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Growth and Characterization of High Frequency Materials

DAAD19-00-1-0054

5. FUNDING NUMBERS

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DAAD19-00-1-0054

6. AUTHOR(S)

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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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8. PERFORMING ORGANIZATION

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11. SUPPLEMENTARY NOTES

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12 a. DISTRIBUTION / AVAILABILITY STATEMENT

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12 b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

This instrumentation award had following objectives:

1. Obtain a sputtering deposition system
2. Upgrade a UHV system to a Molecular Beam Epitaxy system
3. Build new FMR (ferromagnetic resonance) systems
4. Upgrade BLS (Brillouin Light Scattering) system

All of these objectives have been accomplished. We purchase a sputtering system equipped with 5 guns and e-beam system. We upgraded deposition system to a single chamber MBE system. We built four FMR systems working at 24, 35, 45 and 55 GHz and upgraded our BLS system. The details are provided in the narrative part of this report.

14. SUBJECT TERMS

Instrumentation Award

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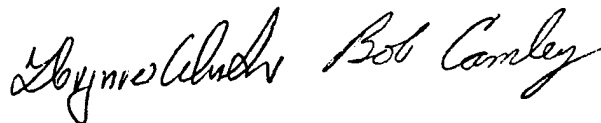
December 15, 2001

Dr. Mikael Ciftan
US Army Research Office
P. O. Box 12211
Research Triangle Park, NC 27709

Dear Mikael,

Please find enclosed the final report for our instrumentation grant. This instrumentation grant REALLY helped us. We obtained a sputtering system and created 4 new ferromagnetic resonance systems (24 GHz, 35 GHz, 45 GHz and 55 GHz) and upgraded our Brillouin Light Scattering (BLS) system and finally built a second Molecular Beam Epitaxy (MBE) system. All this equipment is now actively in use and is helping substantially in our effort to study the physics of magnetic materials at high frequencies and to develop high frequency applications. Thank you so very much for your support.

Sincerely



Z. Celinski and R. E. Camley

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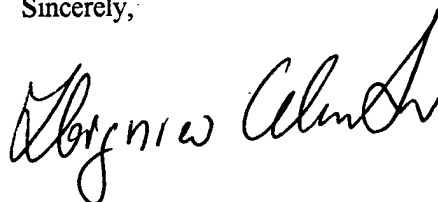
CONTRACT/GRANT NUMBER: DAAD19-00-1-0054

REPORT TITLE: Growth and Characterization of High Frequency Materials

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SUBMITTED FOR PUBLICATION TO (applicable only if report is manuscript):

Sincerely,



Growth and Characterization of High Frequency Materials

(DAAD19-00-1-0054)

Statement of Major Problems Studied

The frequency range of 10-100 GHz has become increasingly important for commercial and military communication and tracking. We are working on developing new magnetic materials and devices that can be useful for signal processing in this range. To do this we requested funds to improve our capabilities for the growth of high-quality magnetic materials and for the characterization of these materials. In specific we requested funds for the following:

1. Obtain a sputtering deposition system
2. Upgrade a UHV system to a Molecular Beam Epitaxy system
3. Build new FMR (ferromagnetic resonance) systems
4. Upgrade BLS (Brillouin Light Scattering) system

All of these tasks have now been accomplished. The sputtering system has been delivered and has created its first samples. In the upgrade of the UHV system to an MBE we installed UHV linear e-gun, RHEED system, thickness monitor and cryo-pumps. In addition we modified an existing UHV manipulator to work in this system. We created 4 new ferromagnetic resonance systems (24 GHz, 35 GHz, 45 GHz and 55 GHz). A low temperature dewar for measurements from room temperature to 24 K was purchased and implemented on the 24 GHz system. The BLS system was upgraded by adding new software and new electronics for the the Fabry-Perot interferometer. A second electro-magnet which allows us to reach 8.5 kG was also installed.

A further portion of the instrumentation grant proposal read "This equipment will be used to train students of all levels (undergraduates through PhD) in the growth and characterization of magnetic based devices." This too has been accomplished. We list below the students working on this equipment.

High School Students:

Brian Camley

Ben Haeffele

Michael Subialka

Christopher Bohm

Reginald Kerr

Undergraduates:

Tammy O'Keevan

Andrew Hutchison

Carl Coffield

Graduates students:

Nick Cramer

Post-Doctoral

Leszek Malkinski

Bijoy Kuanr

Publications:

Although this was an instrumentation grant, it did result in one publication. We certainly expect additional publications based on work with the equipment purchased under this grant.


M. Hekert, D. Tietjen, C. Schneider, N. Cramer, L. Malkinski, R.E. Camley, and Z. Celinski, "Thermal stability and degradation mechanism of NiFe/Cu GMR multilayer systems" J. Appl. Phys. (in press)

FINANCIAL STATUS REPORT

(Short Form)

(Follow instructions on the back)

1. Federal Agency and Organizational Element to Which Report is Submitted Army Research Office	2. Federal Grant or other Identifying Number Assigned By Federal Agency DAAD19-00-1-0054	OMB Approval No.	Page of 1 1 pages
3. Recipient Organization (Name and complete address, including ZIP code) UNIVERSITY OF COLORADO AT COLORADO SPRINGS ACCOUNTING OFFICE-CONTRACTS AND GRANTS PO BOX 7150 COLORADO SPRINGS, CO 80933-7150			
4. Employer Identification Number 184-60000555	5. Recipient Account Number or Identifying Number 4537308	6. Final Report <input type="checkbox"/> Yes <input type="checkbox"/> No	7. Basis <input checked="" type="checkbox"/> Cash <input type="checkbox"/> Accrual
8. Funding/Grant Period (See Instructions) From: (Month, Day, Year) 4/1/00 To: (Month, Day, Year) 9/15/01		9. Period Covered by this Report From: (Month, Day, Year) 3/31/00 To: (Month, Day, Year) 9/15/01	
10. Transactions:	I Previously Reported	II This Period	III Cumulative
a. Total Outlays	0.00	145,000.00	145,000.00
b. Recipient share of outlays	0.00	10,000.00	10,000.00
c. Federal share of outlays	0.00	135,000.00	135,000.00
d. Total unliquidated obligations	0.00	0.00	0.00
e. Recipient share of unliquidated obligations	0.00	0.00	0.00
f. Federal share of unliquidated obligations	0.00	0.00	0.00
g. Total Federal share (Sum of lines c and f)	0.00	135,000.00	135,000.00
h. Total Federal funds authorized for this funding period		135,000.00	135,000.00
i. Unobligated balance of Federal funds (Line h minus line g)	0.00	0.00	0.00
11. Indirect Expense	a. Type of Rate (Place "X" in appropriate box) <input type="checkbox"/> Provisional <input checked="" type="checkbox"/> Predetermined <input type="checkbox"/> Final <input type="checkbox"/> Fixed		
	b. Rate 48%	c. Base -	d. Total Amount -
			e. Federal Share 0.00
12. Remarks: Attach any explanations deemed necessary or information required by Federal sponsoring agency in compliance with governing legislation.			
13. Certification: I certify to the best of my knowledge and belief that this report is correct and complete and that all outlays and unliquidated obligations are for the purposes set forth in the award documents.			
Typed or Printed Name and Title Milagros Togade, Accountant		Telephone (Area code, number, and extension) 719-262-3482	
Signature of Authorized Certifying Official 		Date Report Submitted 12/14/01	

REQUEST FOR ADVANCE OR REIMBURSEMENT		OMB APPROVAL NO.		PAGE
Standard Form 270		1 TYPE OF PAYMENT REQUESTED	a. "X" one, or both boxes ADVANCE REIMBURSEMENT X	2. BASIS OF REQUEST
			b. "X" the applicable box FINAL X PARTIAL	CASH X ACCRUAL
3. FEDERAL SPONSORING AGENCY AND ORGANIZATIONAL ELEMENT TO WHICH THIS REPORT IS SUBMITTED Army Research Office		4. FEDERAL GRANT OR OTHER IDENTIFYING NUMBER ASSIGNED BY FEDERAL AGENCY DAAD19-00-0054		5. PARTIAL PAYMENT REQUEST NUMBER FOR THIS REQUEST
6. EMPLOYER IDENTIFICATION NUMBER 84-6000555	7. RECIPIENT'S ACCOUNT NUMBER OR IDENTIFYING NUMBER 4537308	8. PERIOD COVERED BY THIS REQUEST		
		FROM 03/31/00 TO 9/15/00		
9. RECIPIENT ORGANIZATION Name: University of Colorado, Colorado Springs Sponsored Programs Accounting P.O. BOX 7150 COLORADO SPRINGS, CO 80933-7150		10. PAYEE (Where check is to be sent is different than item 9) Name: SAME as Box 9 Number and Street: City, State and ZIP Code:		
11. COMPUTATION OF AMOUNT OF REIMBURSEMENTS/ADVANCES REQUESTED				
PROGRAM/ FUNCTIONS/ ACTIVITIES	(a)	(b)	(c)	TOTAL
a. Total program outlays to date (As of date)		\$	\$	\$ 145,000.00
b. Less: Cumulative program income				
c. Net program outlays (Line a minus line b)				145,000.00
d. Estimated net cash outlays for advance period				
e. Total (Sum of lines c & d)				145,000.00
f. Non-Federal share of amount on line e				10000
g. Federal share of amount on line e				135,000.00
h. Federal payments previously requested				131,625.00
i. Federal share now requested (Line g minus line h)				3,375.00
j. Advances required by month, when requested by by Federal grantor agency for use in making prescheduled advances	1st month			
	2nd month			
	3rd month			
12. ALTERNATE COMPUTATION FOR ADVANCES ONLY				
a. Estimated Federal cash outlays that will be made during period covered by the advance				\$
b. Less: Estimated balance of Federal cash on hand as of beginning of advance period				
c. Amount requested (Line a minus line b)				\$
13. CERTIFICATION				
I certify that to the best of my knowledge and belief the data above are correct and that all outlays were made in accordance with the grant conditions or other agreement and that payment is due and has not been previously requested.	NAME AND TITLE			TELEPHONE (AREA CODE, NUMBER, EXTENSION)
	 ACCOUNTANT III 12/14/07			719. 262-3482

RESEARCH AGREEMENT NO. DAA19-00-1-0054

810100 50% DOWN PAYMENT FOR 1 EACH	5/3/2000	44,950.00
530101 T F S TECHNOLOGIES CELINSKI	5/12/2000	1,958.52
810100 50% BALANCE PAYMENT FOR 1 EACH	11/8/2001	44,950.00

SPUTTERING DEPOSITION SYSTEM **\$ 91,858.52**

530101 CAMERAWORKS CELINSKI	4/21/2000	245.00
810100 CRYOSTAT SYSTEM, SPECIAL MODEL	11/9/2000	16,131.80
530101 JRS SCIENTIFIC INSTRUMENTS	12/19/2000	1,416.45

FMR (FERROMAGNETIC RESONANCE) SYSTEMS
BLS (BRILLOUIN LIGHT SCATTERING) SYSTEM **\$ 17,793.25**

530101 LAB EQUIPMENT	4/7/2000	1,778.38
530101 HSD ENGINEERING INC CELINSKI	4/12/2000	339.90
530101 DUNIWAY STOCKROOM C CELINSKI	4/21/2000	140.43
537600 ADAMANT COMPUTERS CELINSKI	4/21/2000	570.31
530101 K SPACE ASSOCIATES CELINSKI	4/25/2000	2,214.05
530101 RBD ENTERPRISES CELINSKI	5/5/2000	2,564.68
530101 RBD ENTERPRISES CELINSKI	5/5/2000	912.71
537600 ADAMANT COMPUTERS CELINSKI	5/9/2000	896.40
530101 K SPACE ASSOCIATES CELINSKI	5/16/2000	500.00
530101 APPLIED VACUUM TECH CELINSKI	8/4/2000	919.83
530101 KURT J LESKER CO CELINSKI	8/14/2000	80.00
530101 ACOUSTICAL SOLUTION REHORST	8/23/2000	278.44
530101 RBD ENTERPRISES CELINSKI	8/23/2000	2,340.00
530101 LAB EQUIPMENT	9/9/2000	2,300.00
530101 LAB EQUIPMENT	11/20/2000	3,788.45
530101 STRUCTURE PROBE CELINSKI	11/27/2000	109.16
530101 THERMIONICS NORTHWE CELINSKI	12/5/2000	1,215.58
530101 LAB EQUIPMENT	1/31/2001	2,870.00
530101 REPAIR CRYOPUMP MODEL APD-8	4/14/2001	3,504.00
530101 LAB & TECH SHOP SUPPLIES	11/27/2001	(1,974.09)

UHV SYSTEM UPGRADE TO MOLECULAR BEAM EPITAXY SYSTEM **\$ 25,348.23**

TOTAL **\$ 135,000.00**

BUDGET:

SPUTTERING DEPOSITION SYSTEM	95,000.00
UHV SYSTEM UPGRADE TO MOLECULAR BEAM EPITAXY SYSTEM	35,000.00
FMR (FERROMAGNETIC RESONANCE) SYSTEMS	10,000.00
BLS (BRILLOUIN LIGHT SCATTERING) SYSTEM	5,000.00
	<u>145,000.00</u>
PAID BY UNIVERSITY OF COLORADO AT COLORADO SPRINGS	(10,000.00)
	<u>135,000.00</u>

Thermal stability and degradation mechanism of NiFe/Cu GMR multilayer systems

M. Hecker, D. Tietjen, C.M. Schneider,

IFW Dresden, P.O.B. 270016, 01171 Dresden, Germany

N. Cramer, L. Malkinski, R.E. Camley, and Z. Celinski

Department of Physics, UCCS, Colorado Springs, USA

Abstract

Ni₈₀Fe₂₀/Cu multilayers show large giant magneto resistance (GMR) at low magnetic saturation fields. The GMR signal is known to degrade irreversibly at elevated temperatures. Clarification of the relevant deterioration mechanisms refines our basic understanding of the GMR effect and may help to improve the thermal stability of devices. We therefore investigated structural, transport and magnetic properties of sputtered Ni₈₀Fe₂₀/Cu multilayers in the as-deposited state and after different anneals (up to 600°C) by X-ray techniques, transport measurements, ferromagnetic resonance (FMR) and magneto-optical Kerr effect (MOKE). Multilayers with the second maximum of the antiferromagnetic (afm) coupling showed a sharp drop of the GMR at about 250°C. The changes of the transport properties were associated with a series of structural alterations. These ranged from grain growth and defect reduction through texture sharpening and stress evolution up to the onset of interdiffusion. Interdiffusion changed the NiFe layer composition and the interface structure and finally caused layer intermixing with a loss of the former multilayer structure. Further insight into the magnetic behavior was gained from FMR and MOKE measurements, from which we determined the in-plane magnetic anisotropies, the strength of the afm coupling (bilinear and biquadratic) and the homogeneity of the layer magnetization as a function of the annealing temperature.

The discoveries of antiferromagnetic exchange coupling¹ and giant magnetoresistance (GMR) effects^{2,3,4} have opened a possibility for novel applications in different areas, such as magnetic recording, non-volatile memories and magnetic sensors. One of the most important applications was the development of spin valve structures by IBM⁵. In order to optimise the performance of magnetic devices, many different material combinations have been studied to obtain optimum properties. Among them, the Ni_xFe_{1-x}/Cu (x≈0.81, in the following denoted as NiFe or Permalloy) system^{6,7} has attracted significant attention due to the low anisotropy in permalloy, the small saturation magnetic field and negligible hysteresis effects.

The performance of the magnetic devices based on the NiFe/Cu material combination must withstand different working conditions, such as elevated temperature and mechanical stress. Depending on the individual layer thickness, an irreversible degradation of the GMR occurs in NiFe/Cu multilayers at elevated temperatures⁸. However, little is known about the underlying individual deterioration mechanisms; one must perform comprehensive structural analysis in order to understand these mechanisms. For multilayers with 100 nm individual layer thicknesses and thicker, structural investigations by Auger electron spectroscopy (AES) and XRD indicated the onset of Ni diffusion into the Cu layers above a critical temperature⁹. The present investigation concerned the question of how the degradation of the magnetic properties is correlated with irreversible structural changes for *nanoscaled* GMR multilayers. To do this, we used a whole spectrum of methods, including X-ray diffraction and reflectometry, electron microscopy, measurements of the transport properties, magneto-optical Kerr effect (MOKE) and ferromagnetic resonance (FMR).

We employed DC magnetron sputtering to deposit [NiFe(1.7nm)+Cu(2.1nm)]₃₀ + NiFe(1.7nm) structures onto thermally oxidized Si (001) wafers in an Ar atmosphere of 60 mbar. The Cu layer thickness corresponded to the second afm maximum in the NiFe/Cu system. We

employed a Philips-XPert diffractometer with Cu-K α radiation to carry out the X-ray diffraction experiments and a standard 4-point probe set-up to measure the transport properties. We conducted anneals for one hour in vacuum (pressure 10^{-6} mbar) at different temperatures (T_{an}) in the range between 75 °C and 600 °C. After the anneals, we carried out structural, transport and magnetic measurements at room temperature.

The GMR effect in the as-deposited structures was on the level of 10%. Annealing up to 220 °C increased the GMR to 12%, however, additional annealing (at 300 °C or higher temperatures) resulted in a sharp decrease of the GMR signal (see Figure 1). We found that the total resistivity of the samples was nearly constant up to 220 °C and strongly increased after annealing at 300 °C. The saturation field behaved in a similar fashion, it was nearly constant (approximately 80 Oe) for annealing up to 220 °C and then increased dramatically to approximately 1500 Oe for higher annealing temperatures.

To correlate the changes in the GMR properties with possible changes of the interface properties, we used X-ray reflectometry (XRR). The XRR patterns clearly showed that the bilayer sequence is stable up to an annealing temperature of 300 °C¹⁰. For higher annealing temperatures we observed degradation of the layered structure, which completely intermixed after annealing at 600 °C. In contrast, the total thickness of the metallic structure (120 nm) was preserved even after annealing at 600 °C. From simulation calculations of the XRR curves, we calculated the mean roughness parameter (σ) of the interfaces to be 0.5 nm up to $T_{\text{an}} \sim 250$ °C, followed by a sharp increase to values above 1 nm at $T_{\text{an}} \sim 400$ °C.

The wide angle diffraction patterns (Fig. 2) indicated that a predominantly $\langle 111 \rangle$ texture was preserved during annealing. Even the texture sharpened during annealing, i.e., the half-width of the pole figure cuts decreased¹⁰. The grains possessed a typical vertical size of approximately 25 nm after deposition and showed a columnar structure. Annealing at $T_{\text{an}} > 220$ °C increased the

grain size significantly, causing a growth of a certain fraction of grains through the complete layer stack, as seen also in Co/Cu multilayers¹¹. Still more striking was the lateral grain growth, which lead to maximum grain sizes in the micrometer range and a mean size of 700 nm after the 400 °C anneal. This was measured using the electron back scattering diffraction (EBSD) technique in a SEM. AES measurements of NiFe(100 nm)/Cu(200 nm) stacks showed the onset of interdiffusion at 250 °C, when Ni atoms preferentially diffused into the Cu layers⁹. The X-ray and AES measurements indicated that anneals above 250 °C strongly affected the multilayer structure. The strong increase in grain size and roughness parameters, in combination with preferential interdiffusion of Ni into Cu, finally resulted in a complete destruction of the layered NiFe/Cu structure. More detailed results of the XRR and of the texture studies are presented elsewhere¹⁰.

We performed magnetic measurements at room temperature as a function of the angle within the film plane. Figure 3 shows hysteresis loops measured by MOKE for different orientations. The change in the shape of the hysteresis loops for different angles (only two shown) clearly indicated the presence of a uniaxial in-plane anisotropy. Measurements along one direction yielded "S"-shaped loops (hard axis) and for measurements 90° off this axis we observed hysteresis loops that are typical for afm coupled systems. The value of the saturation along the easy axis (approximately 75 Oe) was in agreement with the GMR measurement for the as-deposited samples. The shape of the hysteresis loop along the easy axis was typical for the presence of both biquadratic and bilinear coupling contributions. Two critical fields were seen. The first represented the initial deviation from the collinear (saturated) configuration and the second represented a field at which the neighbouring layers became antiparallel. From these measurements, we determined the strength of both, the bilinear (J_1) and the biquadratic (J_2) exchange coupling¹² ($J_1 = -0.0012 \text{ erg/cm}^2$ and $J_2 = 0.001 \text{ erg/cm}^2$) and the strength of the small

uniaxial in-plane anisotropy ($H_u = 20$ Oe). The strength of the exchange coupling was very small and nearly constant up to $T_{an} \sim 260$ °C. At this temperature the hysteresis loops showed visible deformations with respect to the as-grown data. Instead of two well-defined critical fields we observed a few smaller jumps that indicated different switching fields in different regions of the sample.

The FMR measurements confirmed the presence of the uniaxial in-plane anisotropy and resulted in values that were similar to those determined by the fitting of the MOKE data¹³. The uniaxial in-plane anisotropy (20 ± 5 Oe) was nearly constant up to $T_{an} \sim 260$ °C. Then we observed a significant increase to 60 Oe at $T_{an} \sim 330$ °C. The $4\pi M_{eff}$ behaved in a similar fashion. Up to 260 °C, the value of $4\pi M_{eff}$ was nearly constant at 6.5 kG; this was followed by a rapid decrease to 5.6 kG at 330 °C.

The measurements of the FMR linewidth revealed an interesting behaviour. The line width was nearly constant ($\Delta H = 120$ Oe) up to $T_{an} \sim 150$ °C. After annealing at 180 °C the linewidth decreased to 75 Oe. However, after annealing at higher temperatures we observed increased values of the linewidth, which reached a maximum value of 250 Oe after annealing at 330 °C – see Figure 4. The observed minimum of the linewidth corresponded well to the increased GMR at this annealing temperature--a finding that was discussed in more detail by Hecker, et. al.¹⁰.

All our experimental results pointed to a temperature of approximately 250 °C, at which we found critical changes in our NiFe/Cu structures. We observed two tendencies. First, for annealing below 250 °C, there was an increase of the grain size and a reduction of defects, as inferred from the XRD experiments. This tendency corresponded to the observed decrease in FMR linewidth, which indicated increased magnetic homogeneity of our layers near an annealing temperature of 200 °C. Second, for annealing temperatures above 250 °C, we observed a

significant intermixing between Ni and Cu that degraded the structural and magnetic integrity of the NiFe/Cu layers. The multilayer structure became less defined, and as a result we observed a significant degradation of the GMR effect. In conclusion, it is the alloying tendency of Ni and Cu above 250 °C that determined the decay of the GMR and the change in the magnetic properties of our NiFe/Cu multilayers.

Acknowledgement

We acknowledge financial support from DAAD (315/PPP), the National Science Foundation (INT-9815225 and DMR-9970789), US Army Research Office (DAAG55-98-0294 and DAAD19-00-1-0054) and the SFB422.

Figure Captions:

- Fig. 1 Maximum resistance at zero magnetic field (R_{max}), saturation resistance (R_{sat}) and GMR versus annealing temperature
- Fig. 2 Wide angle X-ray diffraction patterns showing the zeroth order {111} and {200} reflection of the NiFe/Cu multilayers
- Fig. 3 The hysteresis loops measured along different axes (easy and hard)
- Fig. 4 FMR linewidth as a function of annealing temperature, measured at 24 GHz

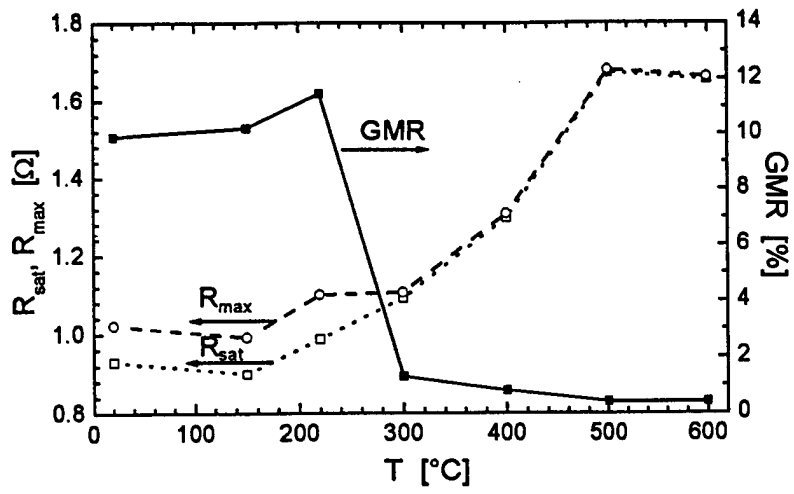


Figure 1 (M. Hecker et al., Journal of Applied Physics)

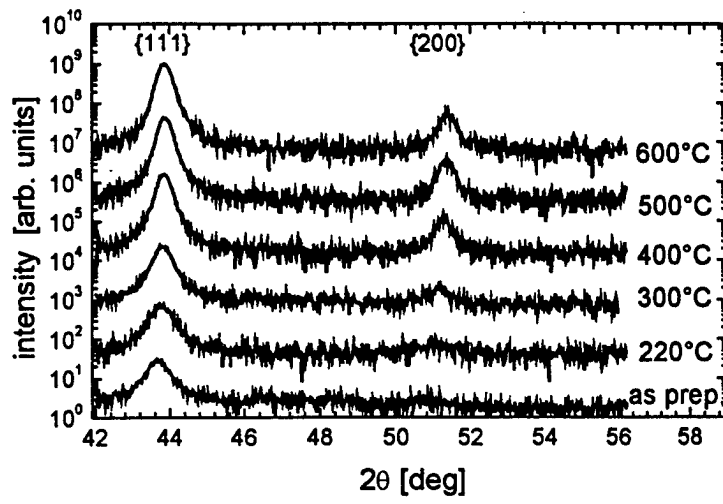


Figure 2 (M. Hecker et al., Journal of Applied Physics)

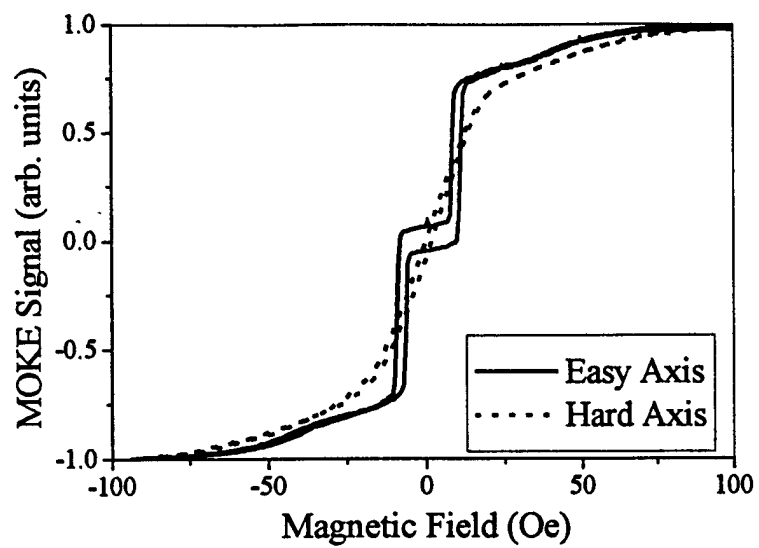


Figure 3 (M. Hecker et al., Journal of Applied Physics)

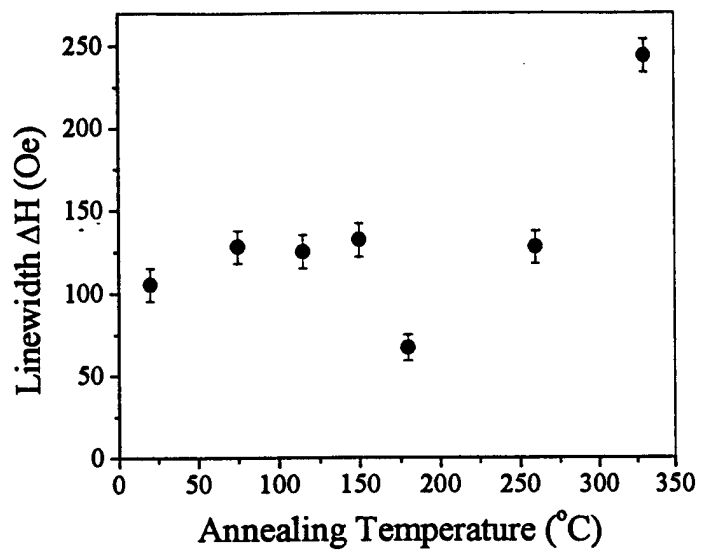


Figure 4 (M. Hecker et al., Journal of Applied Physics)

References:

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- ⁶ S.S. Parkin, *Appl. Phys. Lett.*, **60**, 512 (1992).
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- ⁸ L.van Loyen, D. Elefant, D. Tietjen, C.M. Schneider, M. Hecker, J. Thomas; *J. Appl. Phys.* **87**, 4852 (2000).
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