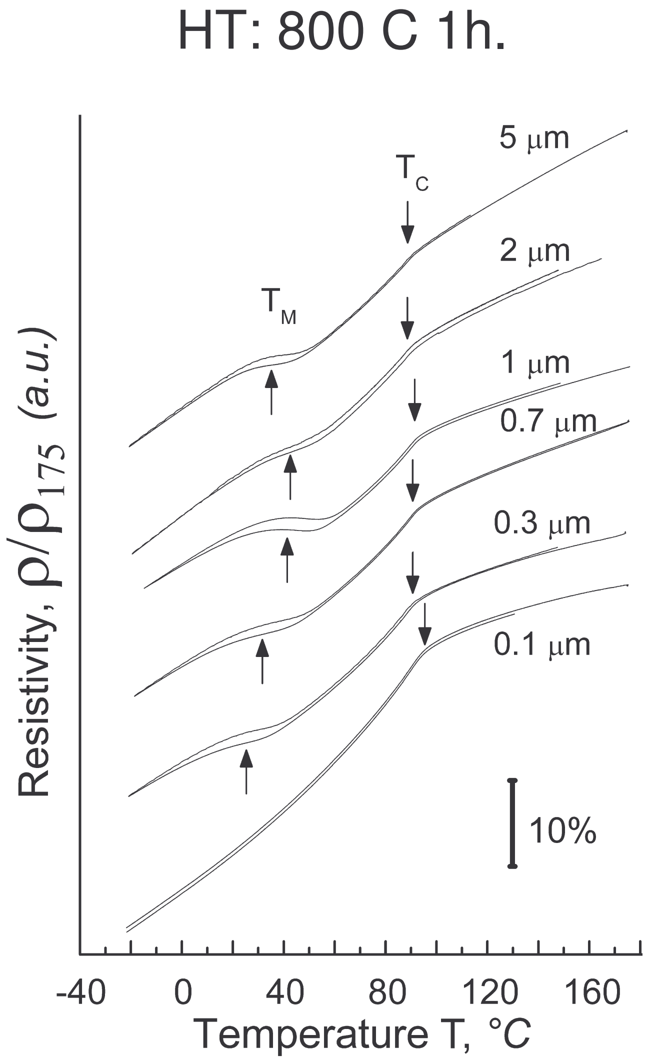



HT: 800 C 1h.



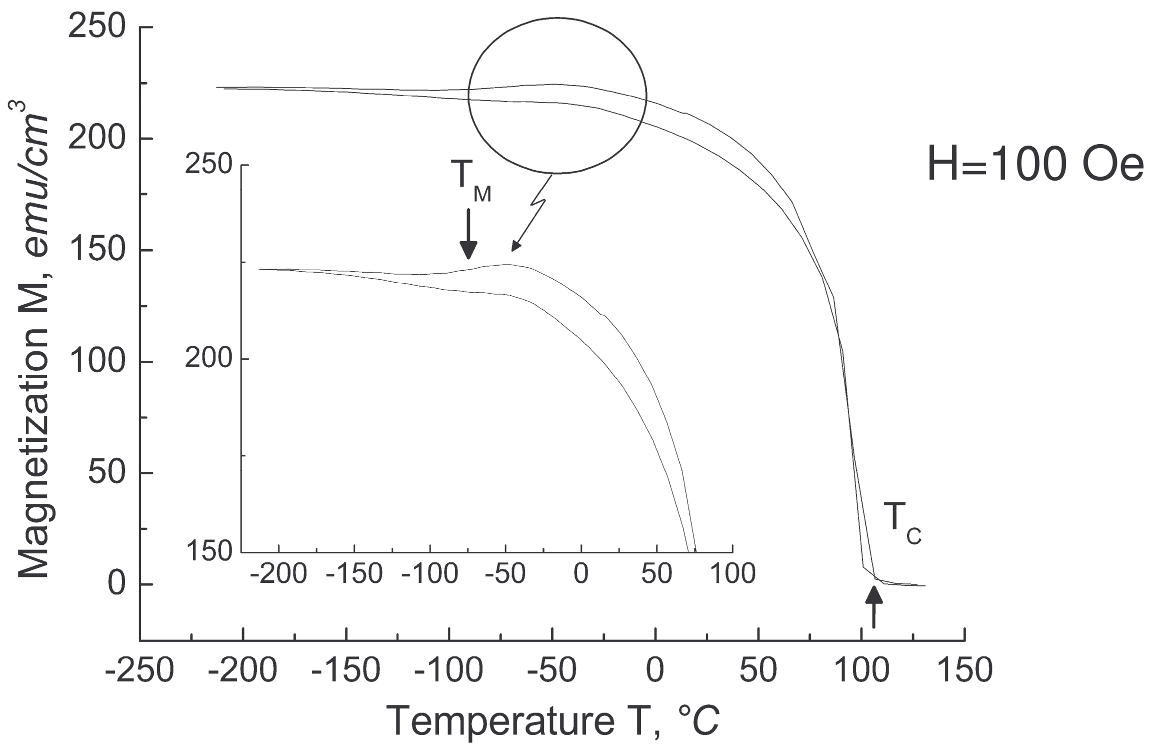
# Transformation behavior of **Ni49/Si(100)**



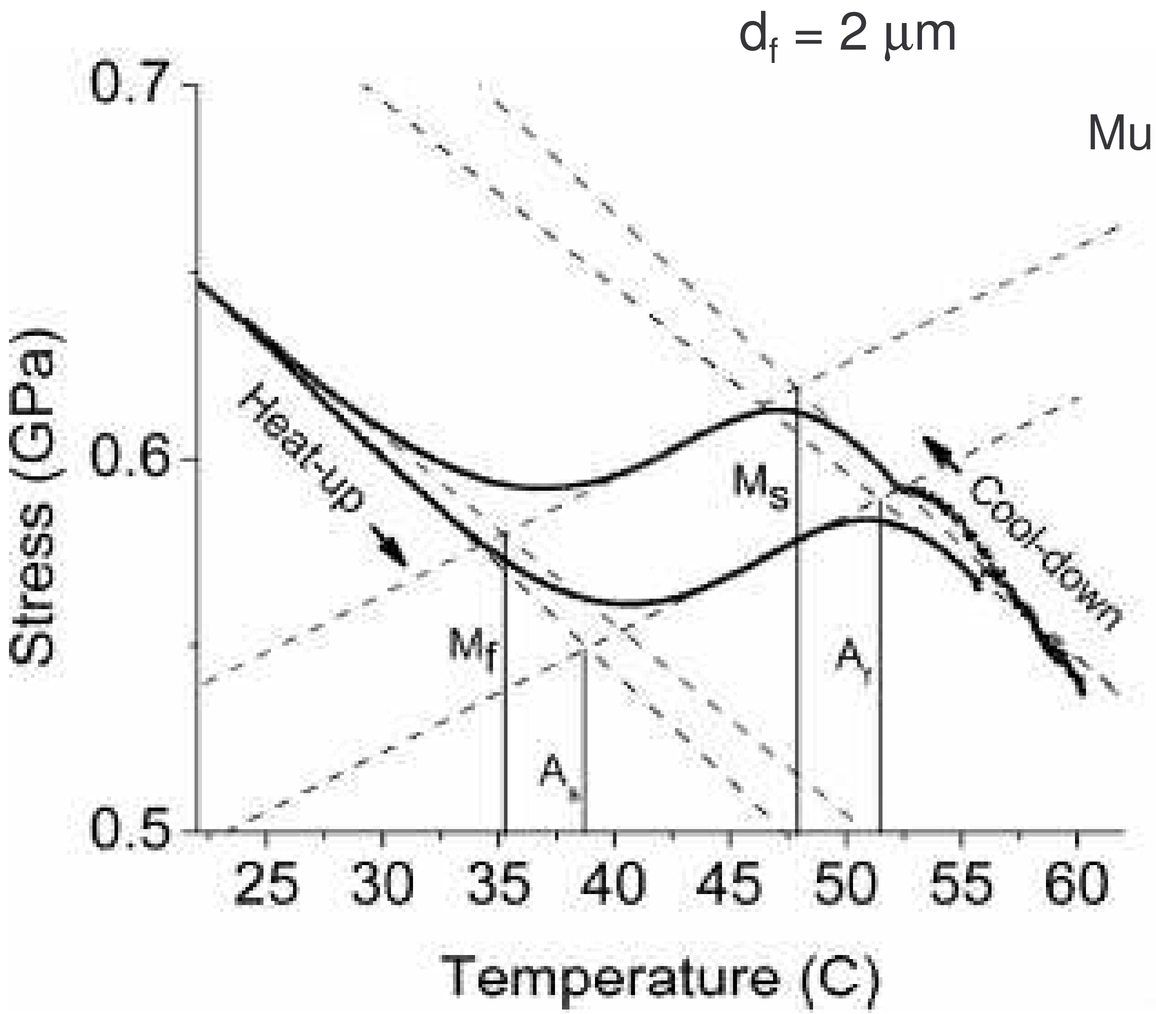



# Transformation behavior of **Ni49/Si(100)**

$d_f = 0.1\ \mu m$



# Substrate curvature analysis for Ni49/Si



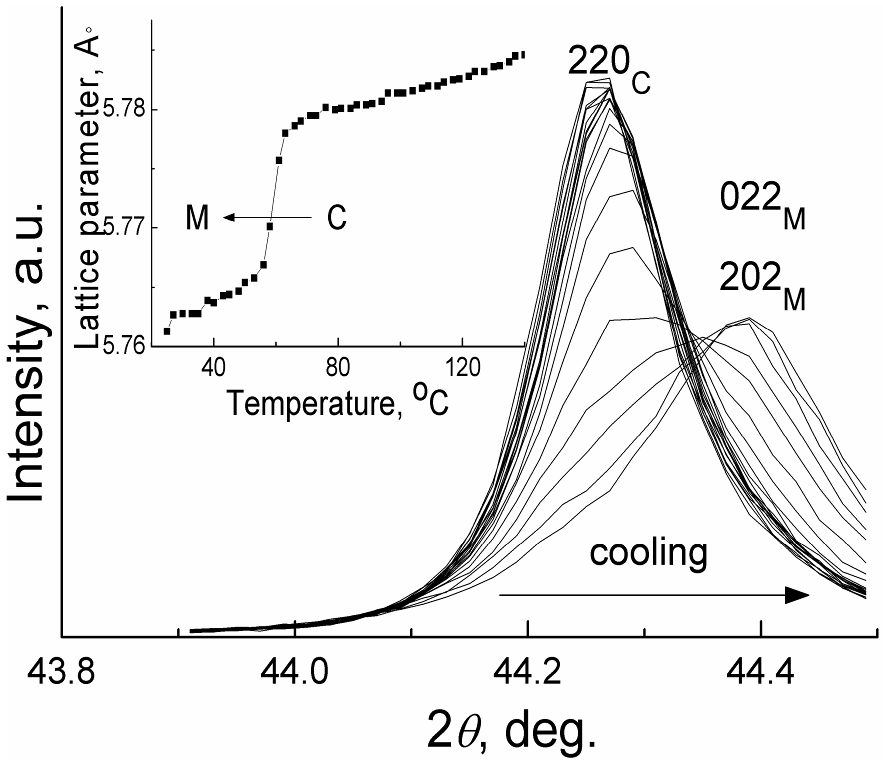

Multi-beam Optical Sensor

$$\kappa - \kappa_0 = \frac{6\sigma_f h_f}{M_s h_s^2}$$

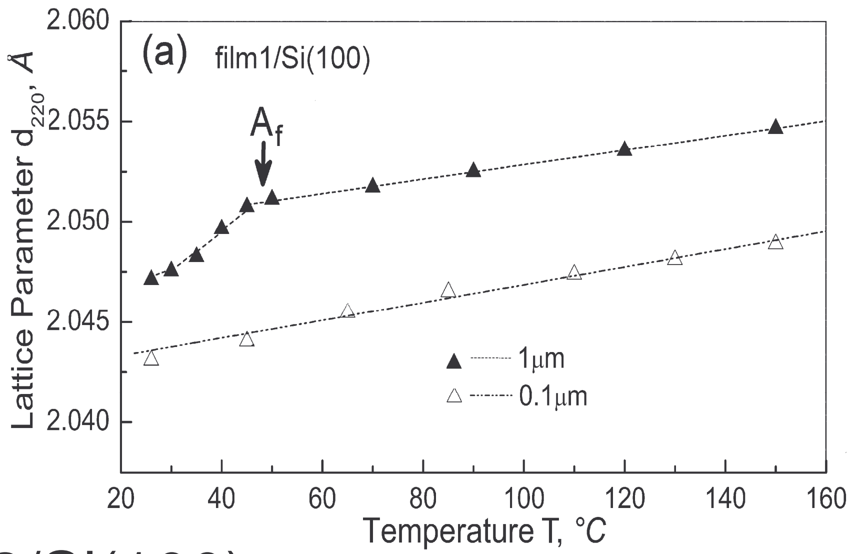
$$\Delta\sigma_f = M_f \Delta\alpha \Delta T$$

# Transformation behavior in XRPD

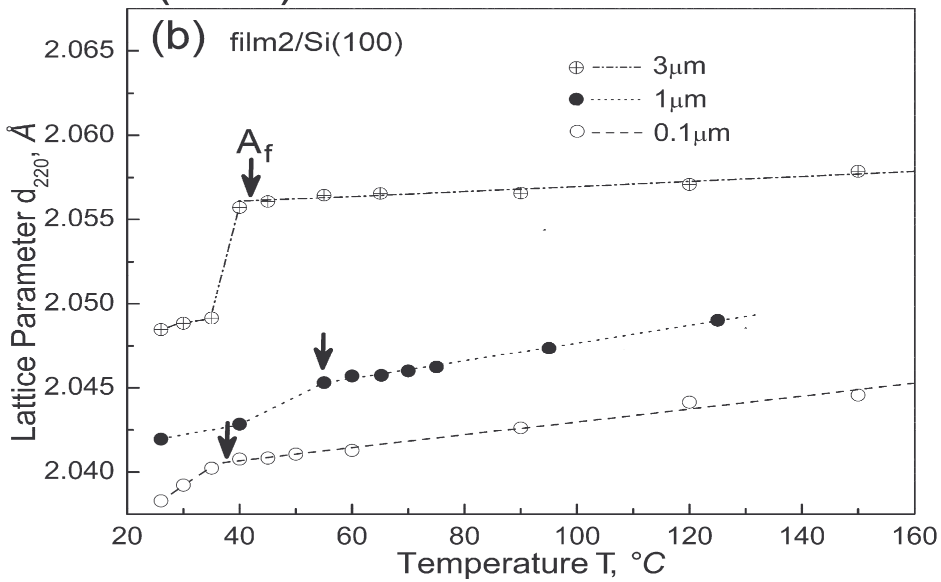
## Ni52/Mo10, $d_f$ = 1 μm




## Ni49/Si(100)

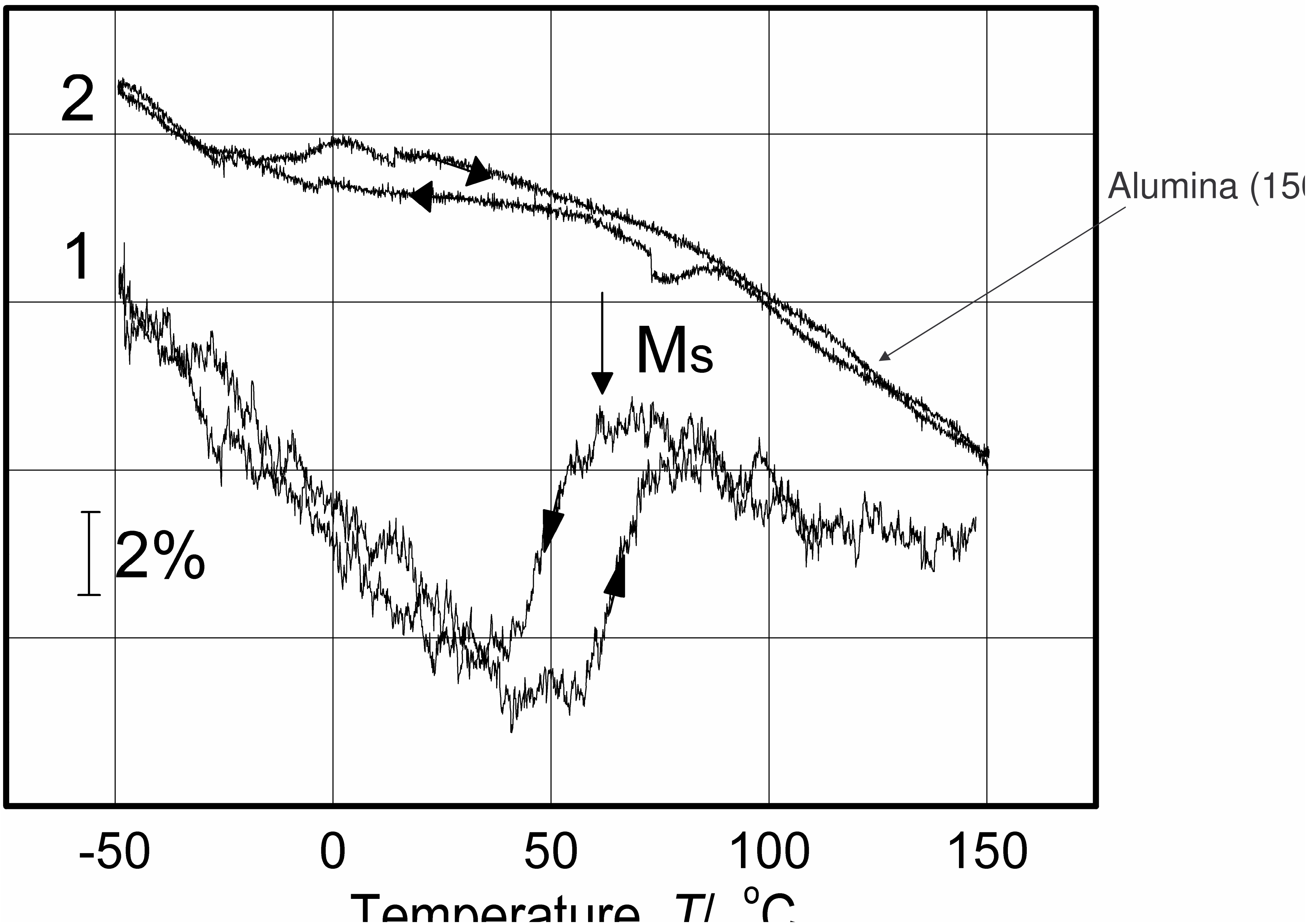



## Ni52/Si(100)



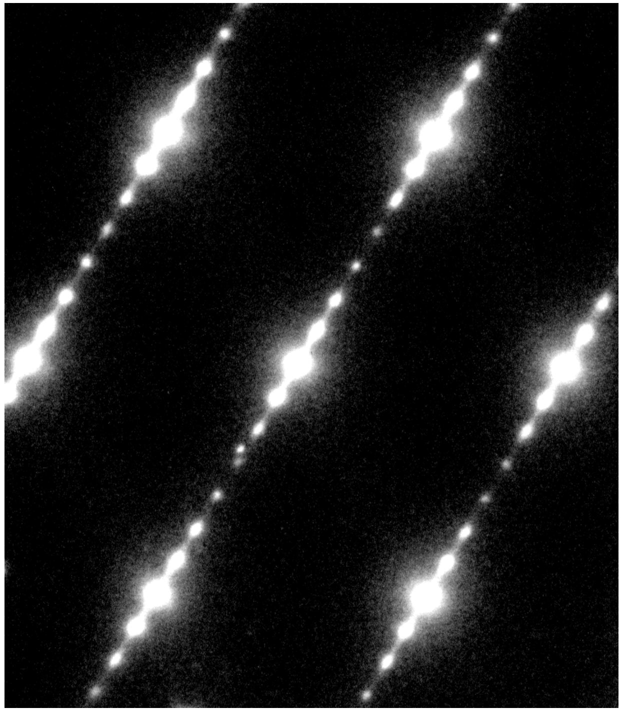
Transformation behavior studied by DMA

Ni49(0.6μm)/alumina(150μm



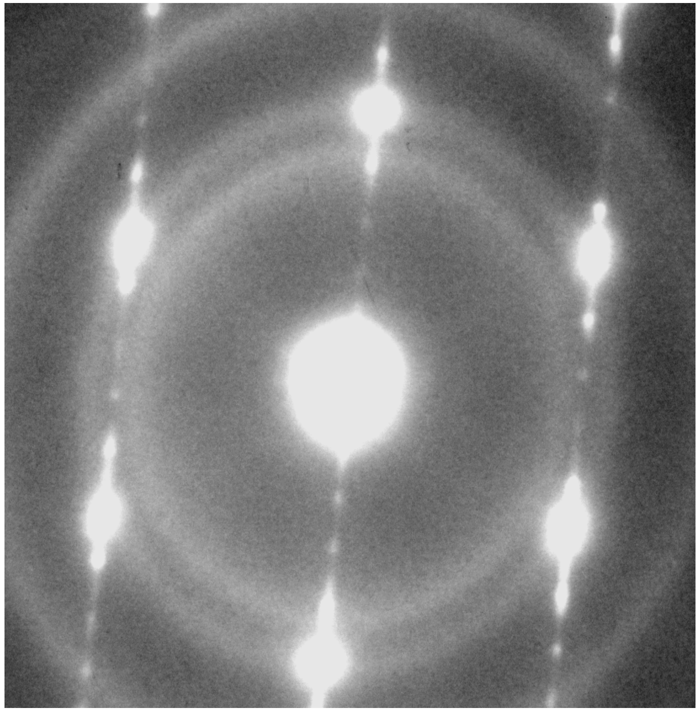
## Structural Properties

**Crystal structure by TEM of 5 μm free films**



**52 at.% Ni**, $T_{HT}$:1073 K (3.6 ks)

**14M-martensite**

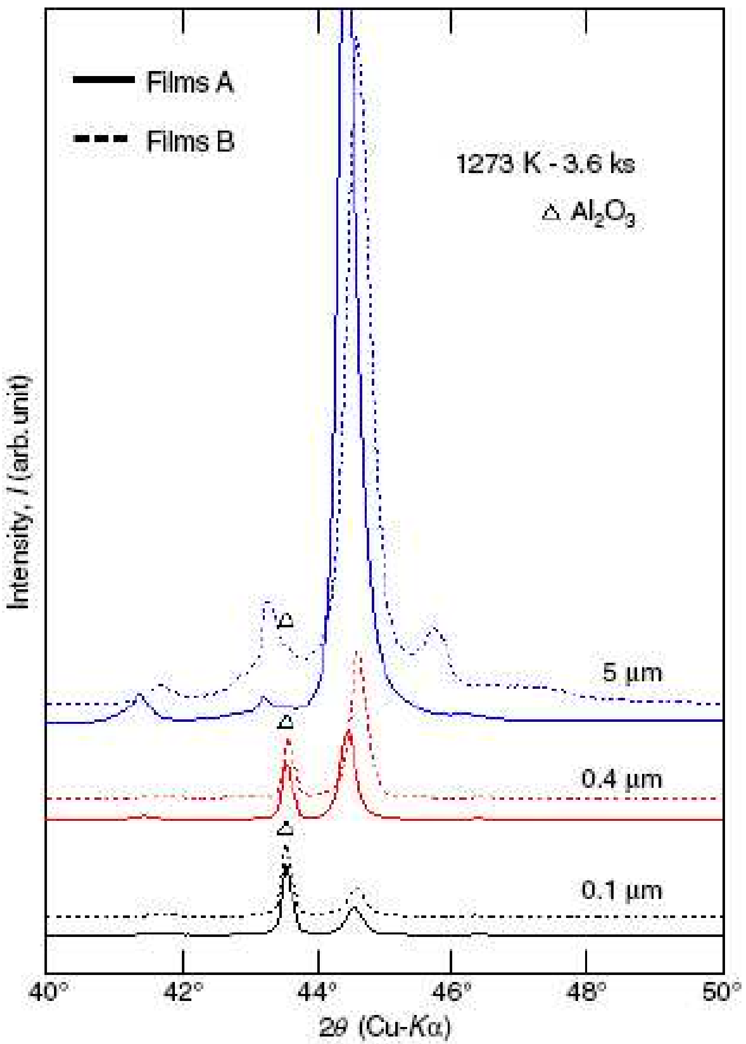


**49.5 at.% Ni**, $T_{HT}$:1073 K (3.6 ks)

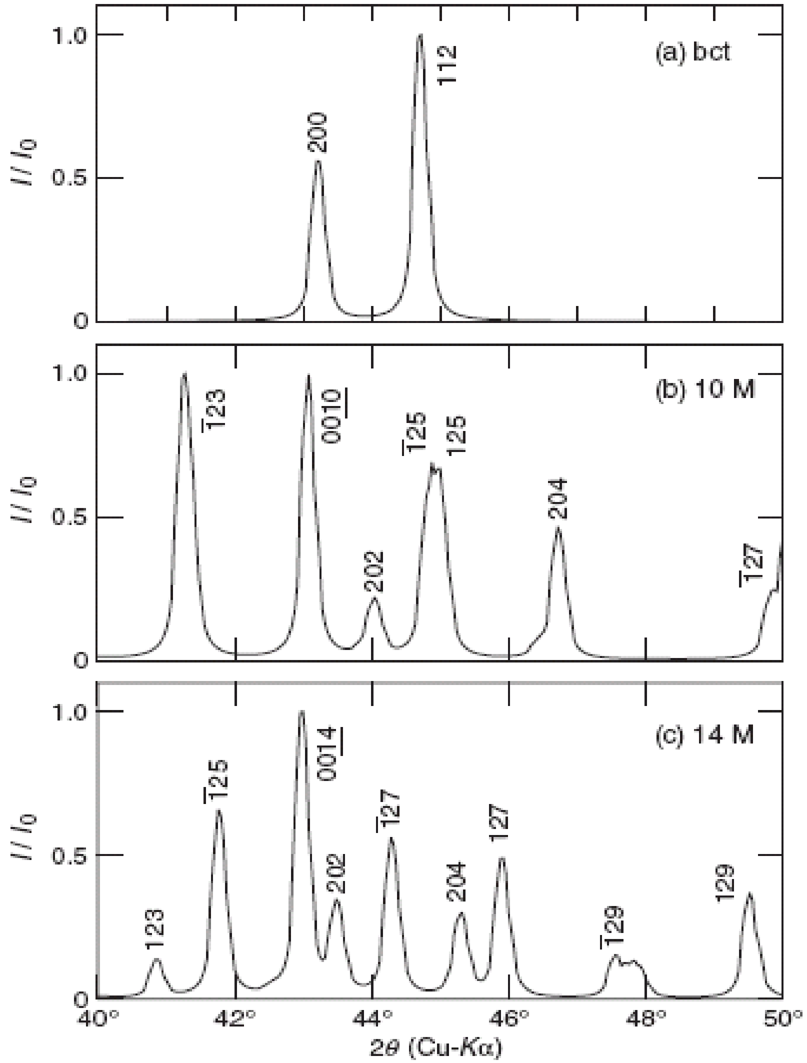
**10M-martensite**

# XRPD

## Ni49,52/alumina

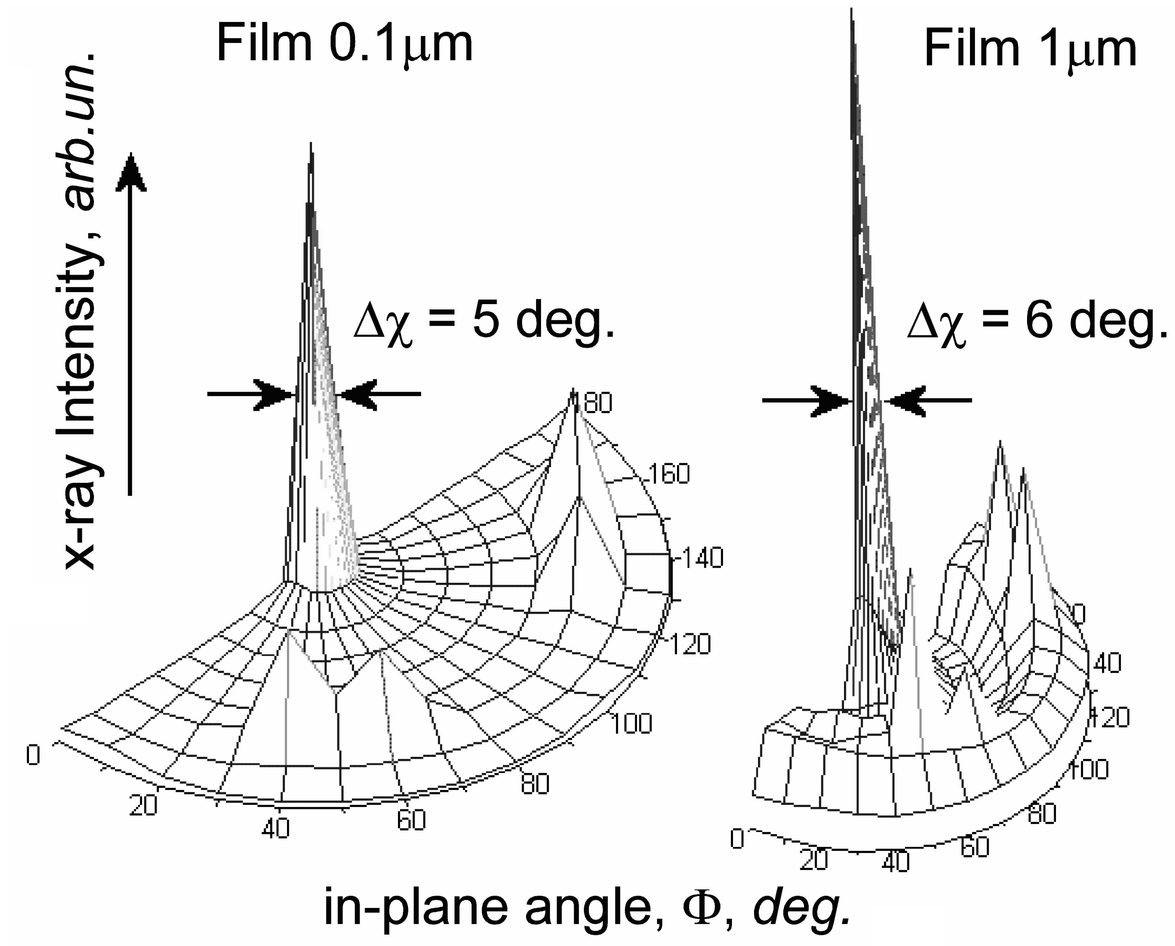



## Calculated



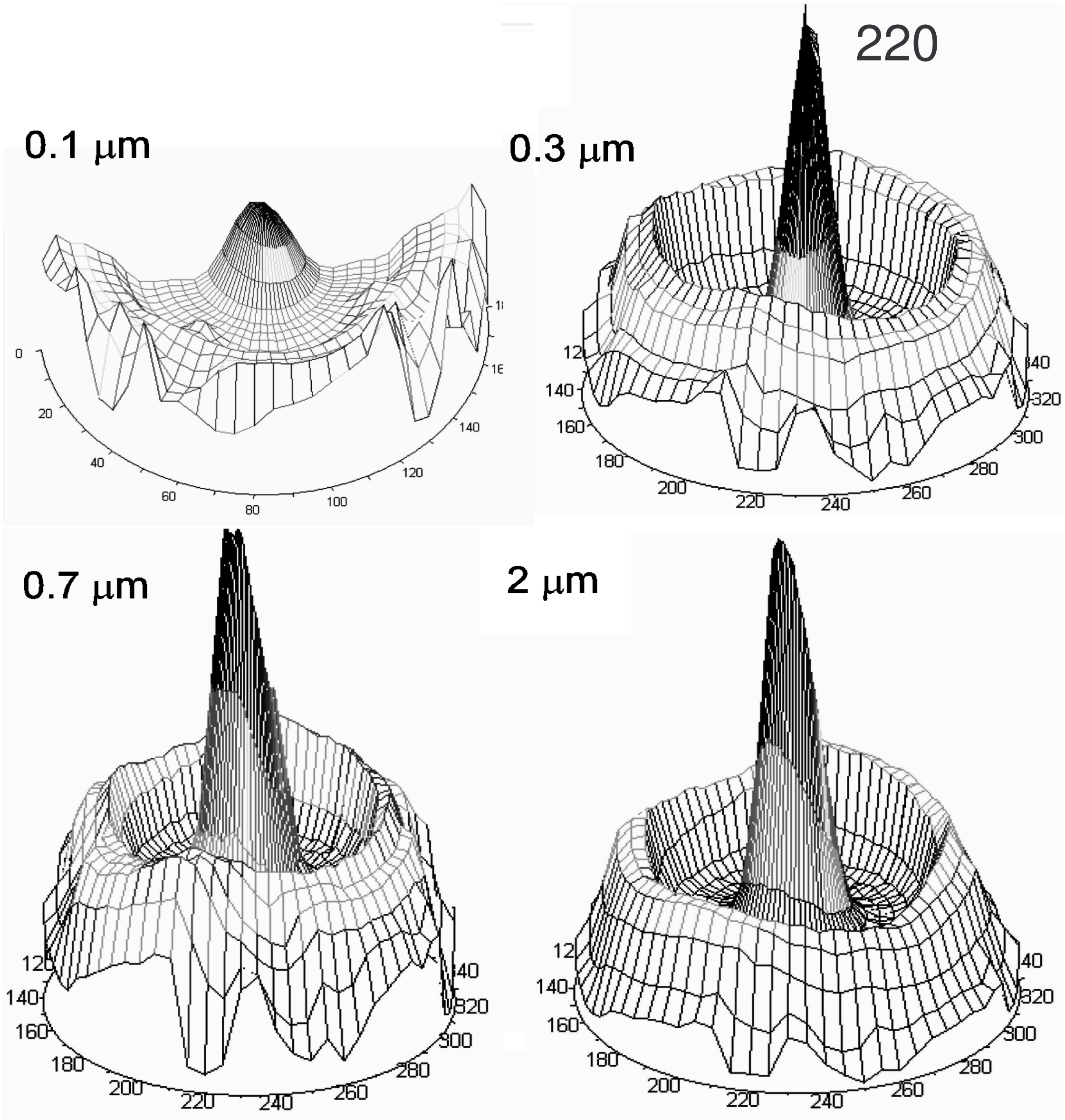

Chernenko et al. ,SMS 2005

# Texture studies **by** X-ray beamline of ANKA synchrotron source (FZK, Karlsruhe)

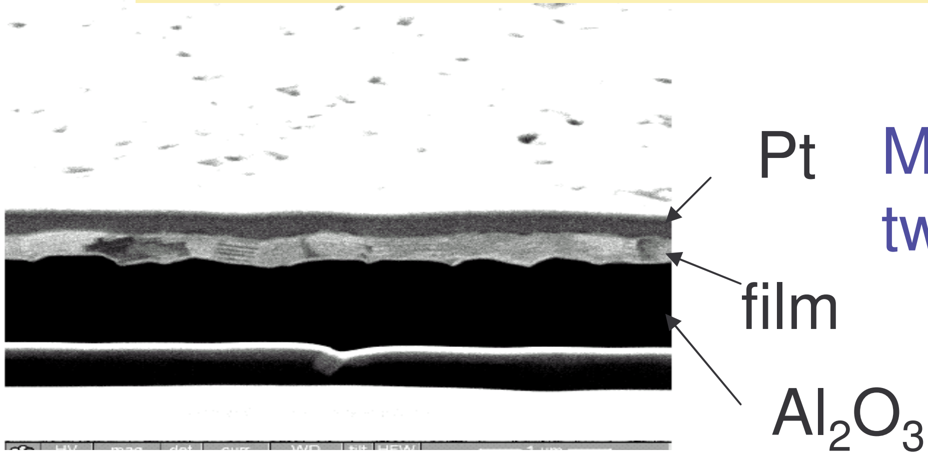



**Ni49/MgO(100)**

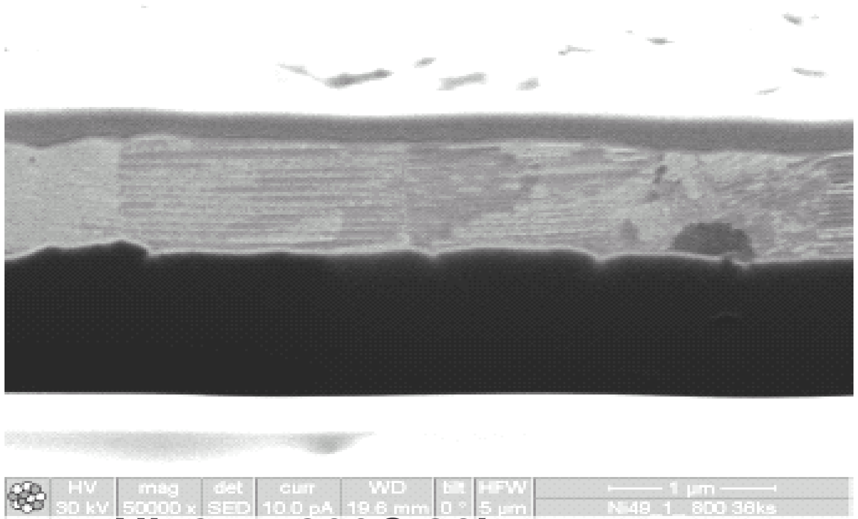
# Fiber texture Ni49/Si(100)



# FIB images of cross-sectional areas of Ni49/$Al_2O_3$ film composites

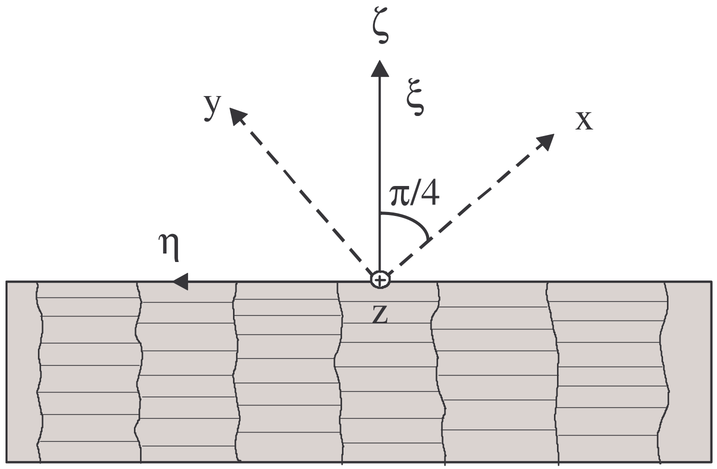



**Ni49_04_800C 36ks**



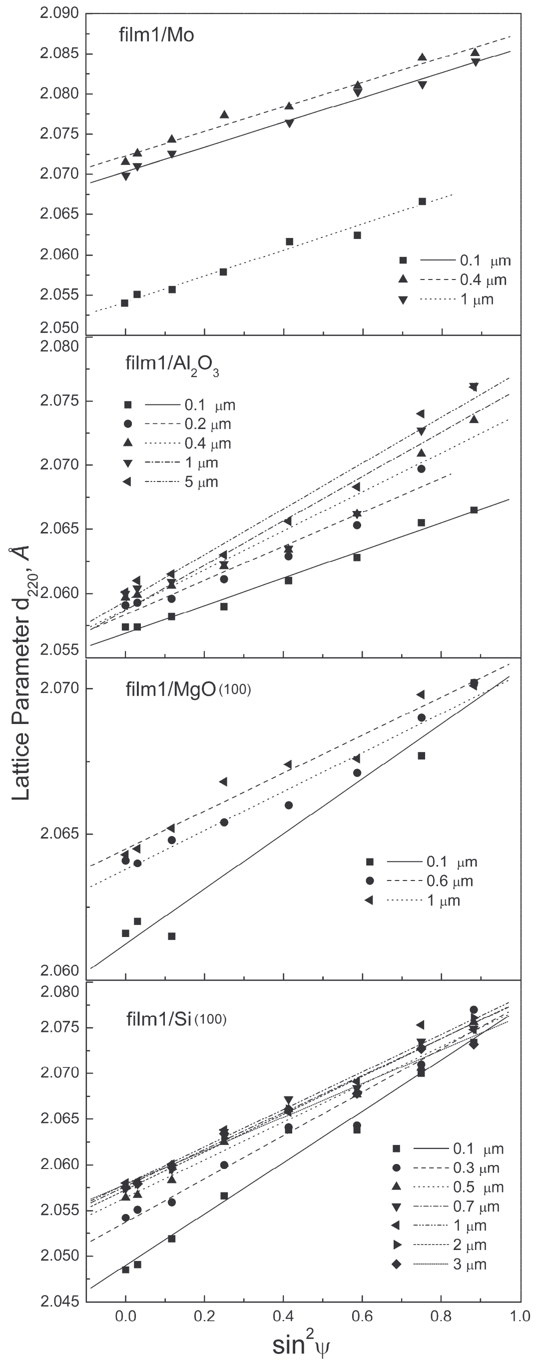
**Ni49_1_800C 36ks**

Model of columnar structure of twinned martensite




Chernenko et al., Mat.Trans.2006

## Ni49/substrate



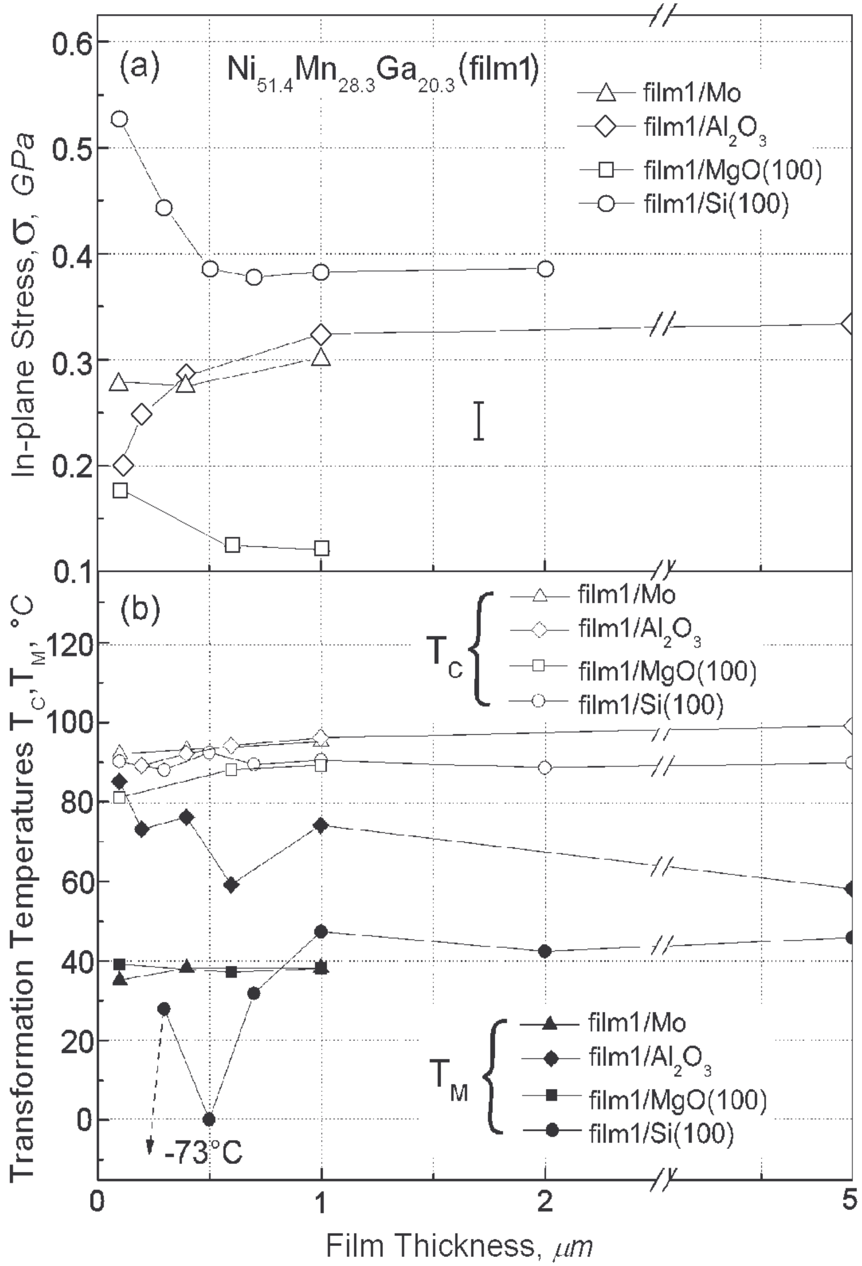

# X-ray stress analysis

$$\sigma = \left(\frac{E}{1+\nu}\right)\frac{1}{d_o}\left(\frac{\partial d_{220}}{\partial \sin^2\psi}\right)$$

$E = 50$ GPa, $\nu = 0.3$

EMRS2007

# Correlation: in-plane film stress and transformation temperatures of Ni49/substrate



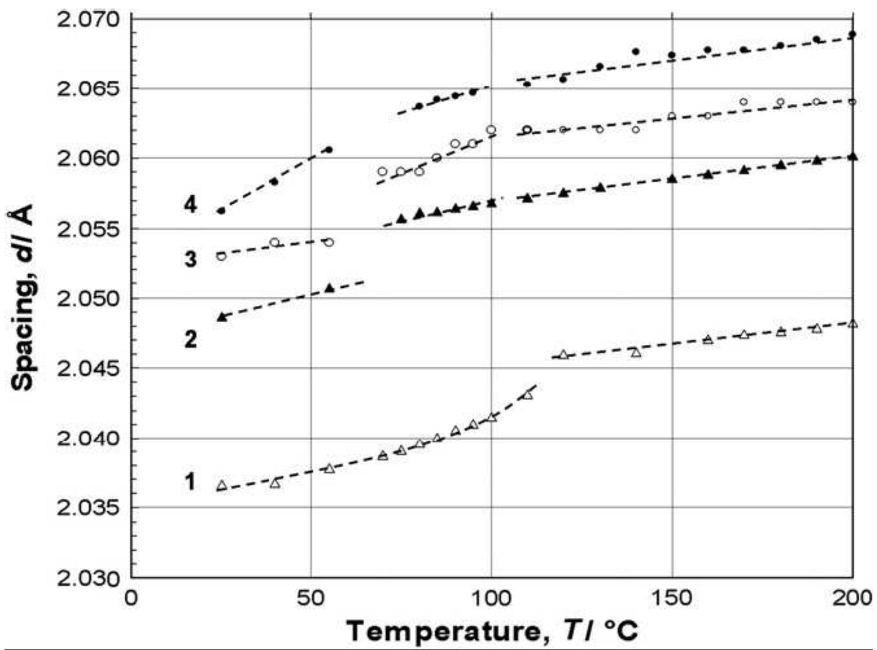

$\sigma = E(\Delta\alpha\Delta T)$ , $\Delta T$ = 650 K

where $\alpha_f$=15, $\alpha_s$=**12.5**; 7.5; 5 and 3x$10^{-6}$ $K^{-1}$

| MgO | alumina | Mo | Si |
|---|---|---|---|
| σ = 0.18 | 0.35 | 0.46 | 0.56 GPa |

EMRS 2007

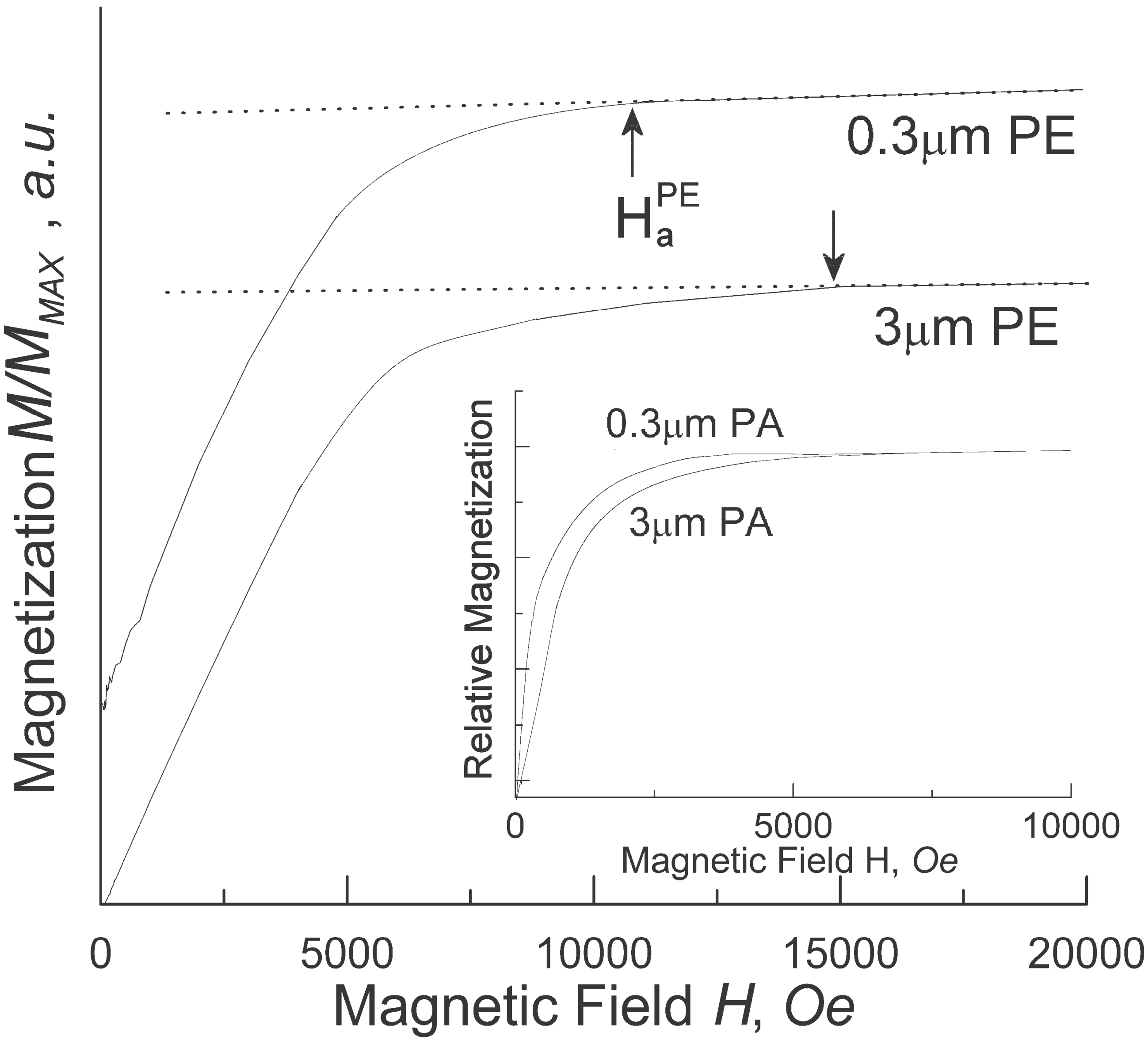
# Thickness dependence of out-of-plane elastic modulus



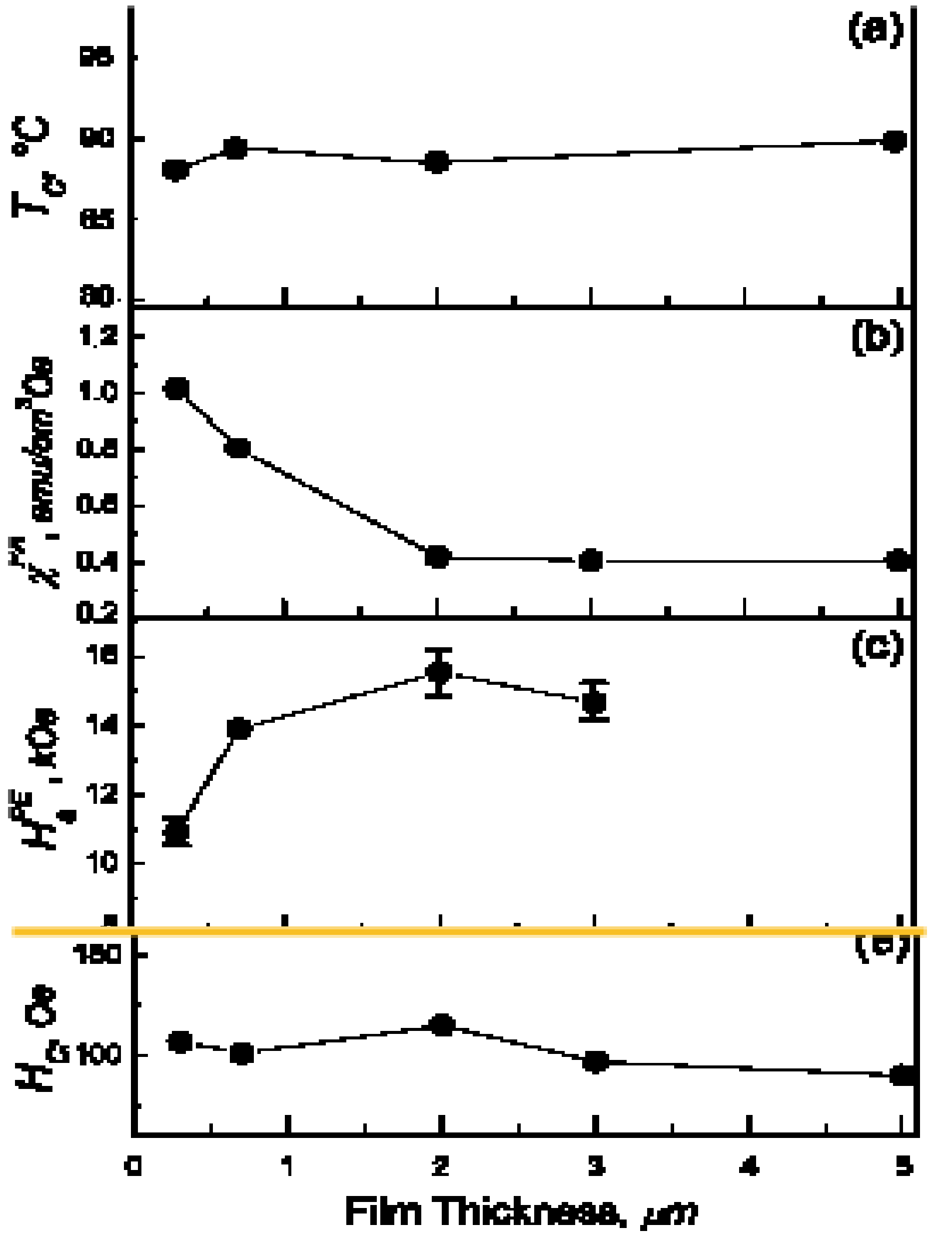

Transformation temperature $A_f$ and $d$ spacing measured at 200 °C for $Ni_{51.4}Mn_{28.3}Ga_{20.3}$ films of different thickness $h$ deposited on alumina ceramic substrate

| $h$ (μm) | $A_f$ (C) | $\Delta T$ (C) | $\Delta\sigma$ (MPa) | $d$ (nm) | $\Delta\varepsilon$ | $E$ (GPa) |
|---|---|---|---|---|---|---|
| 5 | 65 | 0 | 0 | 0.206891 | 0 | – |
| 0.6 | 64 | 1 | ∼0 | 0.206400 | 0.00237 | – |
| 1 | 78 | 13 | 130 | 0.206022 | 0.00426 | 31 |
| 0.1 | 114 | 49 | 490 | 0.204826 | 0.00998 | 49 |

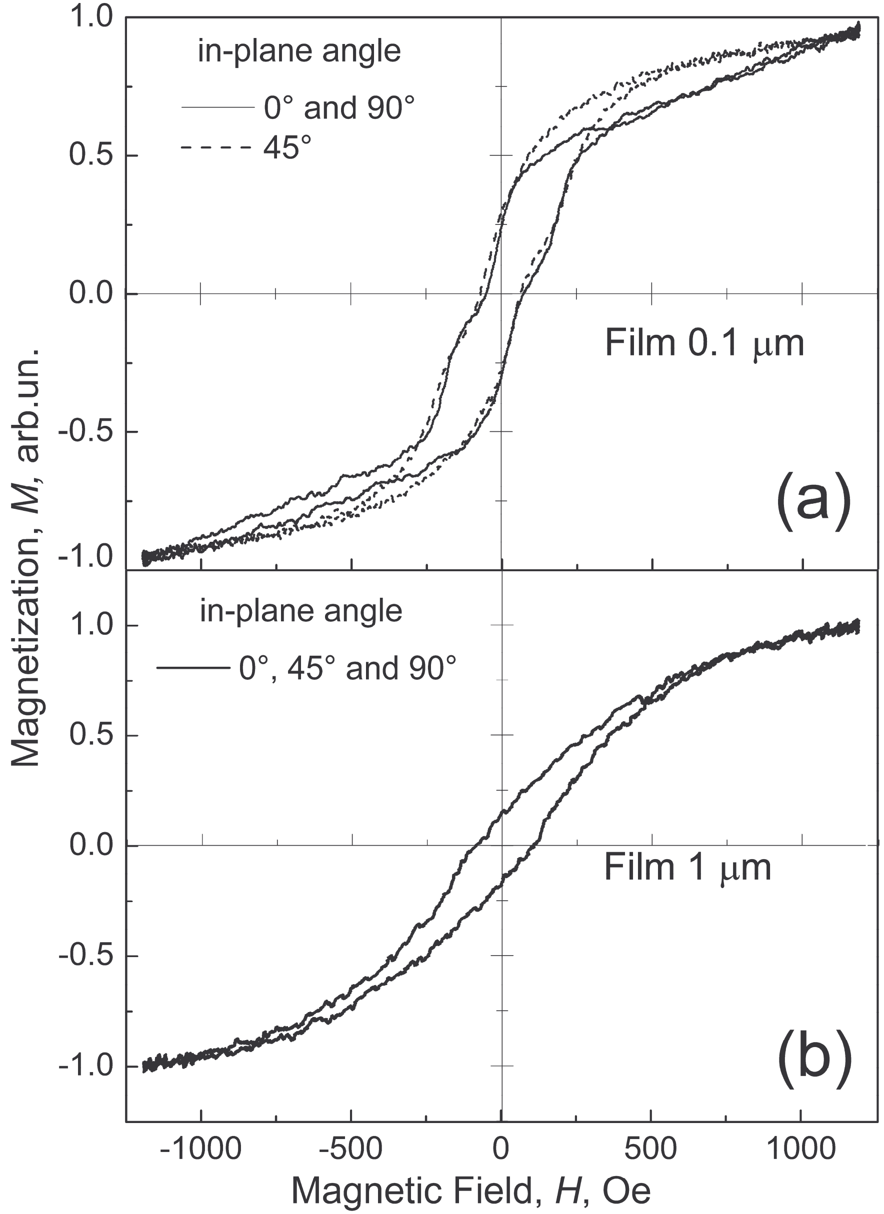
# Magnetization of Ni49/Si(100) composite



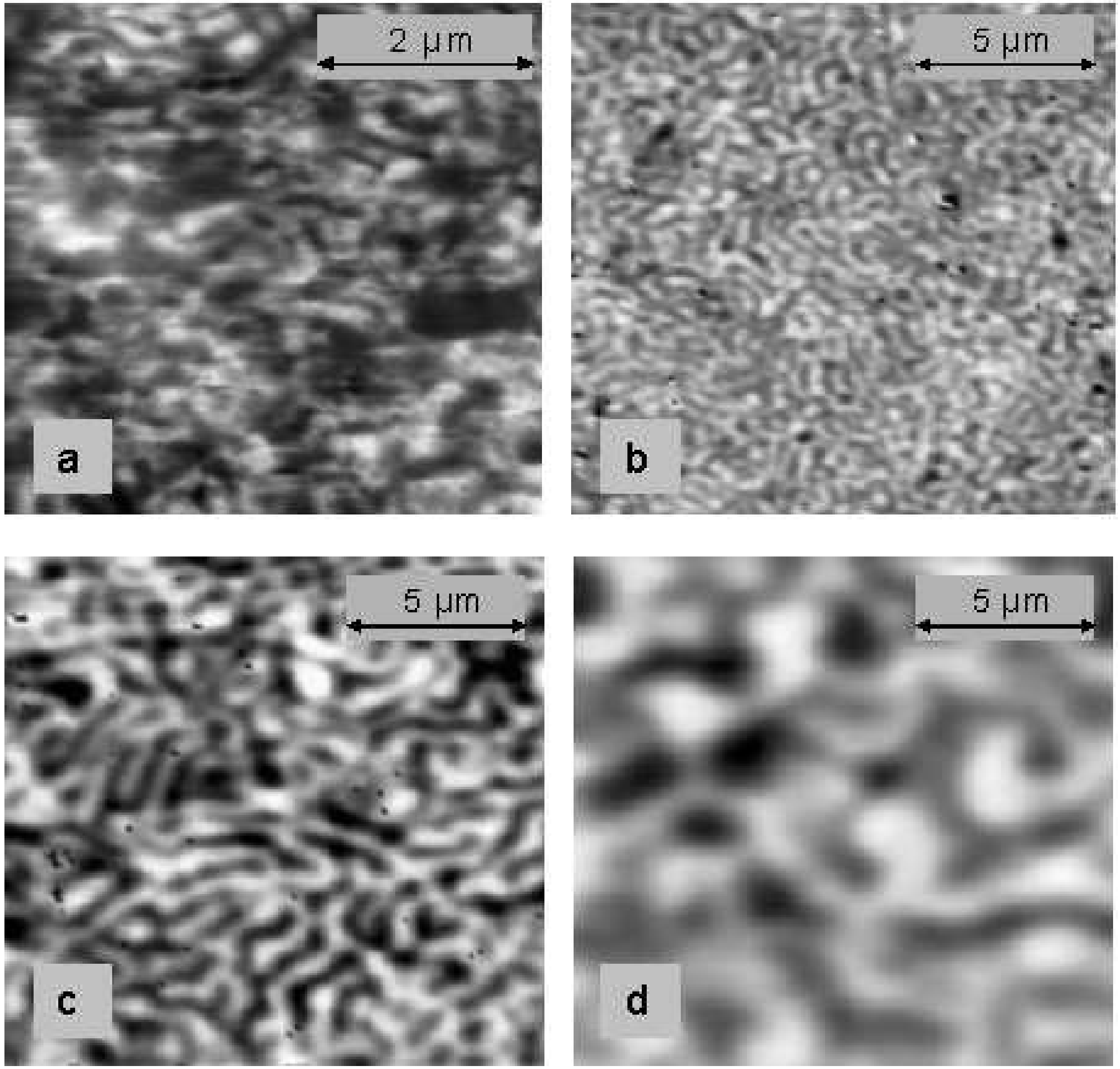
# Thickness dependence of magnetic parameters for Ni49/Si



# In-plane magnetic anisotropy for 0.1 μm NiMnGa/MgO film



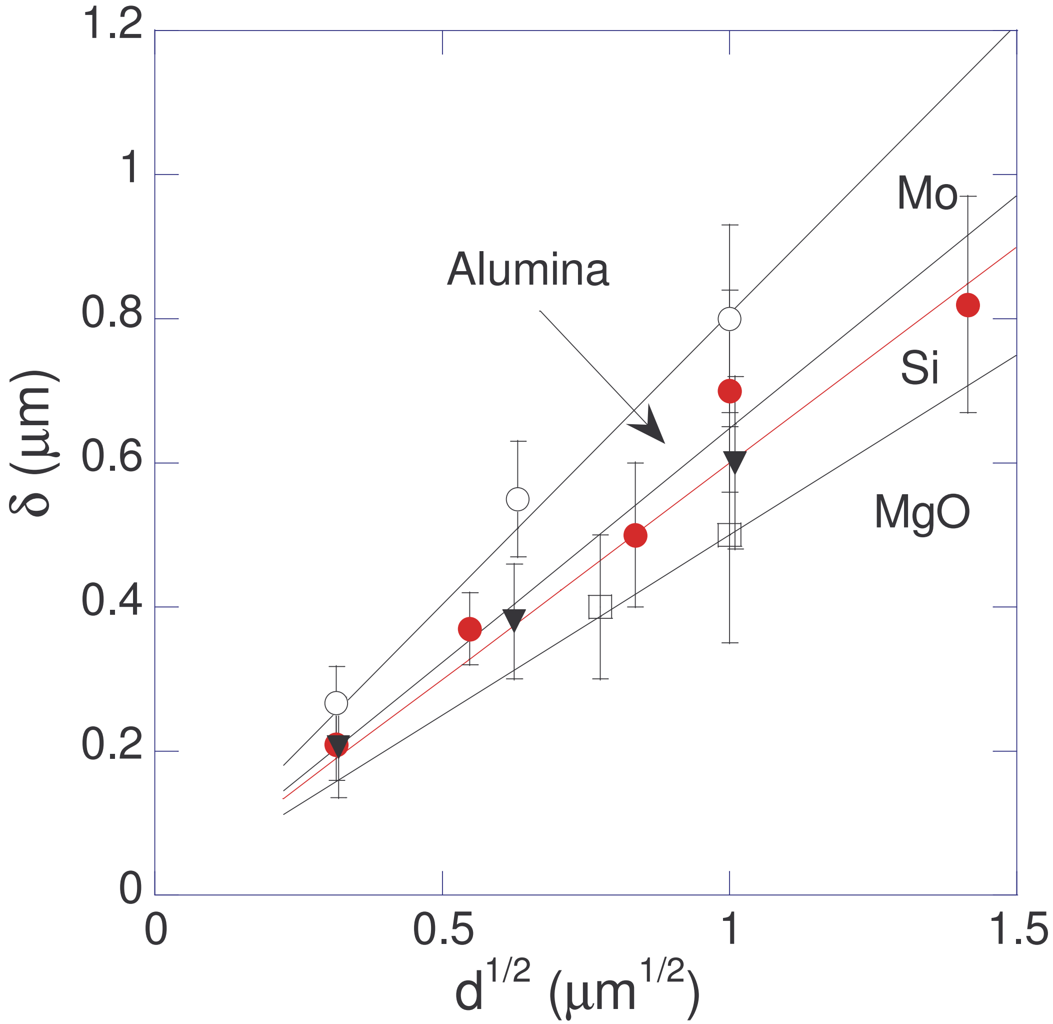
# Magnetic Force Microscopy, H=0




**$Ni_{49.5}Mn_{28.0}Ga_{22.5}/Al_2O_3$**; *d* = 0.1 μm **(a),**
0.4 μm **(b);** 1.0 μm **(c);** 5.0 μm **(d)**

Chernenko et al.,JPCM2005

## Magnetic domain size: influence of substrate nature and microstructure




$$\delta = \sqrt{d\delta_{dw}}$$

$$\delta_{dw} = a\sqrt{E / A}$$

**d<0.4 µm: Domain size becomes larger than film thickness**

Analysis of $\chi$ vs angle between H and film normal and determination of :

(a) angle between magnetization and film normal 67 deg

(b) Q=2.9

for Ni49/alumina 0.1 $\mu$m thick film

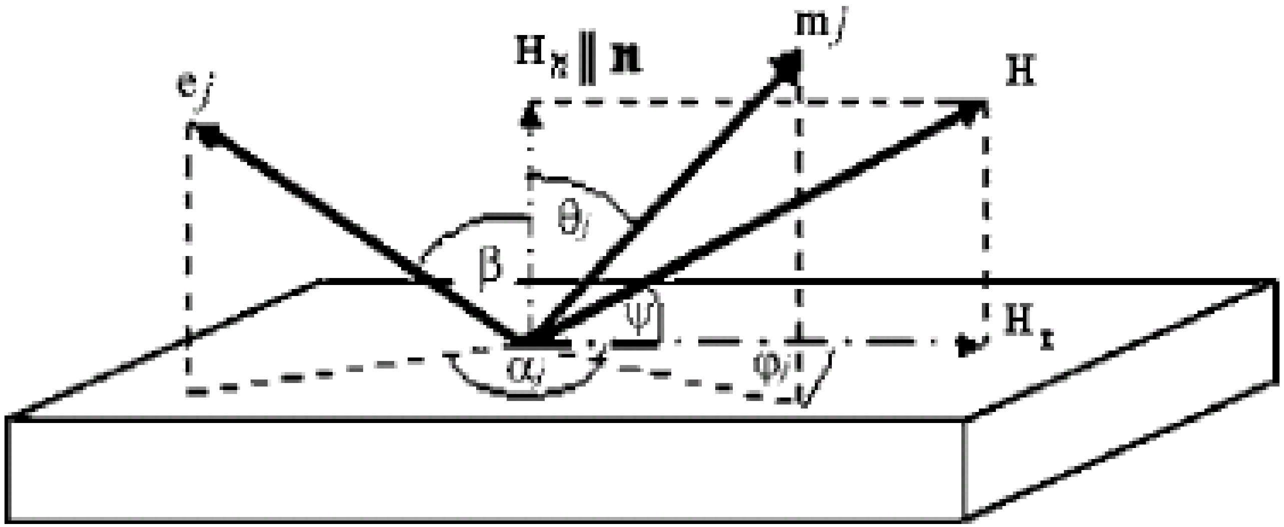
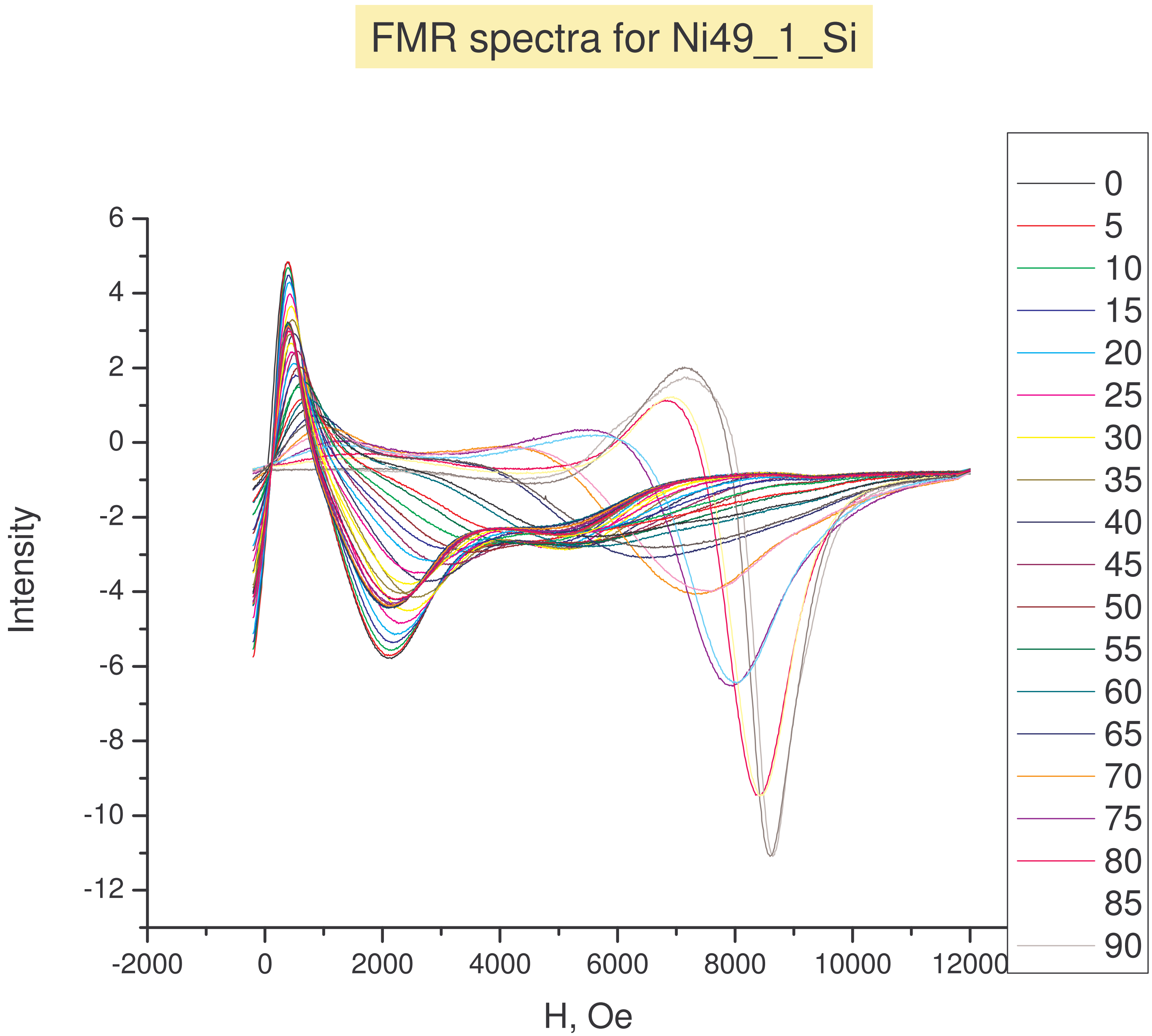



FIG. 3. The angles prescribing the directions of the external magnetic field $\psi$, easy-axis direction $\beta$, and magnetic vector position $\theta$ with respect to film normal or film plane.

$\chi(0^o)/\chi(90^o)>1$

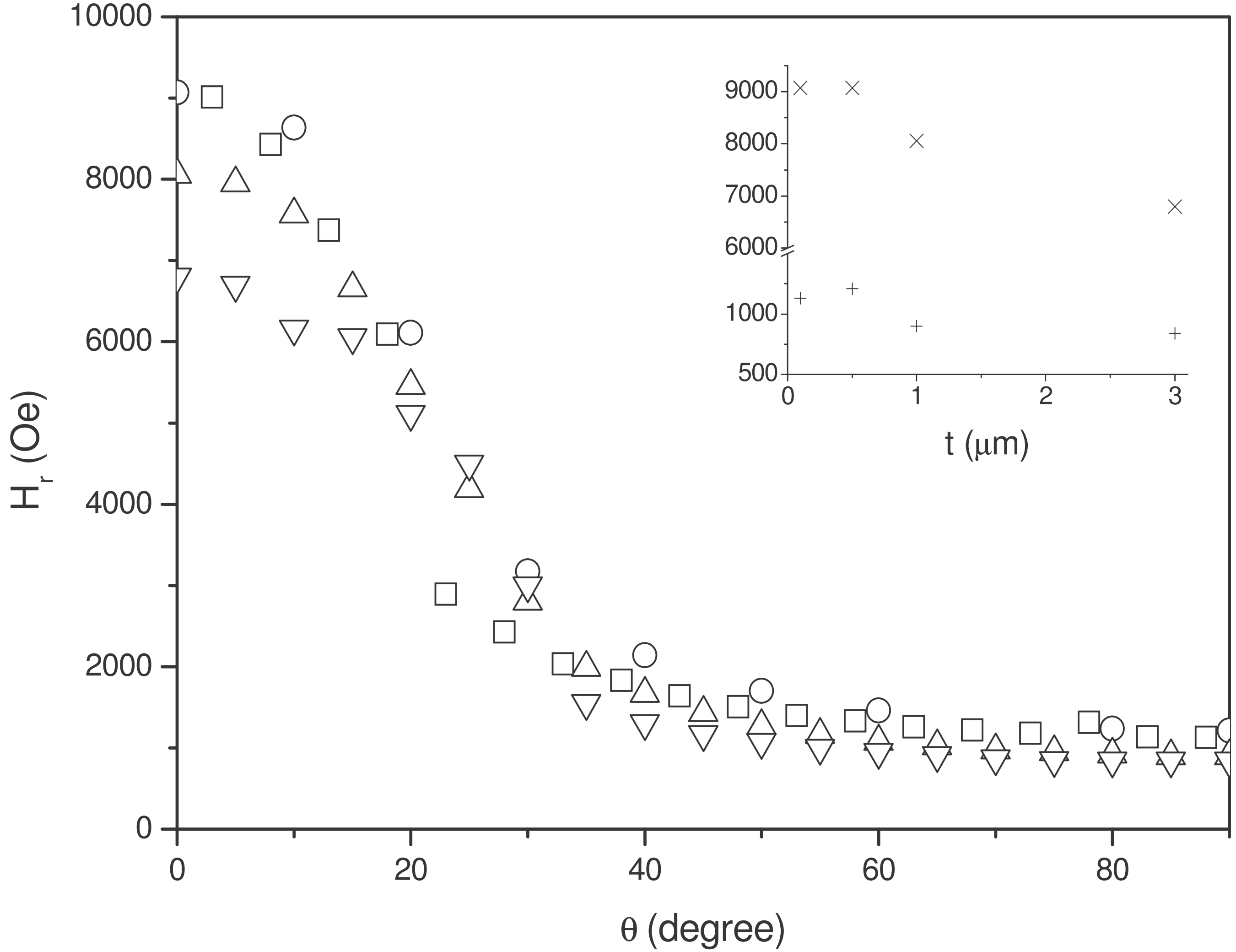
$\theta>45^o$

Chernenko, Lvov et al.,JAP,**101**,2007

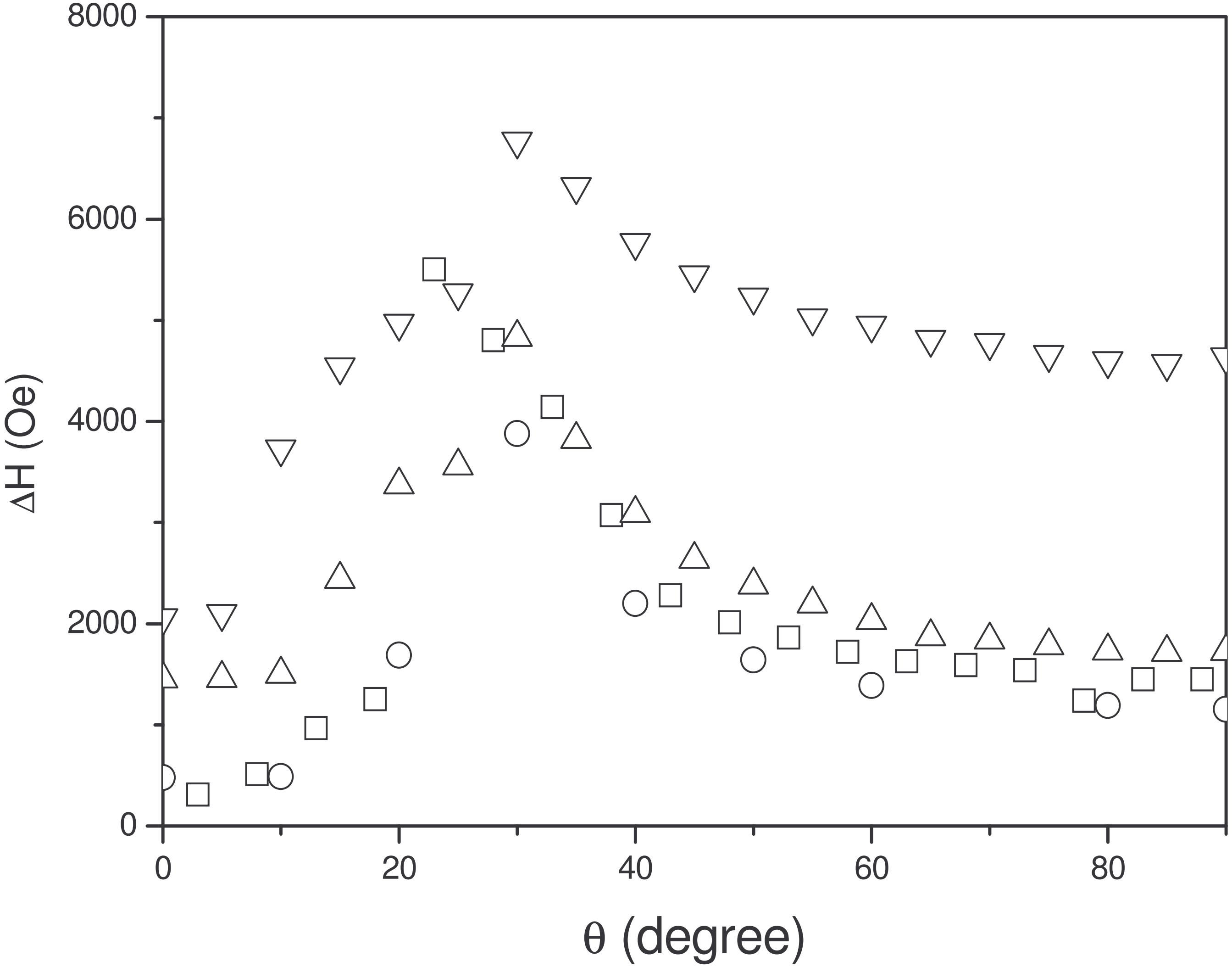

FMR spectra for Ni49_1_Si
Intensity
H, Oe
6
4
2
0
-2
-4
-6
-8
-10
-12
-2000
0
2000
4000
6000
8000
10000
12000
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90

# Resonance field vs angle between film normal and H

## Inset: resonance field vs film thickness



# Resonance linewidth vs angle



Modeling:

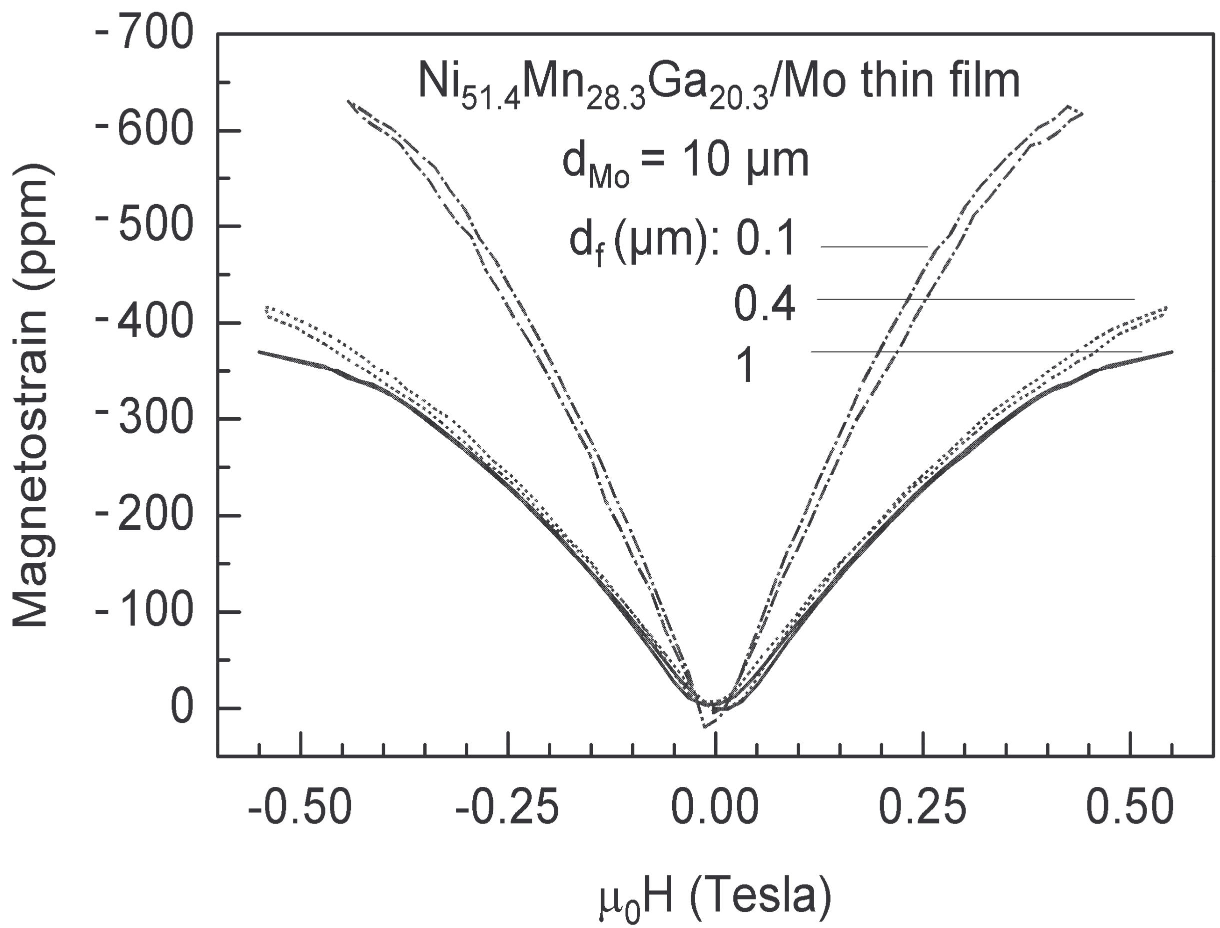
using $M_{sat}$=500 emu/$cm^3$,
g=2
uniaxial anisotropy making an
angle 45 degree with respect to film normal

↓

values of uniaxial anisotropy constant for films Ni49/Si

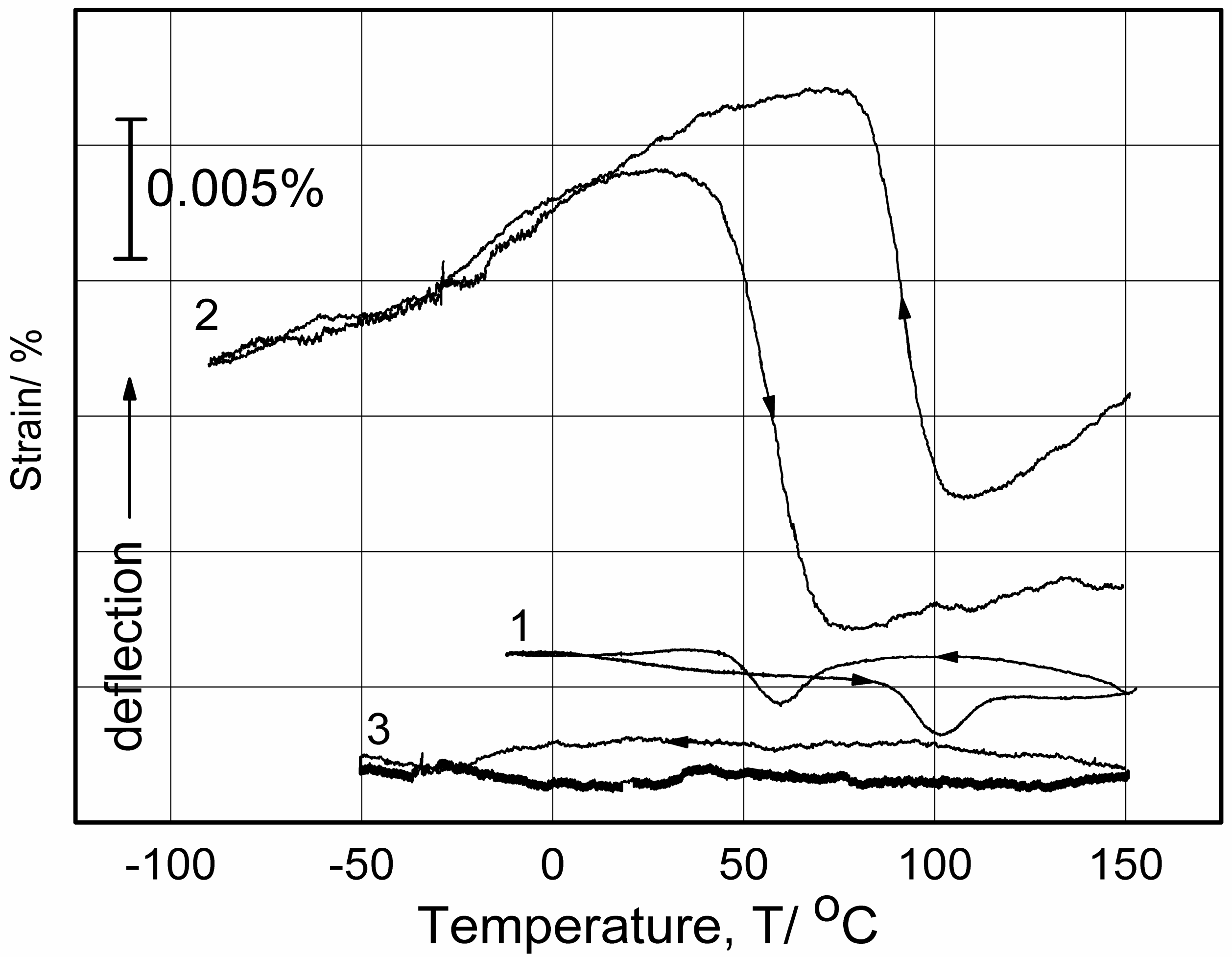
| 0.1μm | 0.5μm | 1μm | 3 μm |
|---|---|---|---|
| $2.8 \cdot 10^5$ erg/$cm^3$ | $2.8 \cdot 10^5$ erg/$cm^3$ | $4.2 \cdot 10^5$ erg/$cm^3$ | $5.1 \cdot 10^5$ erg/$cm^3$ |

# Magnetic activation of Ni49/Mo10



## Thermally activated cantilever Ni49(5μm)/alumina(150μm) actuator

1 - no external load is applied;
2 - cantilever is loaded by the external stress of 150 MPa.
3 - alumina substrate cantilever loaded by the stress of 150 MPa.



# Work in progress on FSMA's/substrate thin films composites

TEM and MFM imaging of films cross-section

X-rays grazing incidence studies

Deposition of films on heated substrates: epitaxial growth

Measurements of elastic modules as a function of film thickness

**Modeling**

Sensing and actuation properties

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Institute of Magnetism

# *Ferromagnetic Shape Memory Alloys: underlying physics and recent advances in thin film technology*


## V. A. Chernenko

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Present address:

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## Milestones/Breakthroughs in MSMA history

| Period | Country, Key Personel | Conceptual approach |
|---|---|---|
| 1984 | UK<br>Webster,Ziebeck et al. | Influence of Structural instability on magnetism of $Ni_2MnGa$ Heusler compound |
| 1989-1995 | Ukraine, Spain<br>Chernenko, Kokorin, Cesari et al. ⇓ | Elaboration of Ni-Mn-Ga alloy system as novel ferromagnetic shape memory alloys |
| 1996-1997 | Finland, Ukraine, USA<br>Ullakko, O'Handley et al. | Giant magnetostriction caused by twin boundary motion |

***Resulting in:***

**1998-present**

**Avalanche-like progress due to extension of experimental technique, modeling and developing other alloy systems**

1993

Proceedings of the International Conference on Martensitic Transformations (1992)
Monterey Institute for Advanced Studies © 1993
Monterey, California, USA

## $Ni_2MnGa$ AS A NEW FERROMAGNETIC ORDERED SHAPE MEMORY ALLOY

Vladimir A. Chernenko and Vladimir V. Kokorin

Institute of Metal Physics
Vernadsky str. 36
Kiev 252680 UKRAINE

ABSTRACT

$Ni_2MnGa$ alloy was found to exhibit the thermoelastic martensitic transformation accompanying by anomalies of the electric, thermal, elastic and
rent cubic
was confirm

1996

## Large magnetic-field-induced strains in Ni2MnGa single crystals

K. Ullakko, J. K. Huang, C. Kantner, and R. C. O'Handley
*Massachusetts Institute of Technology, Cambridge, Massachusetts 02139*
V. V. Kokorin
*Institute of Metal Physics, Kiev, Ukraine*

~Received 3 June 1996; accepted for publication 29 July 1996!

Strains of nearly 0.2% have been induced along @001# in unstressed crystals of Ni2MnGa with magnetic fields of 8 kOe applied at 265 K. These stains are associated with the superelastic motion of twin boundaries in the martensitic phase that is stable below about 274 K. © *1996 American Institute of Physics.* @S0003-6951~96!02439-4#

1974

598

## MAGNETOPLASTIC DEFORMATION OF Dy CRYSTALS

H. H. Liebermann and C. D. Graham, Jr.
University of Pennsylvania, Philadelphia, Pa. 19174

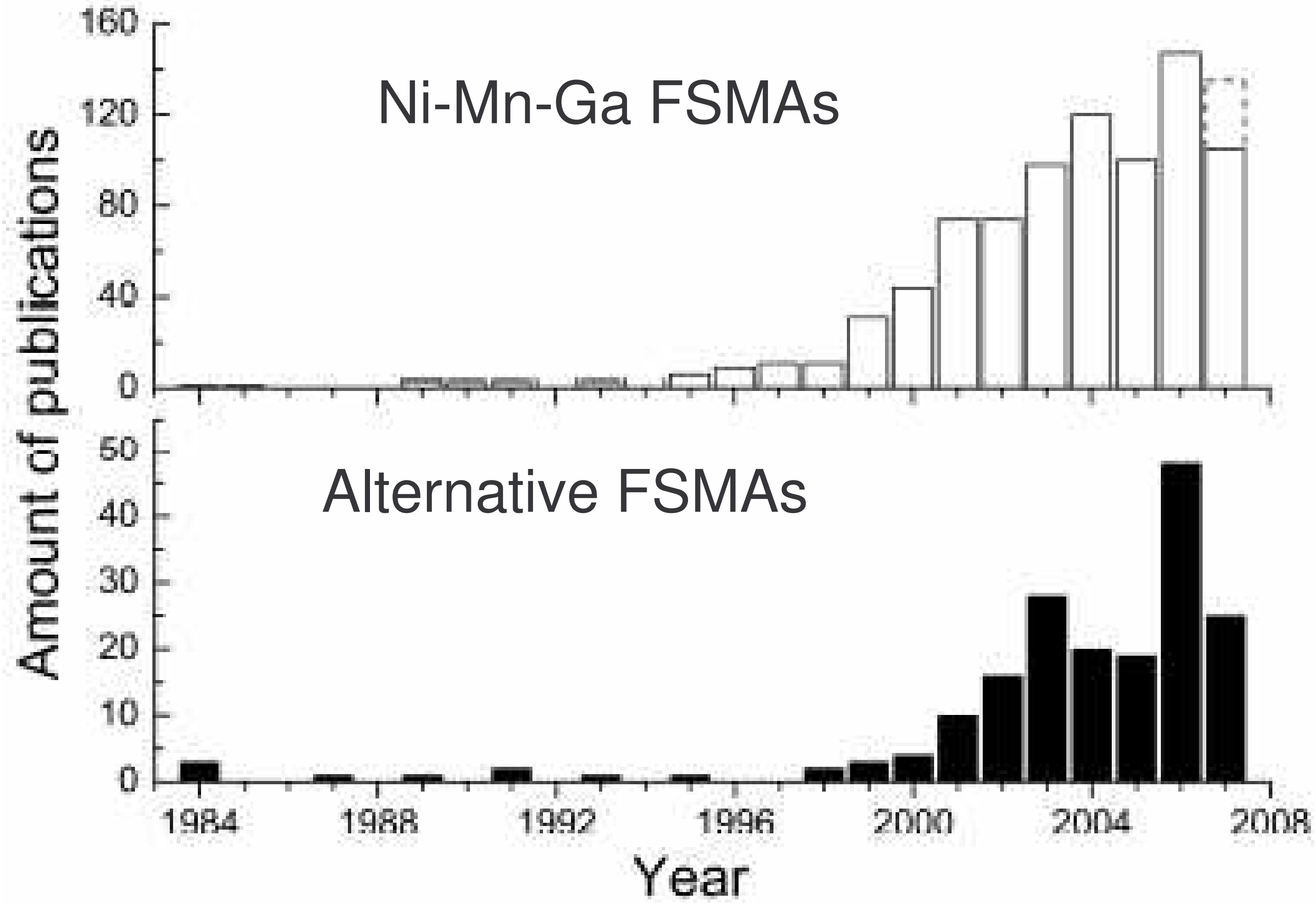
ABSTRACT

Several authors have reported that if single crystals of Dy or Tb are magnetized in fields of order 100 kOe parallel to a magnetic hard direction at 4.2K, a permanent plastic deformation occurs. Neither the

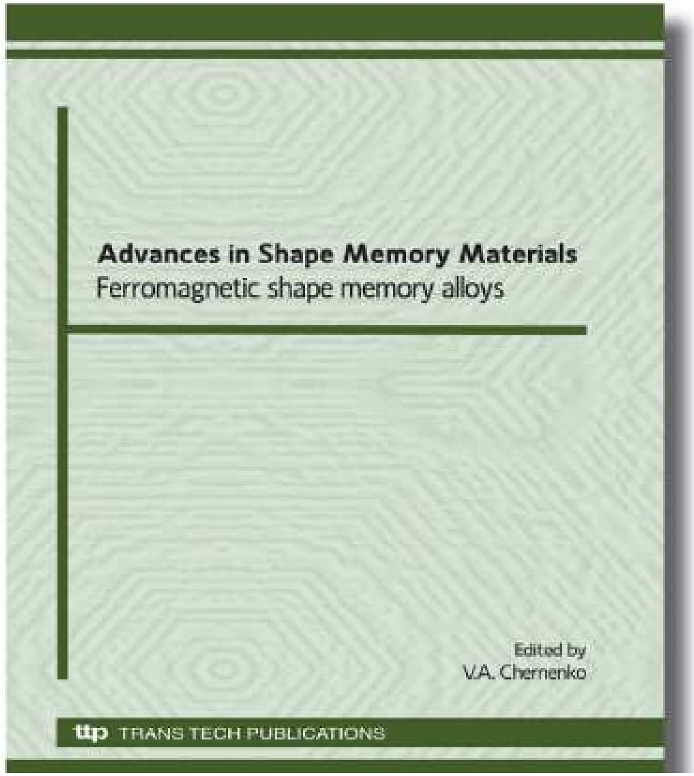
samples were spark-cut from the resulting button.

A major experimental problem was to devise a satisfactory method for holding the sample in the magnetic field. The crystal had to be free to deform, but also had to be held against large mechanical torques resulting from even slight misalignment. For example, if the applied field deviates only 1° from the

# FSMA science – vast interdisciplinary research field




Totally more than 1000 publications on FSMA



Advances in Shape Memory Materials
Ferromagnetic shape memory alloys
Edited by
V.A. Chernenko
ttp TRANS TECH PUBLICATIONS

## 2-nd International Conference on Ferromagnetic Shape Memory Alloys

ICFSMSA 2009, July 1-3, Bilbao, Spain

Guggenheim museum

FSMA science field is affluent by concepts which help understanding FSME and related phenomena

Some of them are:

lattice instability
soft-mode behavior
electron concentration

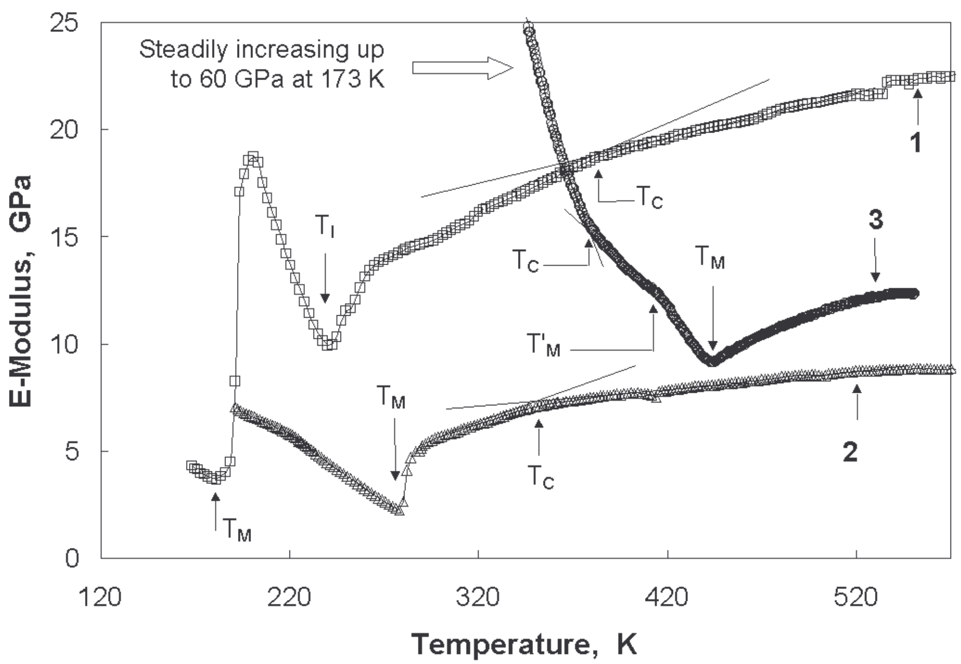
ferromagnetic shape memory effect (FSME)
magnetic-field-induced twin boundary motion

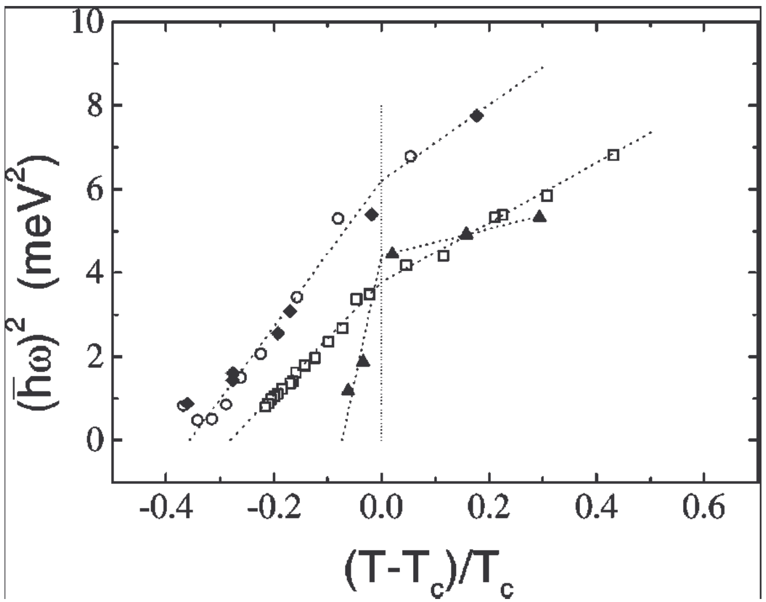
magnetostress
magnetomechanical mechanism

magnetic-field-induced superelasticity
twinning-strain-induced change of magnetization

# Modulus and phonon softening

TA2(1/3,1/3,0)

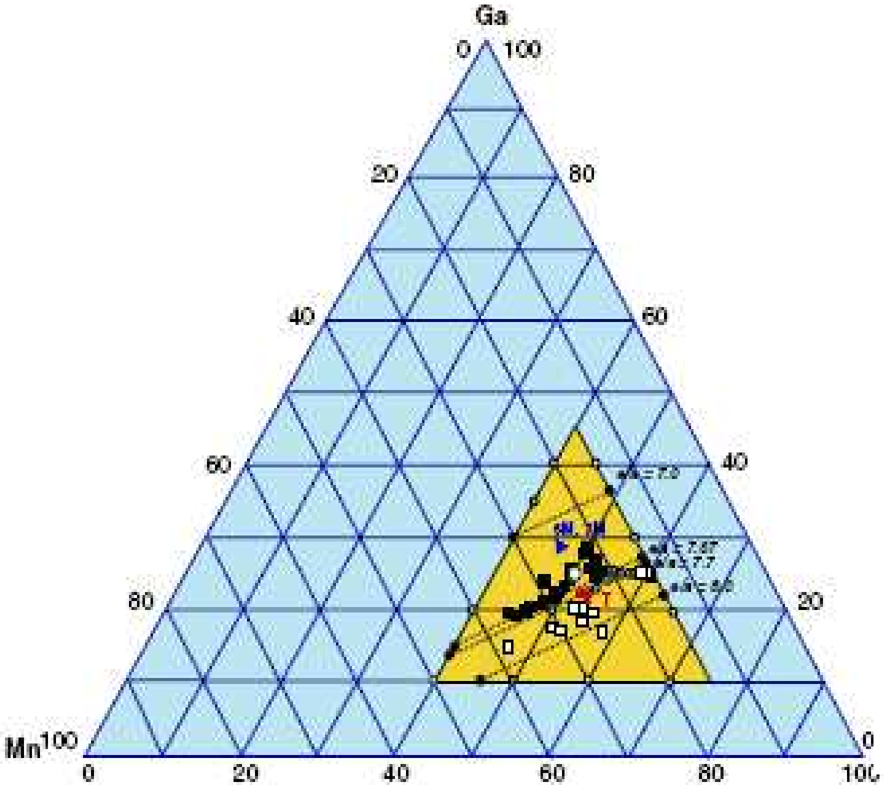



Chernenko et al.2002

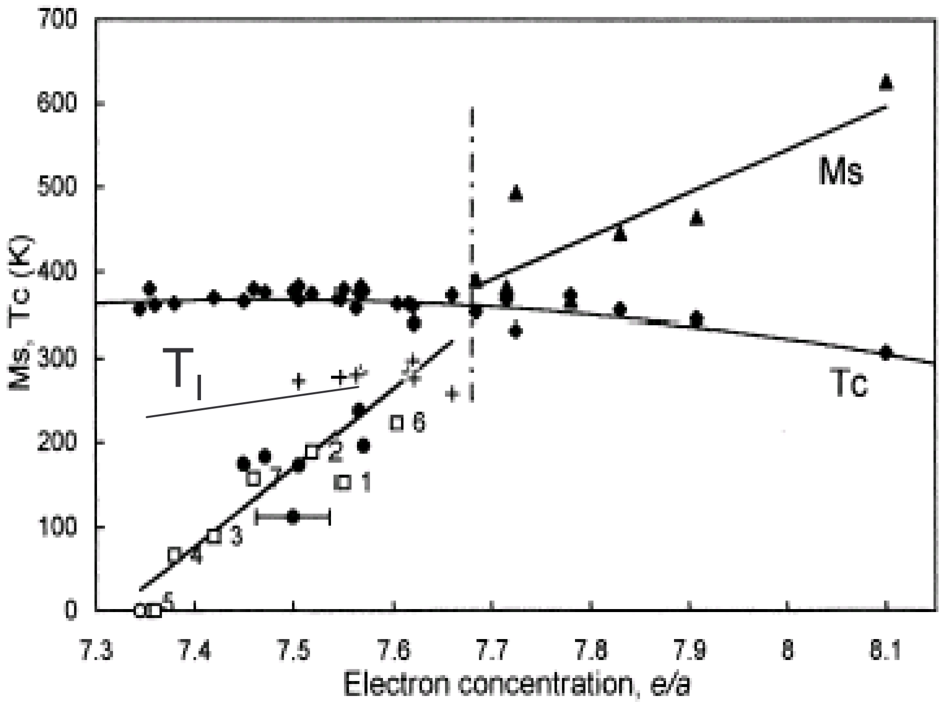
Manosa et al, 2001

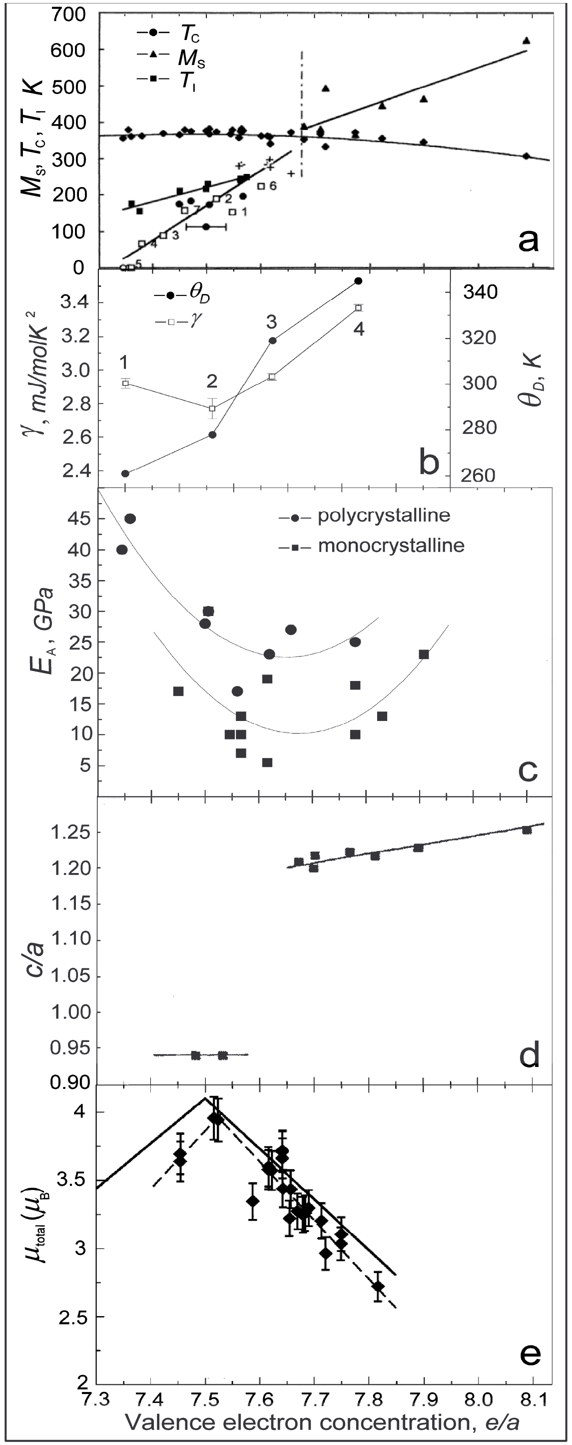
# Concentration dependence of transformations characteristics in Ni-Mn-Ga Heusler alloy system




Chernenko V. et al., 1995

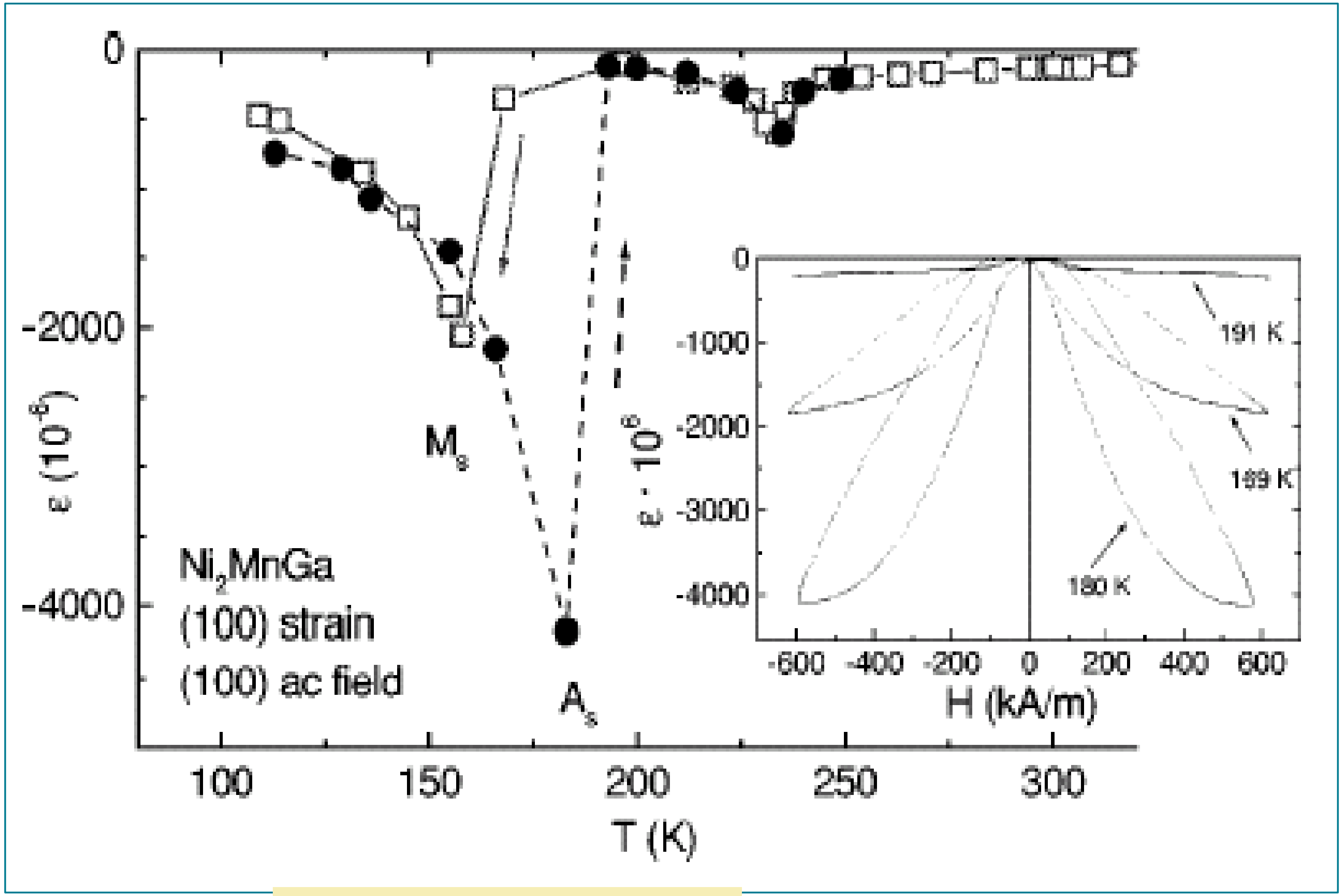
Chernenko V., 1999





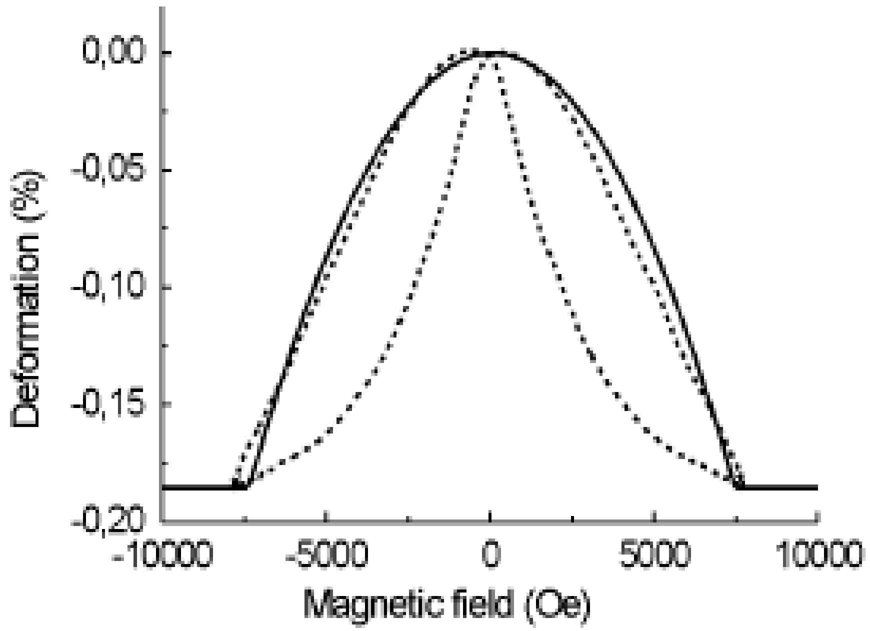

Effect of the valence electron concentration on the lattice, electronic and magnetic instabilities in Ni-Mn-Ga alloy system

# Quasi-elastic magnetostrain in polyvariant martensite




Chernenko et al., 2000

# Quasi-elastic magnetostrain: theory

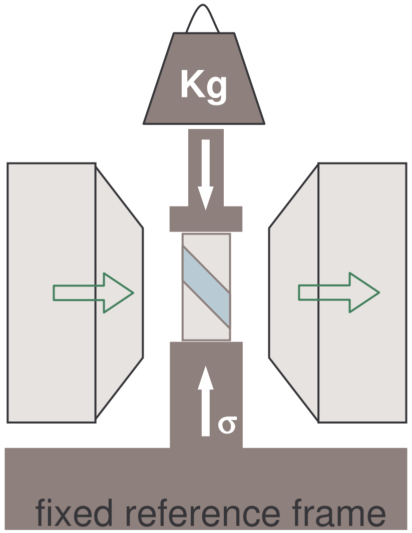


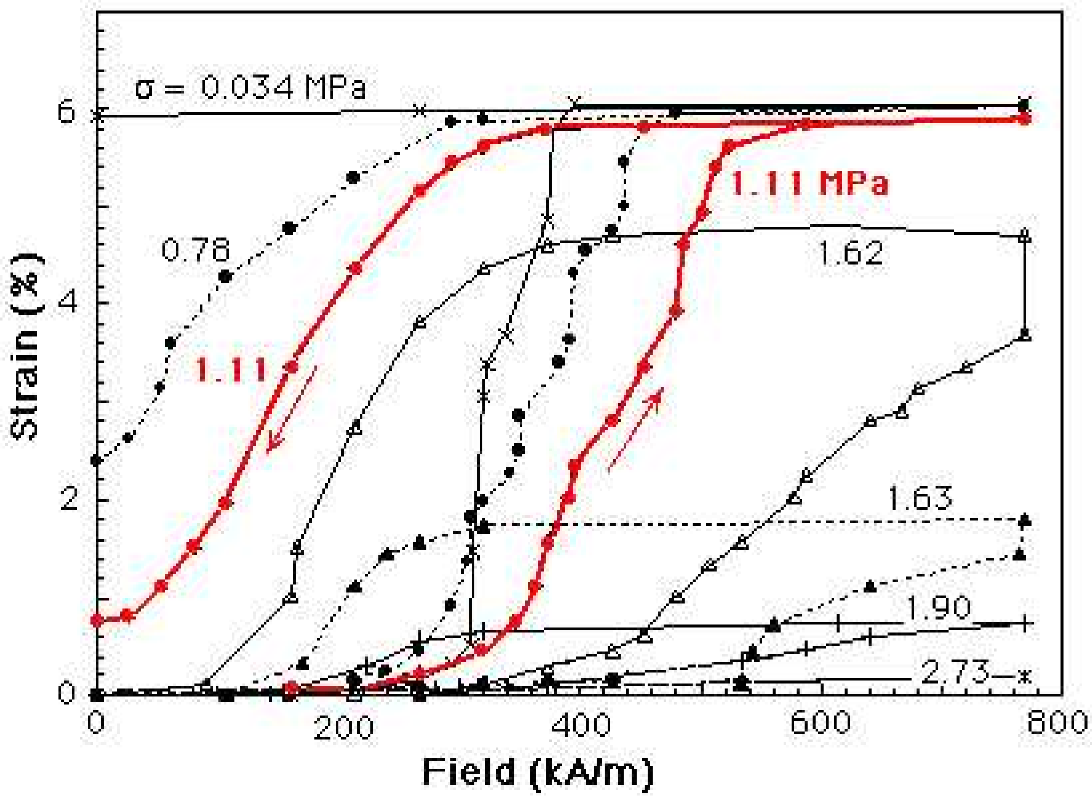

**[100] deformations induced by field H// [001] in $Ni_2MnGa$**

**160 K, M=525 G, δ=-23**

**$(\partial\varepsilon/\partial\sigma_3)^{-1} \approx 4.1$ GPa**

$$\langle \hat{\varepsilon}(H,T) \rangle \approx \frac{2}{3} (\partial \hat{\varepsilon} / \partial \sigma_3)\, 18 \delta M^2(T) \cos^2 \psi_{[001]}$$

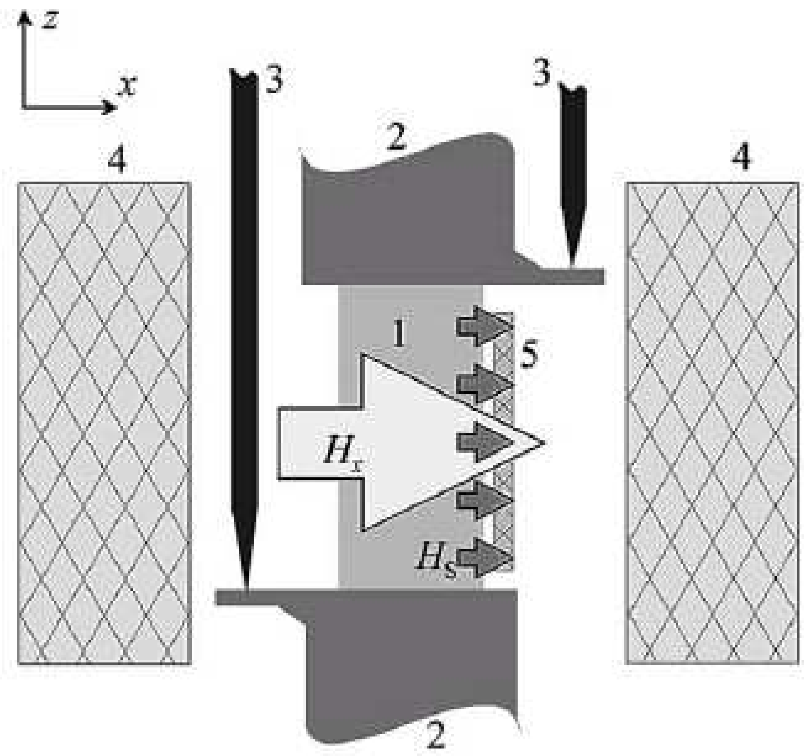



Murray 1999

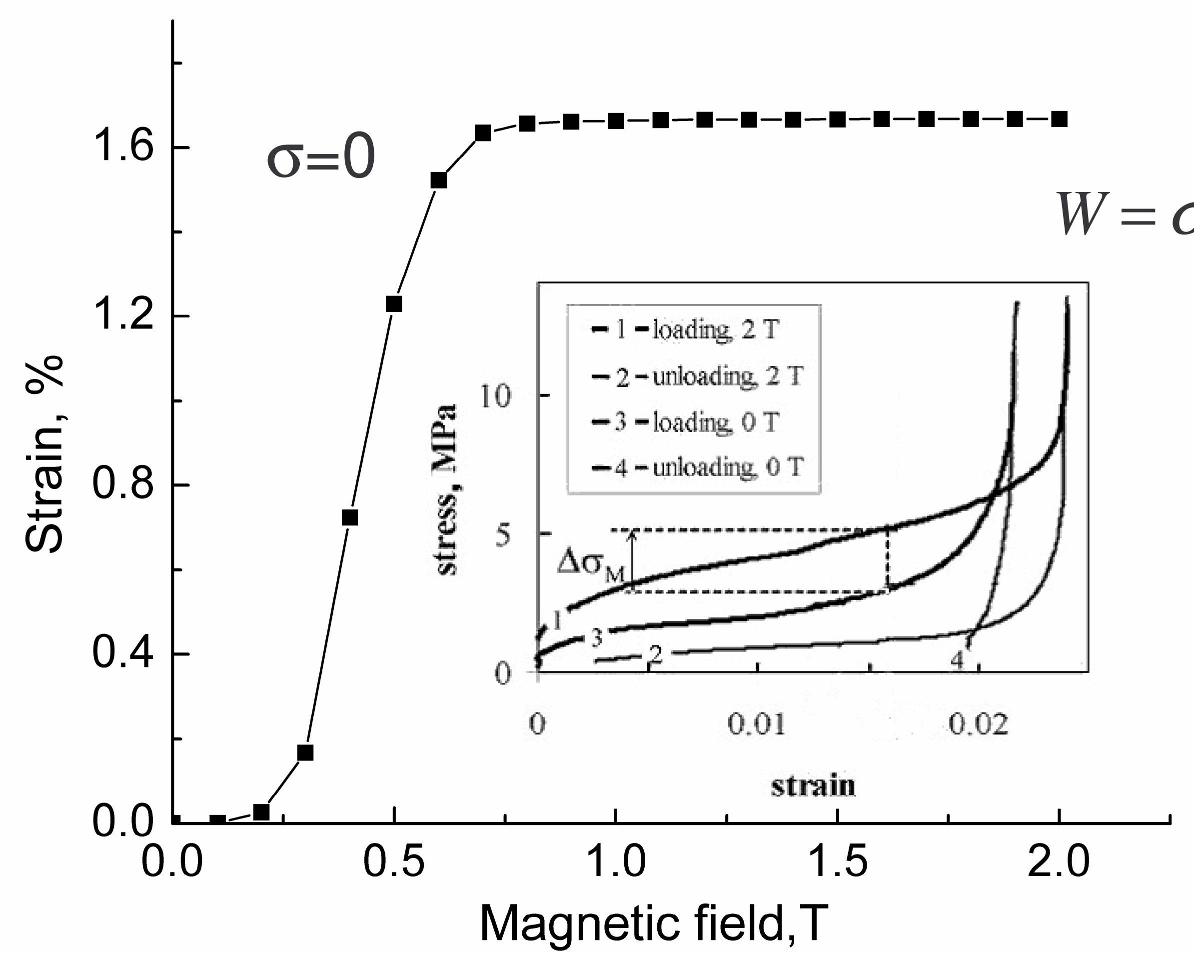
Static driving force experiments

# Experiment with magneto-mechanical orthogonal loading




Muellner et al., 2002;2003

# Strain vs H and stress-strain under $H_{const}$




$$W = \sigma^{(me)} \cdot \Delta\varepsilon^{(t)} \sim \delta M^2 (1 - c/a)$$

Muellner et al., 2002;2003

# MAGNETOELASTIC MODEL OF FERROMAGNETIC MARTENSITE

$$F = F_e + F_m + F_{me}. \qquad (1)$$

In Landau-type approach, Eq. (1) is invariant under symmetry group PM cubic phase.

For cubic – tetragonal MT

$$F_e = \frac{3}{2}(C_{11} + 2C_{12})u_1^2 + \frac{1}{6}C'(u_2^2 + u_3^2) \qquad (2)$$

$$u_1 = (\varepsilon_{xx} + \varepsilon_{yy} + \varepsilon_{zz})/3,$$

$$u_2 = \sqrt{3}(\varepsilon_{xx} - \varepsilon_{yy}), \quad u_3 = 2\varepsilon_{zz} - \varepsilon_{yy} - \varepsilon_{xx}$$

$$F_m = J y^2/2 + M^2(\mathbf{m}\cdot\mathbf{D}\cdot\mathbf{m}) - \mathbf{mH}M, \qquad (3)$$

$$y = M(T)/M(0) \qquad \mathbf{m} = \mathbf{M}(T)/M(T)$$

$$F_{me} = -\delta_0 y^2(T) u_1 - \delta_1\left[\sqrt{3}\left(m_x^2 - m_y^2\right)u_2 + \left(2m_z^2 - m_y^2 - m_x^2\right)u_3\right], \qquad (4)$$

Lvov, Chernenko et a
1998 - 2004

# Magnetostress concept and mechanical equivalence

follows explicitly from magnetoelastic model of martensite

$$F = \frac{1}{2} C' \varepsilon_{ii}^2 + \frac{1}{2} A M_i^2 - \delta M_i^2 \varepsilon_{ii} - M_i H_i$$

$$A \sim \delta(1 - c/a)$$

$$\sigma_{ik} = (\partial F / \partial \varepsilon_{ik})_T$$

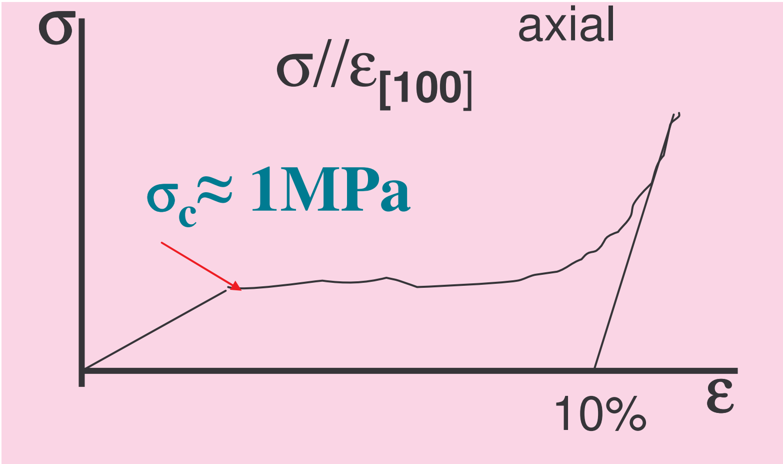
$$\sigma_{ii} = C' \varepsilon_{ii} + \delta M_i^2$$

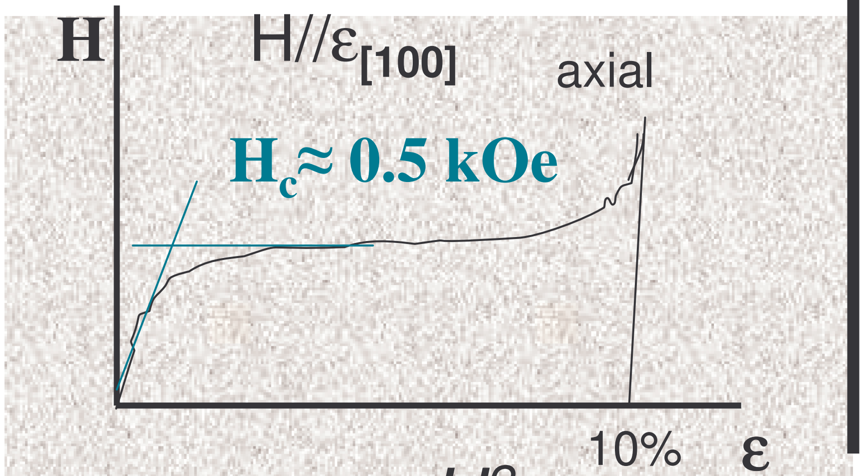
generalized Hooke's law for stressed ferromagnetic crystal

Conclusion:
magnetoelastic coupling is responsible for magnetostress

# Equivalence principle *( JMMM,1999; EPJ-B,2002)*

**magnetoelastic stress state= mechanical stress state**




$$\sigma_c = c_{me} H^2_c$$

**(Quasi-)Elastic regime:**

$$\sigma^{mech}, \sigma^{mag} = S \cdot \varepsilon$$

**where $\varepsilon << \varepsilon^{(MT)}$**

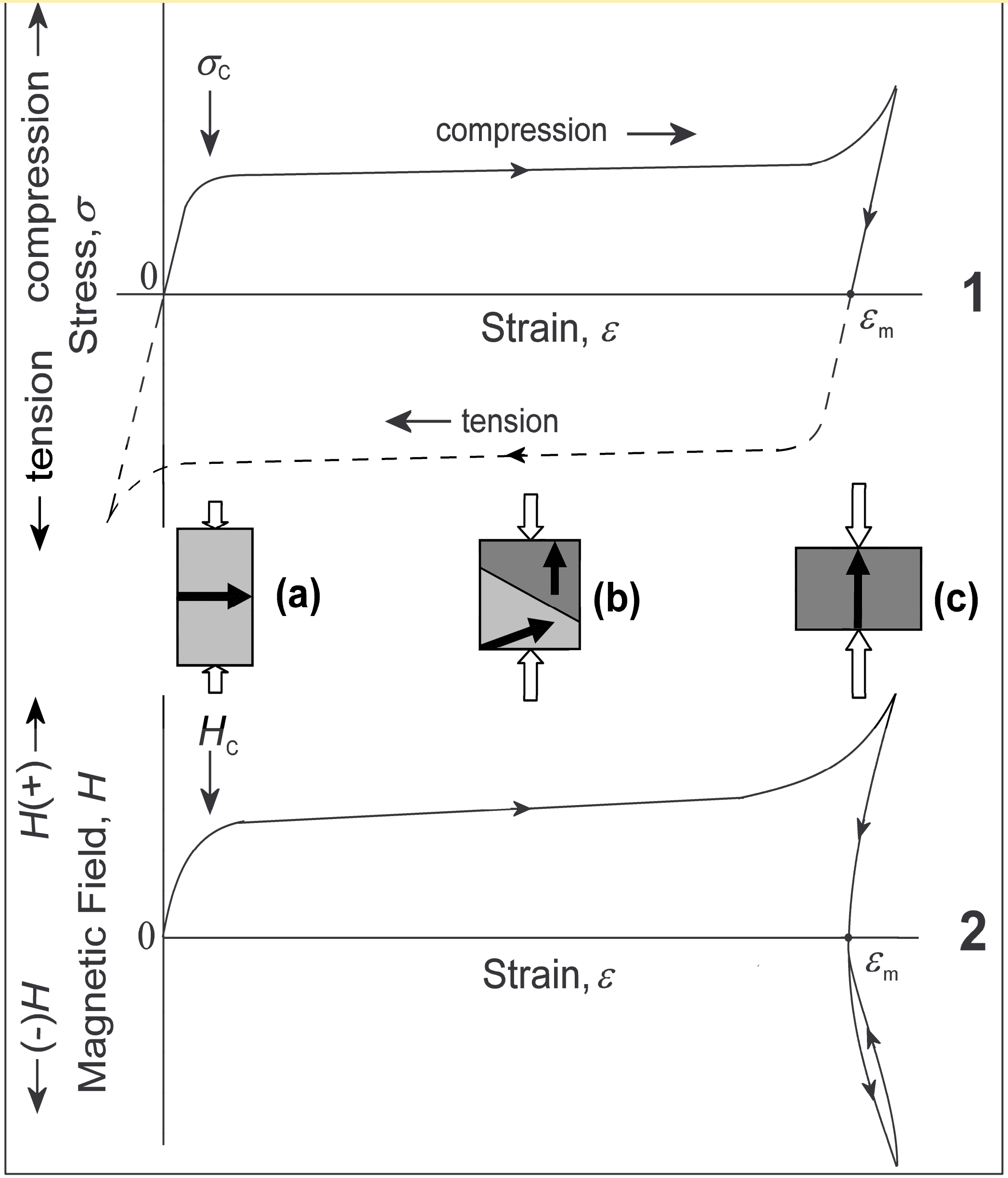
**$\varepsilon$ reversible after force removal**

**Anelastic (flow) regime:**

$$\varepsilon \sim \varepsilon^{(MT)} \sim f(\alpha)$$

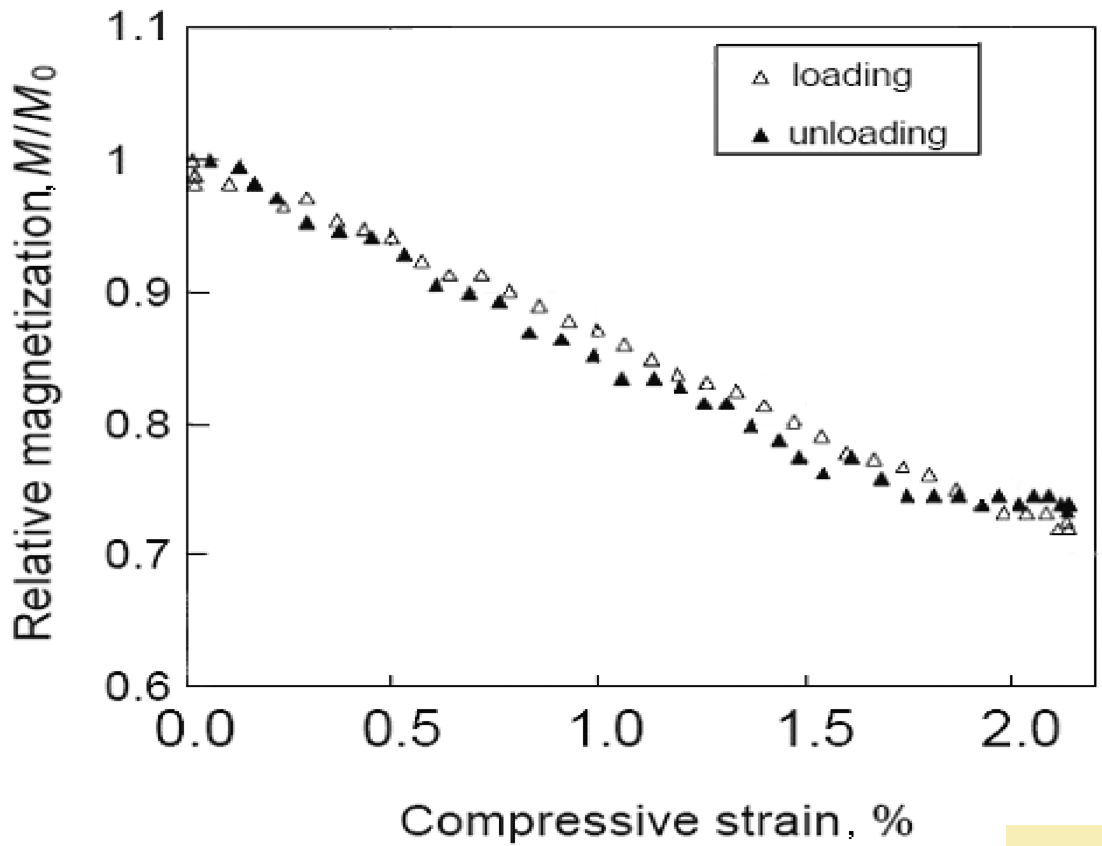
**$\varepsilon$ reversible when force direction is changed**

# Difference in mechanical and magnetic actuation



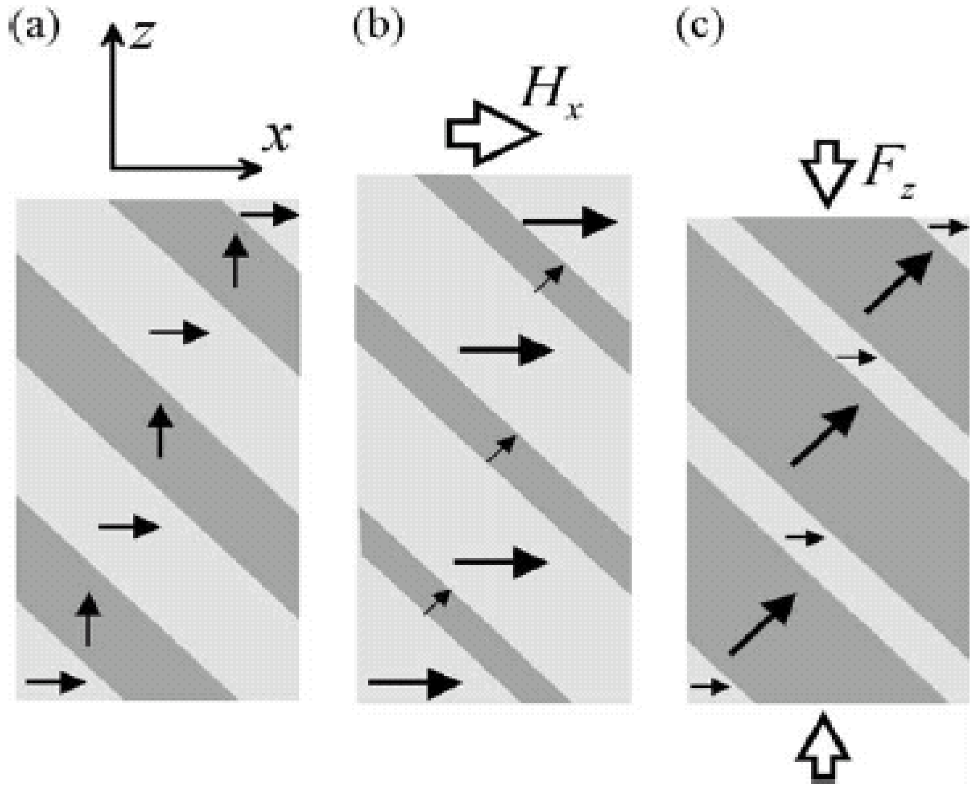
# Reverse to giant MFIS effect

## Magnetization change due to twinning strain under orthogonal loading

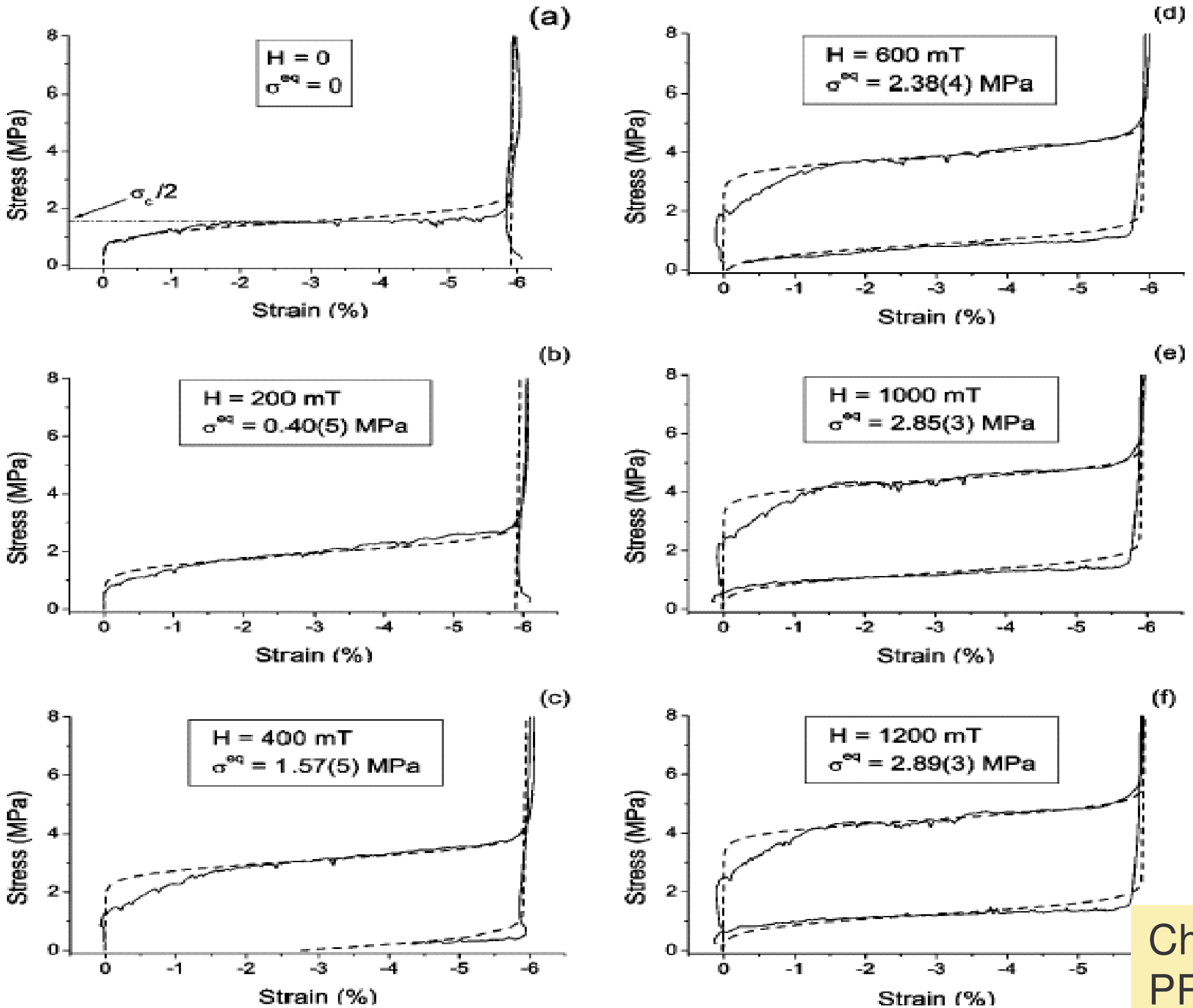



Muellner et al., 2003

# Schematic of magnetization process through deformation in ferromagnetic martensite



# Magnetomechanics of single variant $Ni_{52.0}Mn_{24.4}Ga_{23.6}$ marteniste




ε[010]

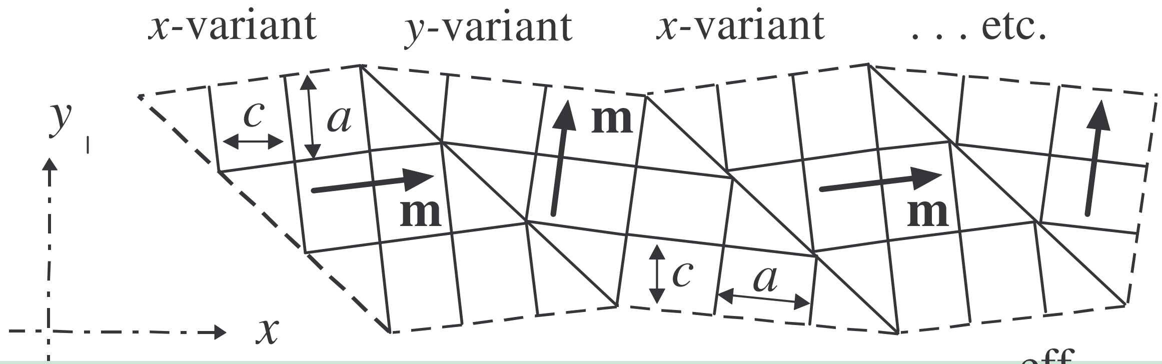
H//[100]

**tetragonal martensite**
**c/a<1**

**$T_{exp}$=293 K**

Chernenko,Lvov et al
PRB, 2004

# Magnetic field-induced superelasticity of Ni-Mn-Ga marteniste: modeling




(i) reorientation of variants is caused by total $\sigma^{\text{eff}} = \sigma_{xx} - \sigma_{yy}$ stress which is sum of mechanical and field-induced stresses, tensile if positive, compressive if negative, it is acting in x direction in each twin variant.

(ii) two-stage twin boundary rearrangement

(iii) critical stress $\sigma_n^{\text{eff}}$ is different for twin variants

(iv) statistical distribution of critical stresses around certain $\sigma_c$

$$p_n = \frac{1}{Z}\exp\left\{-\frac{(|\sigma_n^{\text{eff}}| - |\sigma_c|)^2}{2\sigma_0^2}\right\},$$

$$\varepsilon_{yy}^{(xy)} = -(S^{-1}/2)\sigma^{\text{eff}}(\sigma_{yy}, H) + (c/a - 1)[\alpha_y(\sigma_{yy}, H) - \alpha_y(0,0)].$$

**For each stress value, $N$ of disappeared twins (boundaries jumps) is found from condition** $|\sigma^{\text{eff}}| \geq |\sigma_n^{\text{eff}}|.$ **which gives rise with (ii) to:**

$$N(\sigma^{\text{eff}}) = N_0 \sum_n p_n \theta\left(|\sigma^{\text{eff}}| - |\sigma_n^{\text{eff}}|\right),$$

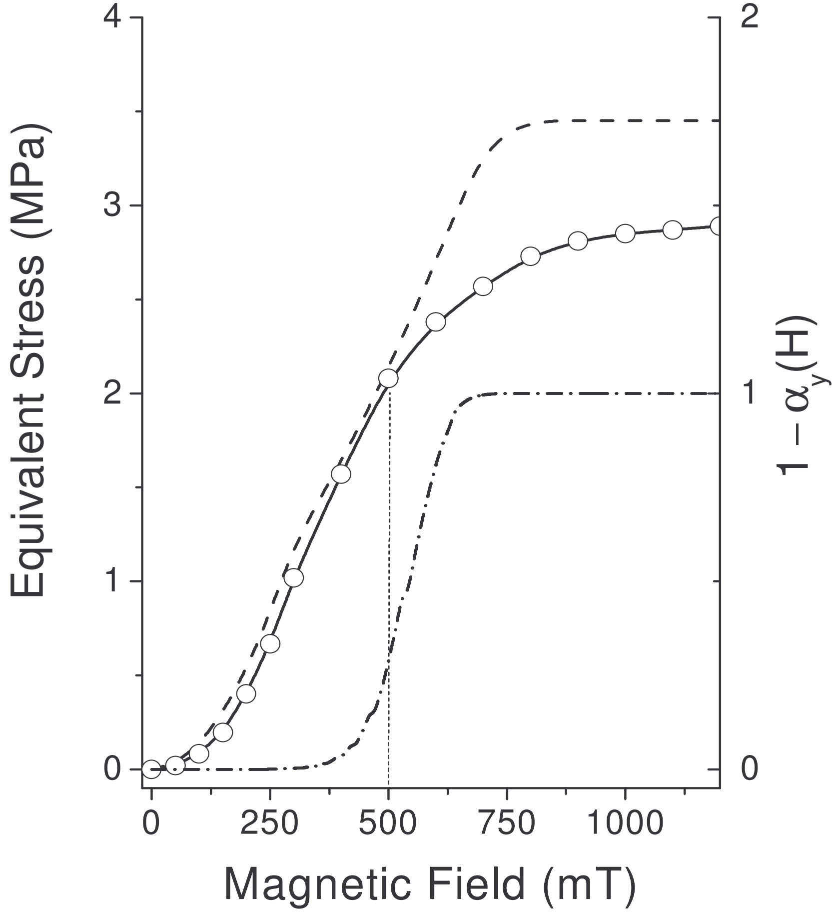
**According to (i):** $\sigma^{\text{eff}} = \sigma^{\text{eff}}(\sigma_{ik}, H_j), \;\; N = N(\sigma_{ik}, H_j).$

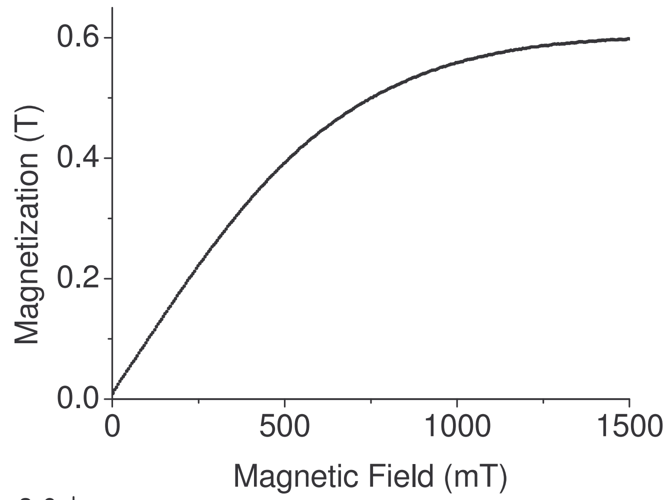
## During compression along $y$ ( H || $x$)

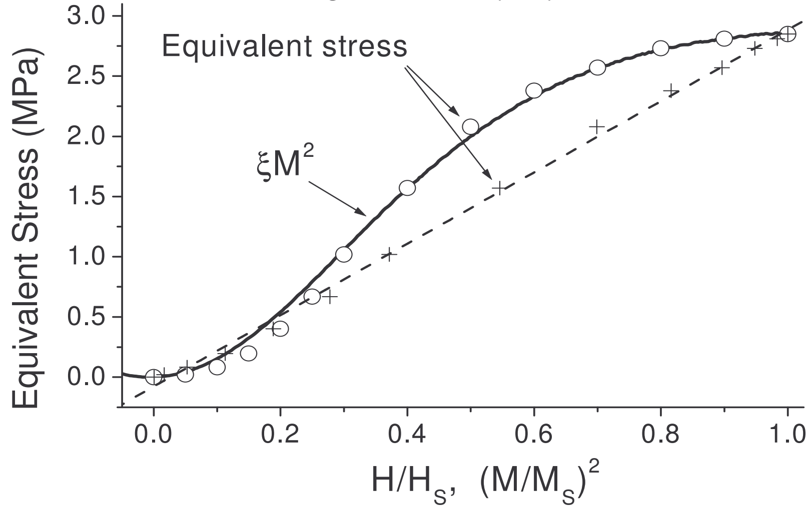
$$\alpha_y(\sigma_{yy}, H) = \begin{cases} \alpha_y(0,H)[1 - N(\sigma_{yy}, H)/N_0], & \sigma^{\text{eff}} < 0, \\ \alpha_y(0,H) + [1 - \alpha_y(0,H)]N(\sigma_{yy}, H)/N_0, & \sigma^{\text{eff}} \geq 0. \end{cases}$$

## During unloading with H || $x$

$$\alpha_y(\sigma_{yy}, H) = \begin{cases} \alpha_y(\sigma_{yy}^{\max}, H)[1 - N(\sigma_{yy}, H)], & \sigma^{\text{eff}} < 0, \\ \alpha_y(\sigma_{yy}^{\max}, H), & \sigma^{\text{eff}} > 0, \end{cases}$$




**Experiment: open circles**
**Fitting: solid line**
**Thermoelastic model: dash**
$\sigma_2(H_S)/2\sqrt{3}=3.45\,\mathrm{MPa}$
***x*-variant fraction:dash-dot**

**Magnetoelastic origin of magneto-strain in martensite !**

# Interdependence between magnetic properties and lattice parameters of Ni-Mn-Ga martensite

Research line followed scheme:

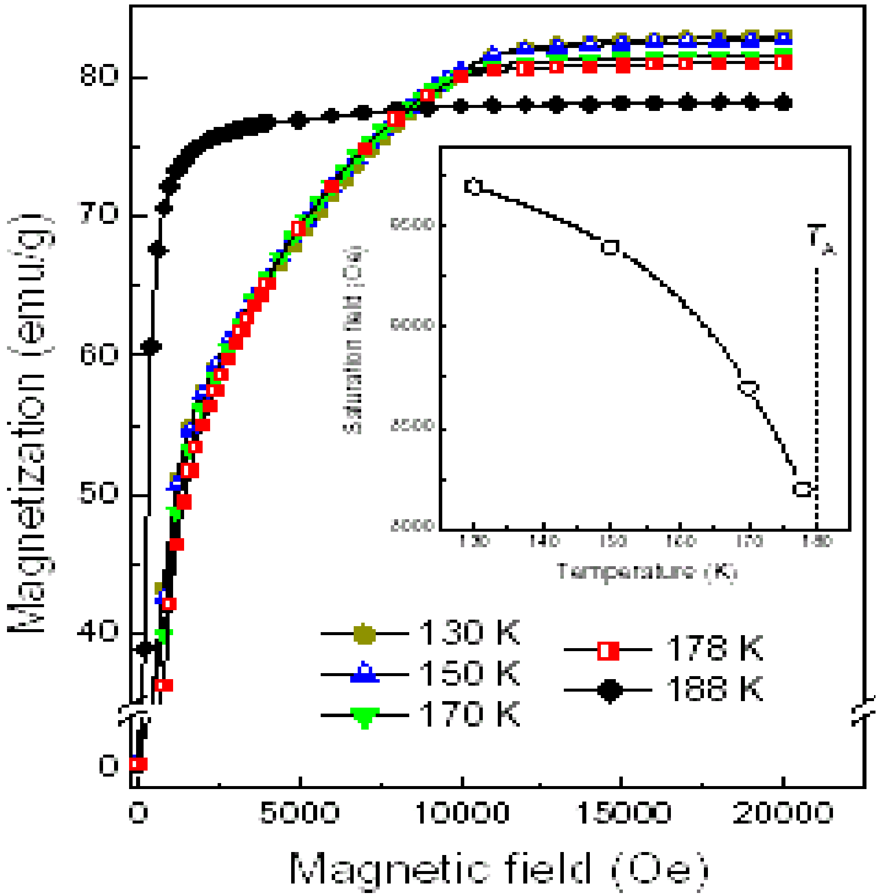
(i) measurements of Hs(T)

(ii) determination of c/a(T) according to our phenomenological theory:

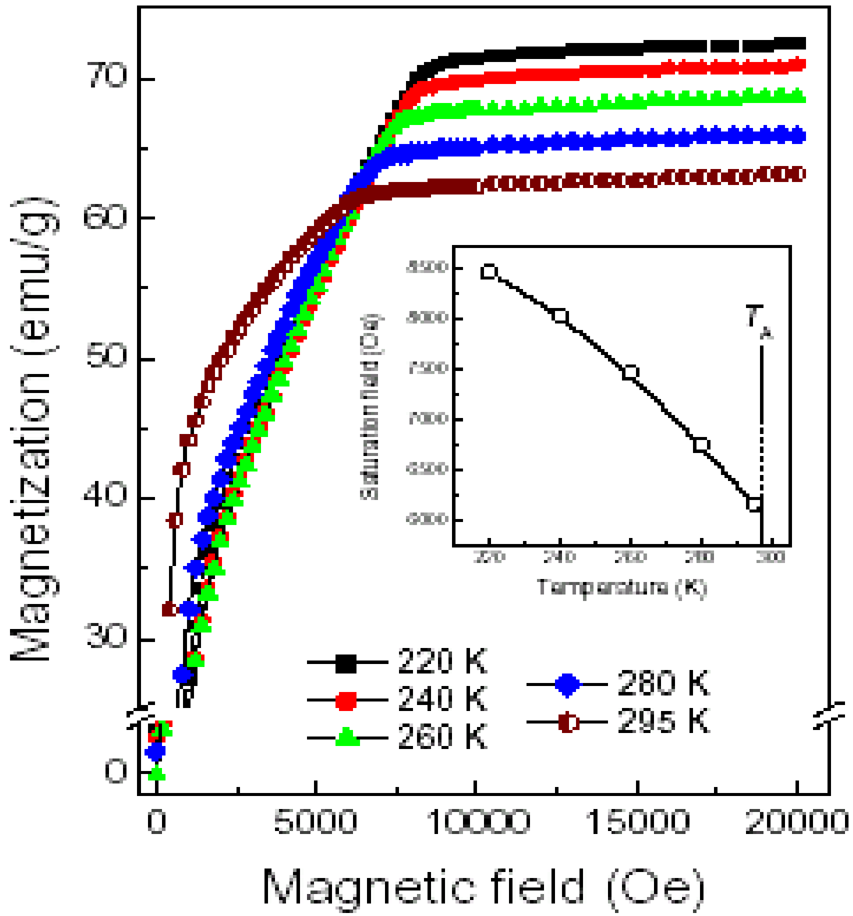
$$c/a = 1 - \frac{\left[(H_s/M) + |D_1 - D_2|\right]}{12|\delta|}.$$

(iii) comparison with c/a(T) function obtained by X-rays measurements.

$Ni_{49,5}Mn_{25,4}Ga_{25,1}$ (A1) : $T_M$ = 170 K, $T_A$ = 180 K , TC = 381 K
$Ni_{52,6}Mn_{23,6}Ga_{23,8}$ (A2) : $T_M$ = 284 K, $T_A$ = 297 K, $T_C$ = 363 K

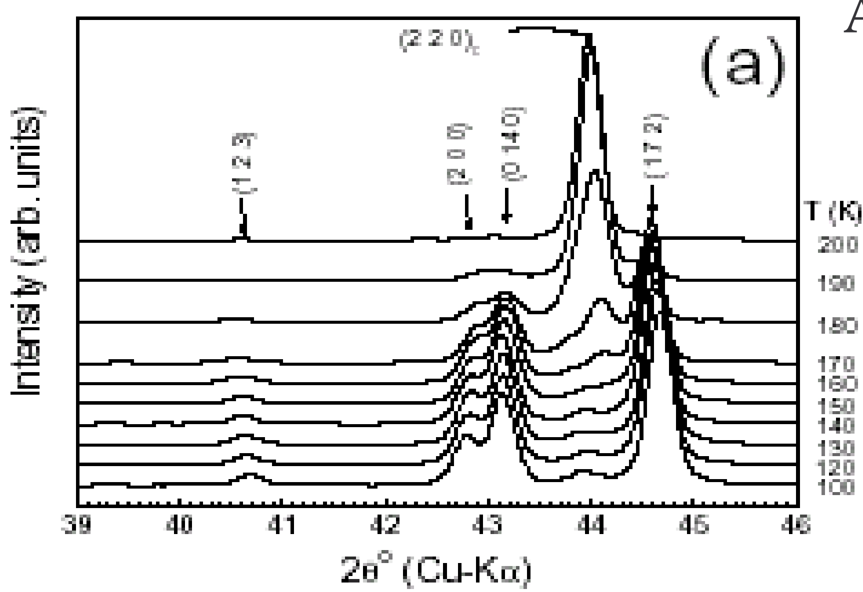


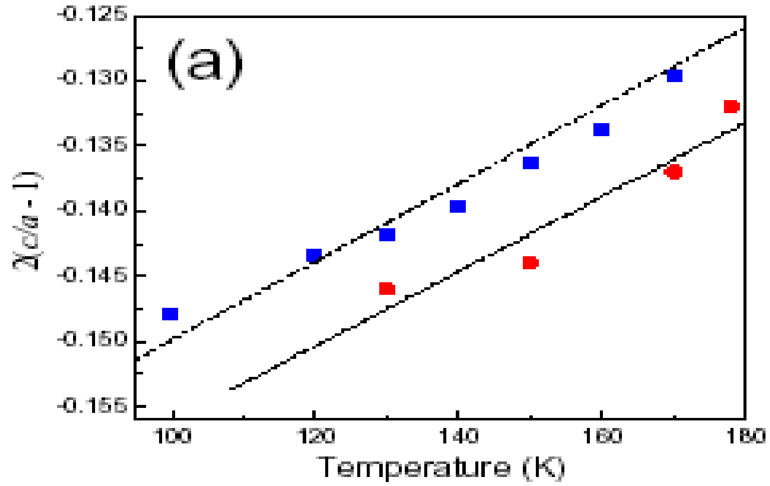

**A1**



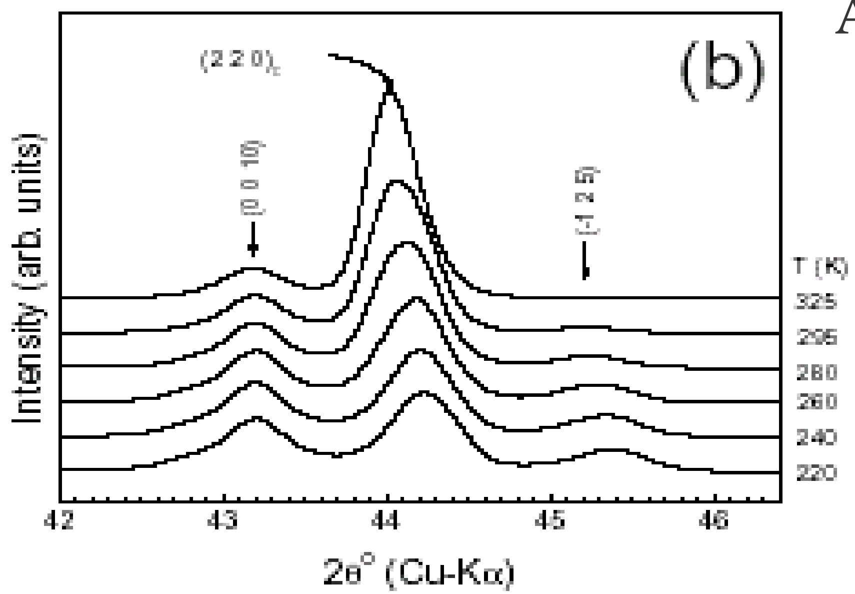

**A2**

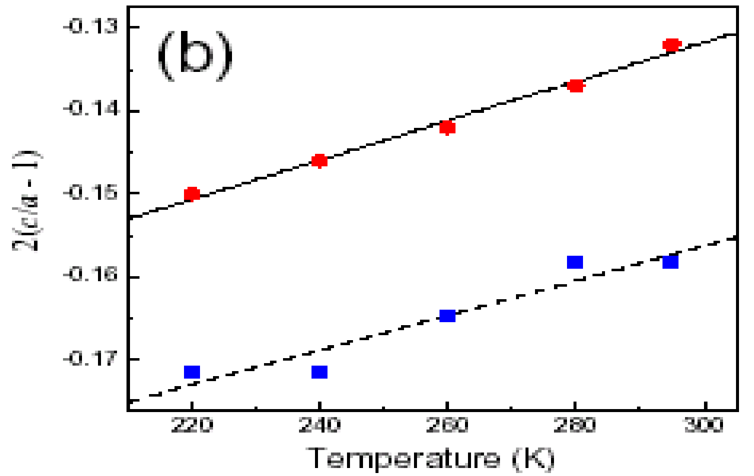
**A1**




**A2**

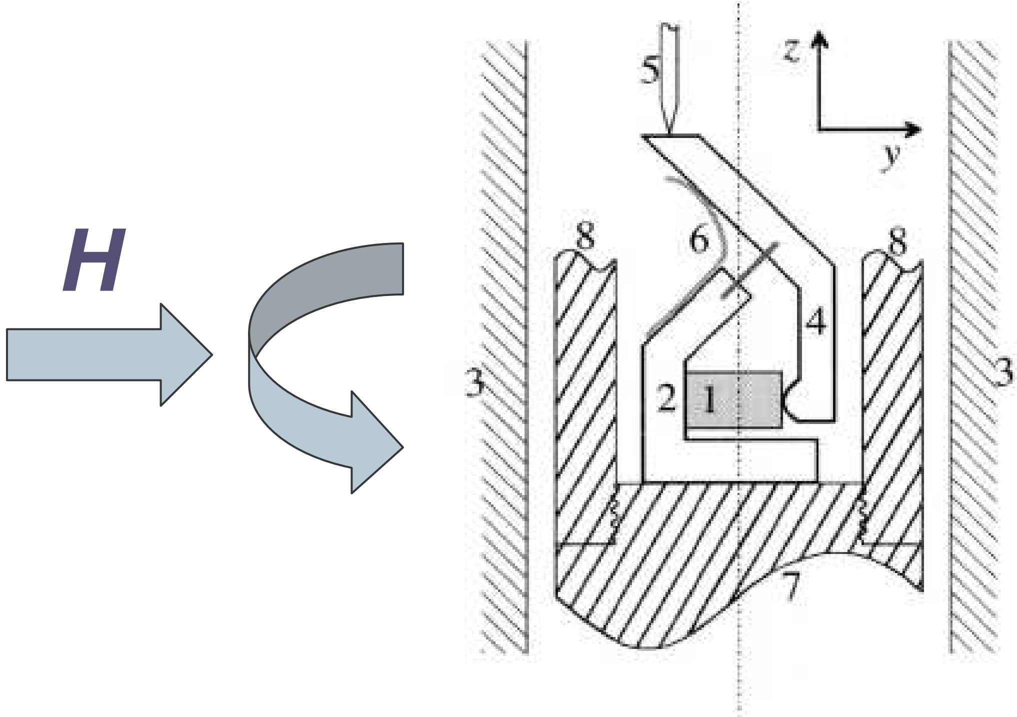



From X-rays (squares)
From M(T) (circles)

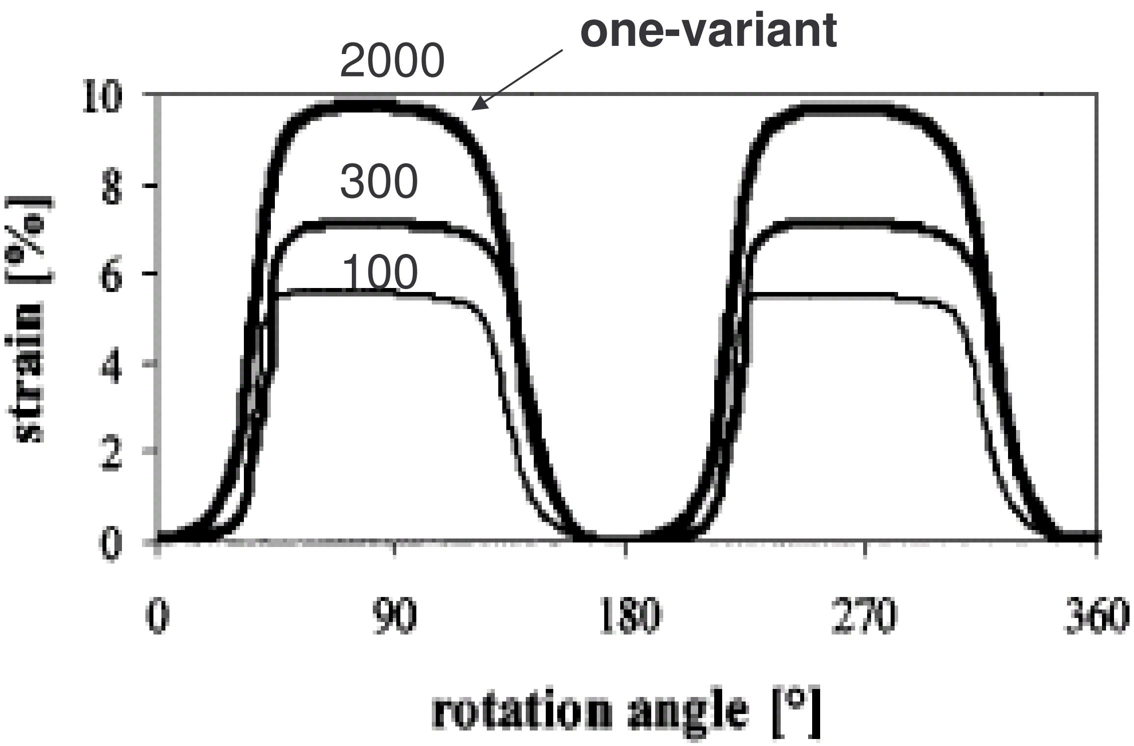
**So, validity of Eq. c/a vs $H_s$ is confirmed!**

# Magnetostress loading in rotation configuration




Muellner et al., 2002;2004

# Periodic field-induced strain in Ni-Mn-Ga 14M martensite in rotating field of 10 kOe




1rot/min

Muellner et al., 2004

## *Ferromagnetic martensite*

**Magnetisation curves (quasielastic regime of magnetostrain**

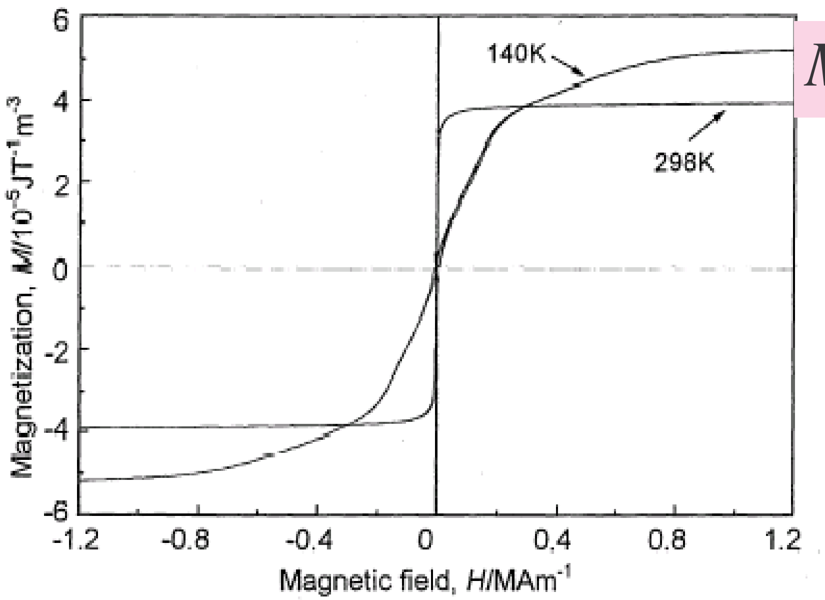
**(i) Reversible displacements of 180 deg. domain walls**

**(ii)Reversible rotation of magnetic vectors of variants**

**(iii)** $\beta = const$

$$M(H,T) = M(T)[\beta\Delta(H,T) + (1-\beta)\cos\psi(H,T)]$$

**H//[100]< Hs**




$$M(T) = M(0)\tanh[(T_C/T)M(T)/M(0)]$$

$$\Delta(H,T) = H/D_2M(H,T)$$

**for** ***H<Hc***

$$\Delta(H,T) = 1$$

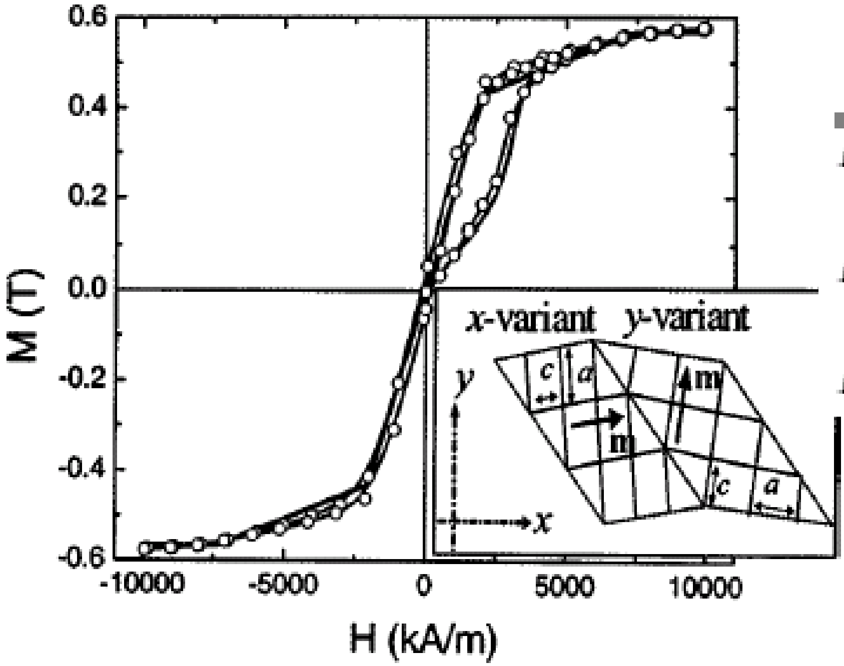
**for** ***H>Hc***

Lvov, Chernenko et al., 2003

## *Ferromagnetic martensite*

# Magnetisation curves (twin rearrangement)

### (iii) $\alpha = f(H)$




**For xy variant, H//[010]**

$$M_{rot} = M_S[(H/H_S)\theta(H_S - H) + \theta(H - H_S)],$$

$$M_{dis} = M_S[(H/H_c)\theta(H_c - H) + \theta(H - H_c)],$$

$$M_{rel} = \alpha_y(H)M_{dis} + [1 - \alpha_y(H)]M_{rot},$$

$$\alpha_y(\sigma) = \begin{cases} \alpha_y(0)[1 - N(\sigma)/N_0], & \sigma < 0 \\ \alpha_y(0) + [1 - \alpha_y(0)]N(\sigma)/N_0, & \sigma \geq 0 \end{cases}$$

$$\alpha_y(\sigma) = \begin{cases} \alpha_y(\sigma_{max})[1 - N(\sigma)/N_0], & \sigma < 0 \\ \alpha_y(\sigma_{max}), & \sigma \geq 0 \end{cases}$$

$$\sigma_H = \sigma(H_H)$$

$$\sigma(H) = c_{me}H^2,$$

where $c_{me} = -12\delta M_S^2/H_S^2$

$$\langle M \rangle = \tfrac{1}{3}(M^{xy} + M^{zy} + M^{zx})$$

**H increasing** $M^{xy} = M^{zy} = M_{dis}$

**H decrease** $M^{xy} = M^{zy} = M_{rel},\ M^{zx} = M_{rot}$

## Conclusions

Magnetoelastic model satisfactorily describes magnetomechanics in quasi-elastic regime

## While

In case of twin rearrangements, statistical approach should be additionally involved to account for peculiar magnetomechanics and magnetization phenomena in ferromagnetic thermoelastic martensites

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