

PEEM(光電子顕微鏡)実験の現状

東大 物性研 木下豊彦

協力者

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東大工 小野寛太、木綿秀行、木原隆幸、尾嶋正治

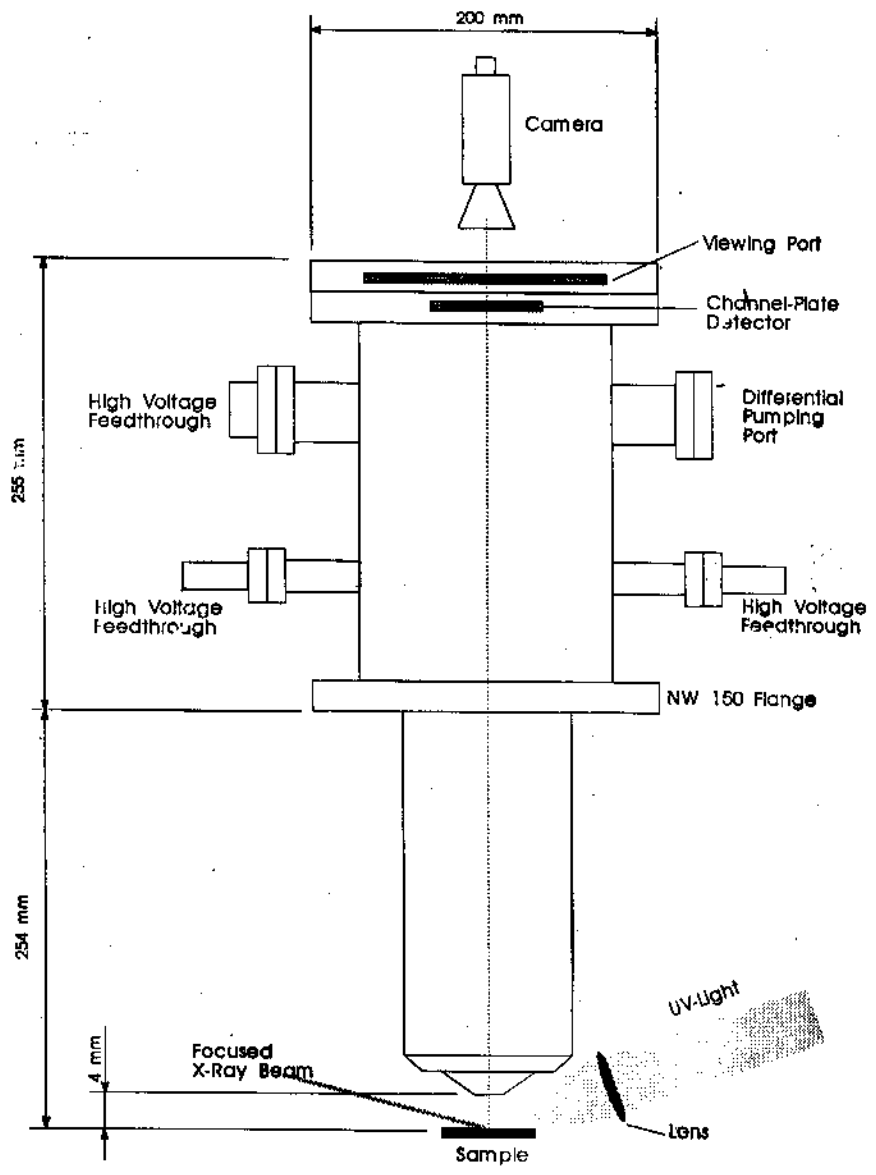
NTT 横尾篤

MPI-HALLE F. U. Hillebrecht

PEEMの特徴(磁気構造観察例)、課題、今後



STAIB
INSTRUMENTE



PEEM - 150 : SCHEMATIC VIEW

entscheidend die Elektronenausbeute, d.h. die pro einfallendem Photon emittierte Anzahl von Elektronen. Die Werte für die Austrittsarbeit der meisten Metalle beispielsweise liegen in einem solchen Energiebereich, daß nur durch energiereiches Ultraviolett-Licht Elektronen ausgelöst werden können. Zu diesem Zweck stehen eine Vielzahl von intensiven Lichtquellen zur Verfügung, wie beispielsweise Quecksilberdampf-Kurzbogenlampen, Xenon-Kurzbogenlampen und Deuterium-Niederdruck-

Vergrößerungsbereich von 100-fach bis 1000-fach abzudecken. Im Gegensatz zu optischen Systemen läßt sich jedoch der Vergrößerungsfaktor ohne mechanische Verschiebung, also lediglich durch Variation der erregenden Linsenspannung einstellen. Eine elektrostatische Linse besteht dabei aus mehreren isoliert aufgebauten Elektroden, die sich auf unterschiedlichen elektrischem Potential befinden.

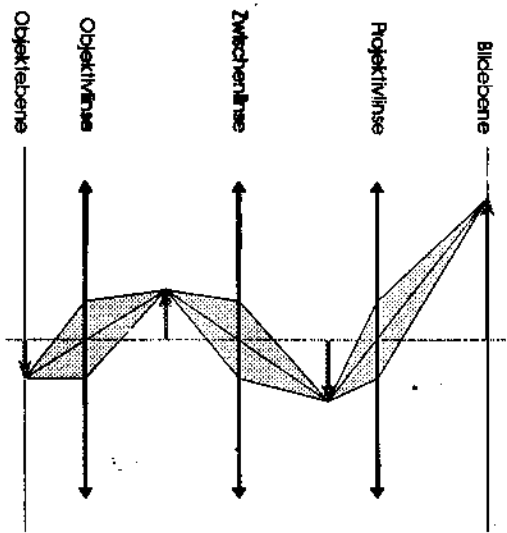


Abb.: Vergrößernde Abbildung durch dreistufige Optik

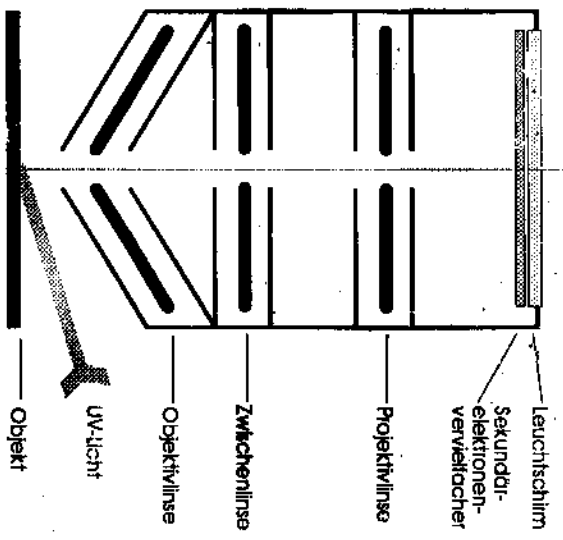


Abb.: Schematischer Aufbau eines Photo-Elektronen-Emissions-Mikroskops

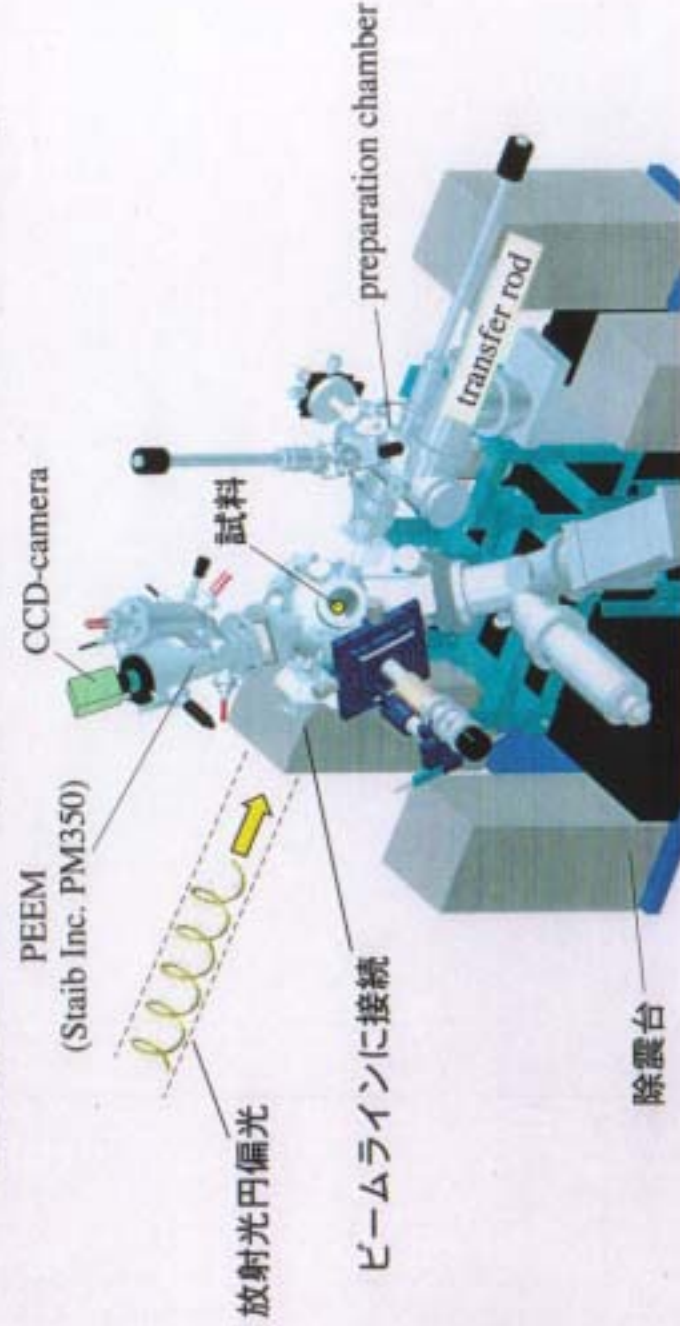
システムの特徴

- ・ 超高真空 (1×10^{-10} Torr)
- ・ 高空間分解能 ……除振
- ・ KEK-PFにて MCD・MLD(I)・MLD(II)配置の実現
……分析系の回転



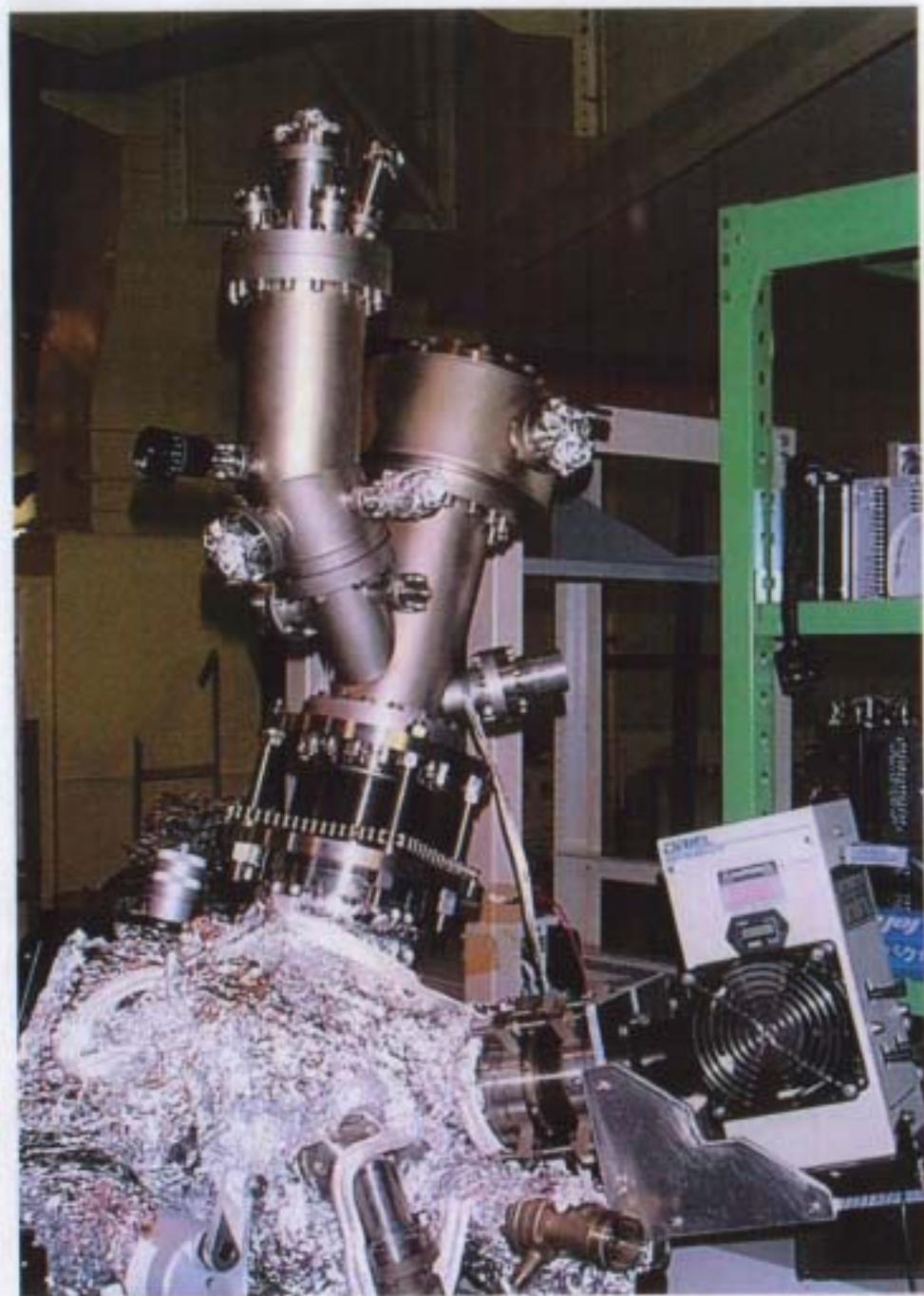
実験(4) 実験装置

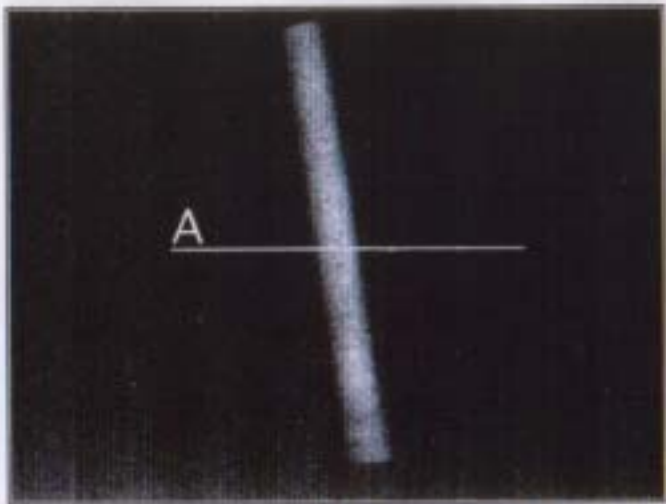
The experiments were performed at BL-11A of Photon Factory, KEK.



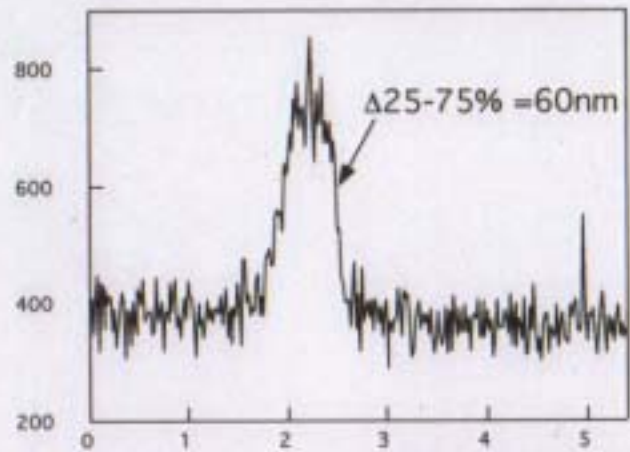
The design of SR-PEEM system

PEEM





Ni Wire ($1\mu\text{m}$)
(Hg lamp , 3sec)



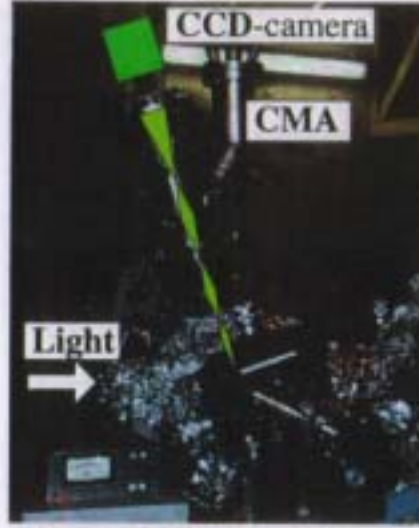
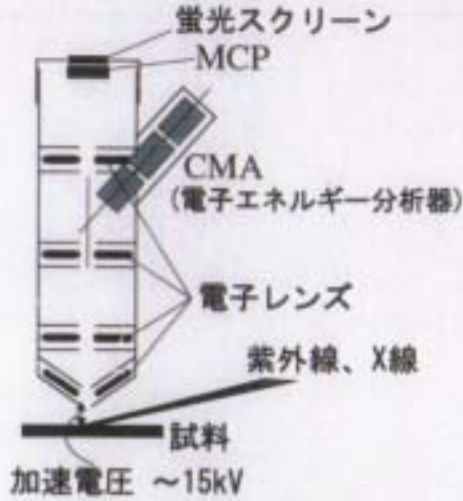
Line profile

SR の場合.

$\Delta R < 200\text{nm}$

振動. 色収差

放射光光電子顕微鏡の原理と特徴 (SR-PEEM)



二次電子放出強度が元素内殻吸収強度に比例
→ 吸収強度の二次元マッピング

放射光

エネルギー可変性

→ 吸収スペクトルの二次元マッピング

円偏光・直線偏光

→ 磁気二色性イメージング

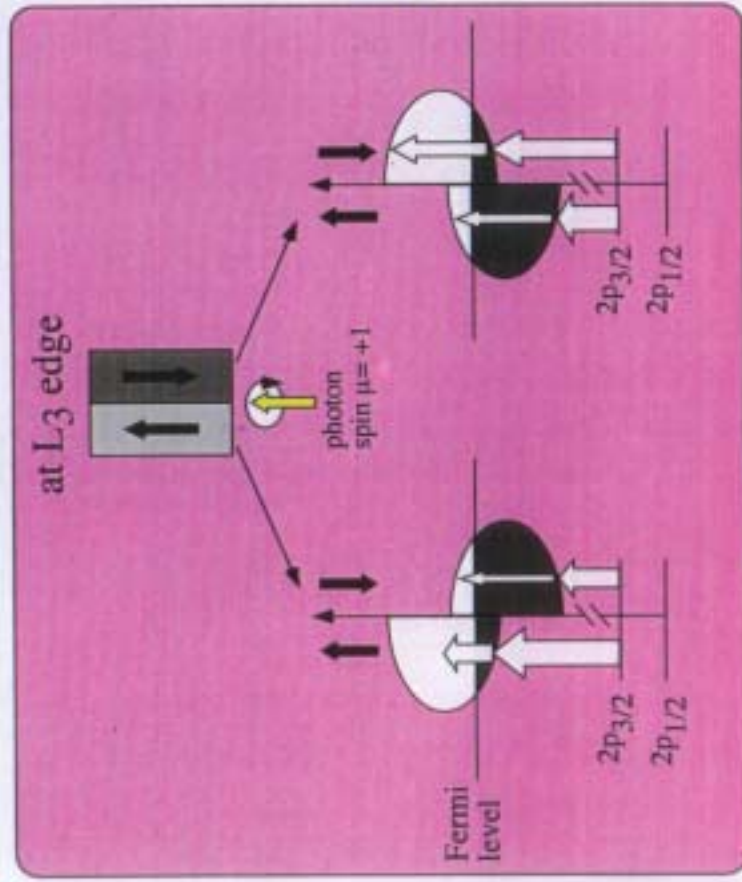
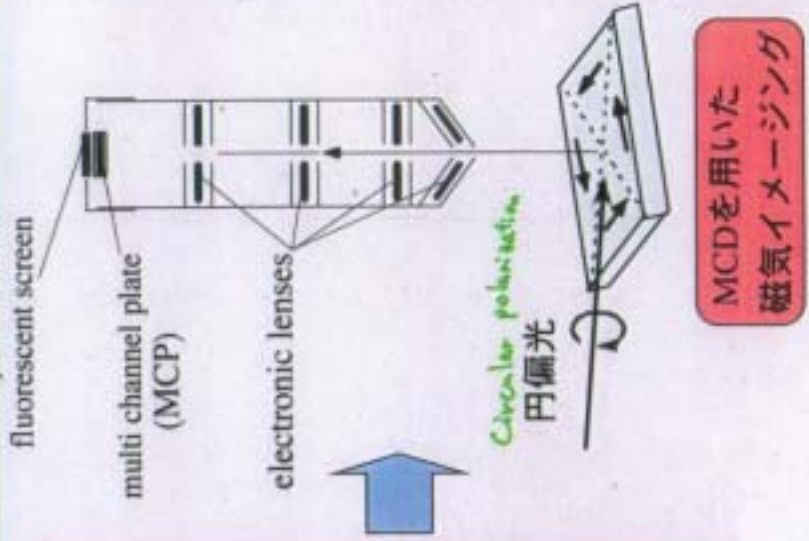
MCD (Magnetic circular dichroism)

MLD (Magnetic linear dichroism)

→ 磁気二色性スペクトルの二次元マッピング

軌道総和則 → **軌道磁気モーメント**
スピン総和則 → **スピン磁気モーメント** の観測

実験(3) 実験の原理



X-MCDの原理

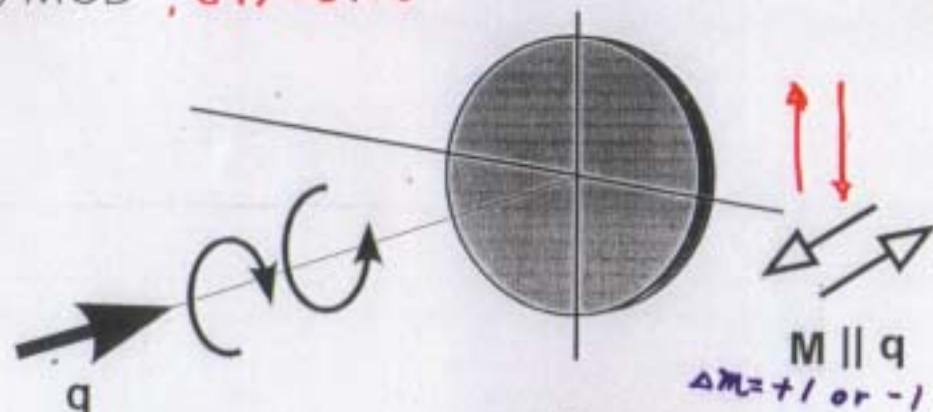
Principle of X-MCD

磁気二色性測定法の配置と特徴

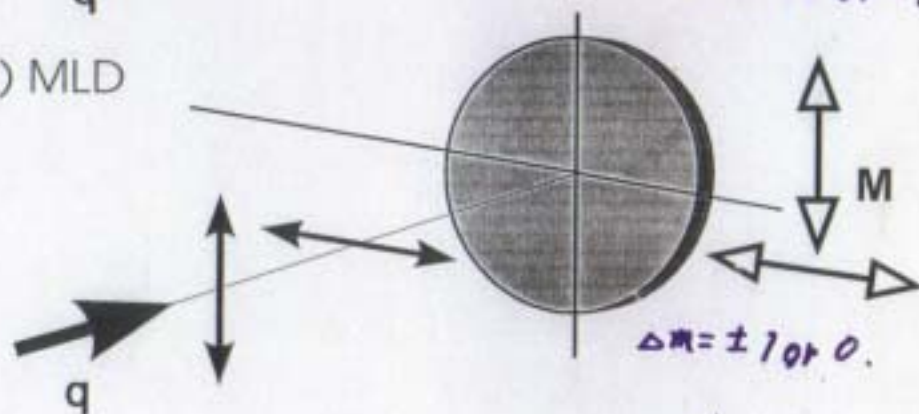
手法	配置	特徴
MCD		<p>シグナルは $\langle \sigma \cdot M \rangle$ に比例</p> <p>強磁性体の観測</p>
MLD (I)		<p>シグナルは $\langle (E \cdot M)^2 \rangle$ に依存</p> <p>反強磁性体にも適用可能 \rightarrow NiO^{※1}, LaFeO₃^{※2}</p>
MLD (II)		<p>シグナルは光入射角度に依存^{※3}</p> <p>強磁性体の観測 磁気二色性の偏光面依存性</p>

^{※1} D. Spanke *et al.*, Phys. Rev. B, 58, 5201 (1998).
^{※2} F. Nolting *et al.*, Nature, 405, 767 (2000).
^{※3} F.U. Hillebrecht *et al.*, Phys. Rev. Lett., 75, 2224 (1995)

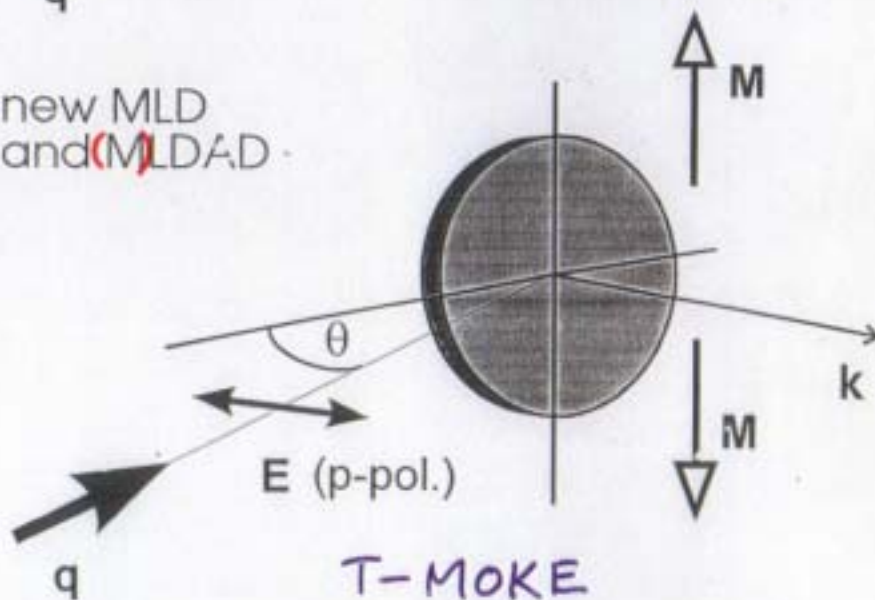
(a) MCD, (M)CDAD



(b) MLD



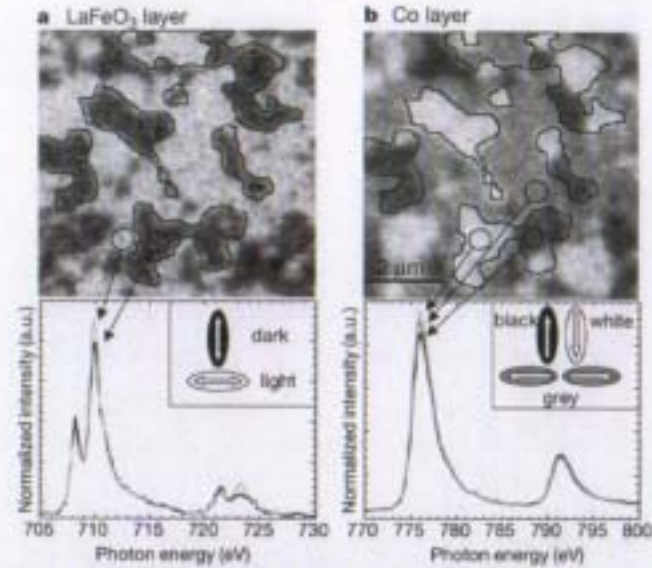
(c) new MLD and (M)LDAD



Advantages of PEEM($\leftarrow \rightarrow$ MFM)

Spin STM

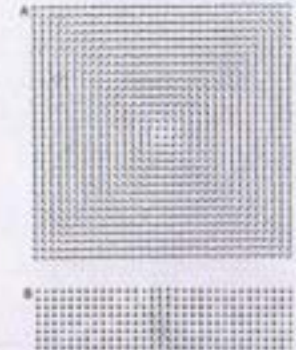
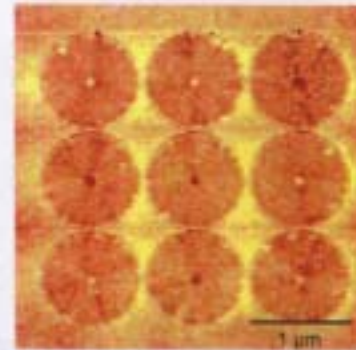
1. Element specific
2. Observation of in plane magnetization ... (Spin STM \rightarrow 0 K)
3. Not destroy magnetic structures
4. Possibility of real time observation
5. Possibility of depth profile?



Direct observation of the alignment of ferromagnetic spins by antiferromagnetic spins

F. Nolting, A. Scholl, J. Stöhr, J. W. Seo, J. Fompeyrine, H. Slegwart, J.-P. Locquet, S. Anders, J. Lüning, E. E. Fullerton, M. F. Toney, M. R. Scheinfein and H. A. Padmore

NATURE, VOL405, 767



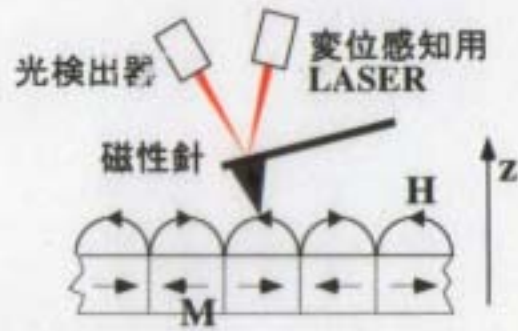
Magnetic vortex core observation in circular dots of permalloy

T. Shinjo, T. Okuno, R. Hassdorf, K. Shigeto and T. Ono

SCIENCE, VOL289, 930

研究の背景 (2)

MFM (磁気力顕微鏡) を用いた 微小磁性体磁気構造の研究



空間分解能
~数十nm

MFM ΔR ~ 10nm

シグナル



Signal
シグナル $\propto \frac{\partial^2 H_z}{\partial z^2}$



磁気モーメント (大きさ・方向) を
観測することが出来ない

Not sensitive to in-plane magnetization

No possibility to measure the moment
directly.

2. Domain Observation Techniques

PEEM	O	④	X	MFM	??
Method of domain observation	very good	direct	quantitative	quantitative	quantitative
Sensitivity to small variations in magnetization	very good	direct	quantitative	quantitative	quantitative
Resolution of the magnetization vector	direct	direct	quantitative	quantitative	quantitative
Altered magnetic field range	100 A/cm	very	very	1000 A/cm	1000 A/cm
Sample preparation quality requirements	moderate-low	high	moderate	high	very high
Necessary capital investment	low	moderate	high	high	very high

Fig. 2.57 Qualitative comparison between different domain observation methods

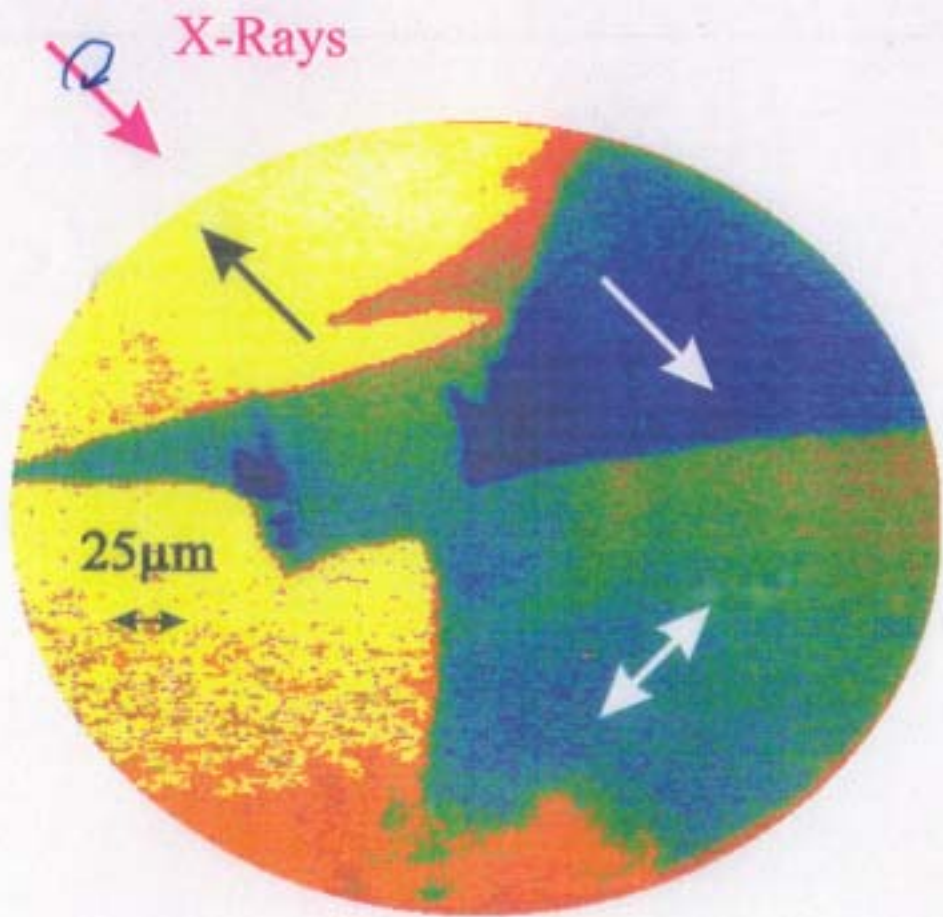


PEEM



Fig. 2.56 Comparison of different domain observation techniques. Indicated are the estimated limits of the properties and their approximate range, depending on the experimental conditions

ESRF HELIOS



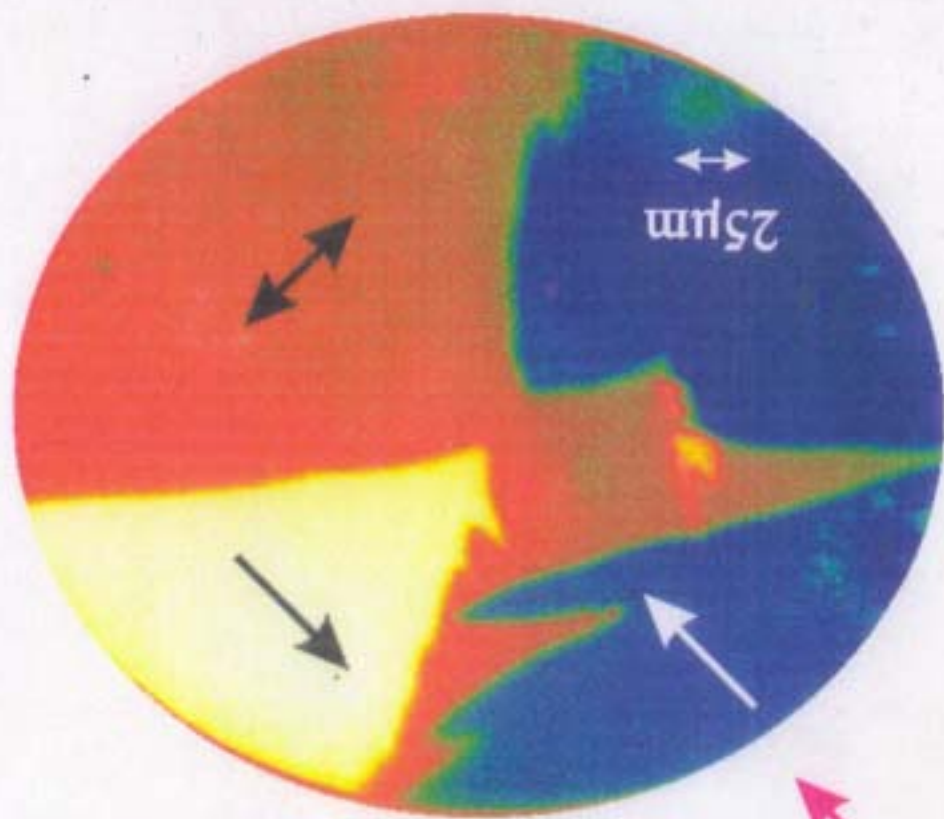
Antiferromagnetic

0.25 ML Mn/Fe/Ag Mn 2p MCD

D. Spasche et al.

Mn / Fe / Ag (001)
0.5 ML

Fe 2p MCD



X-Rays



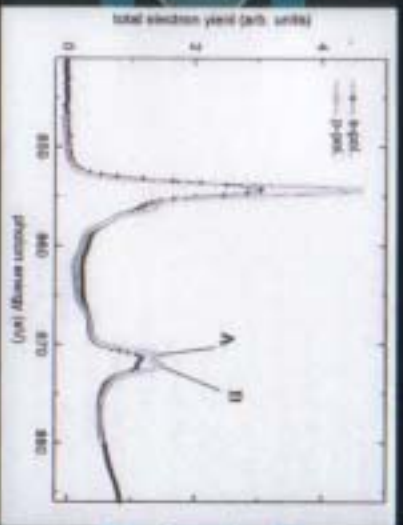
Mn L2吸収端によるイメージング



Ni L2吸収端によるイメージング

MnO/NiO

Domain imaging of anti-ferromagnetic material NiO by means of magnetic linear dichroism (MLD) method



Absorption spectrum at Ni L edges for s- and p-polarized light

$$I_{MLD} \propto 3\cos^2\theta - 1 : A/B, \text{ or } (A-B)/(A+B)$$

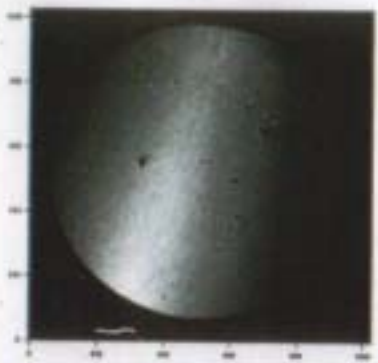
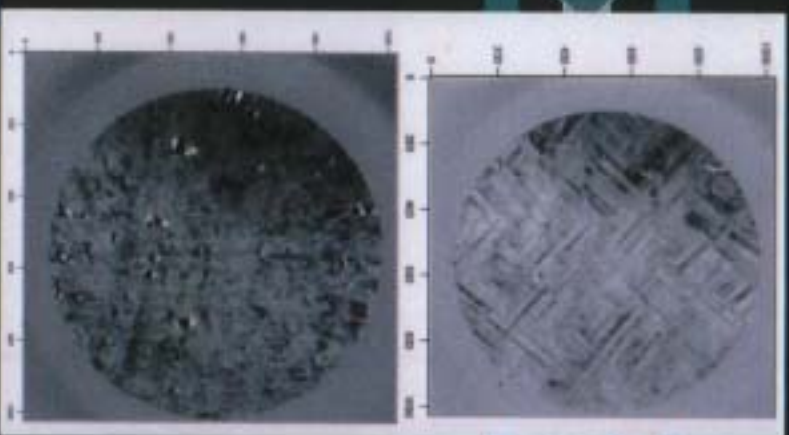
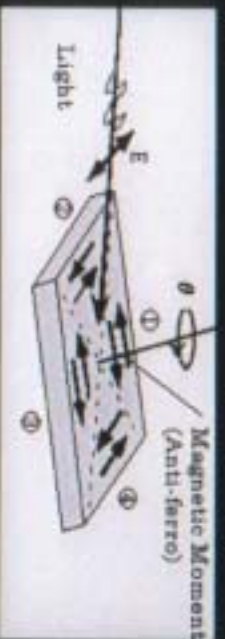


Fig. 1
the image at 871.2eV

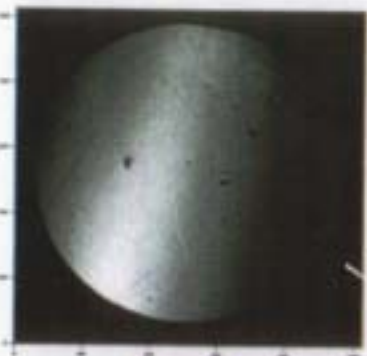


Fig. 2
the image at 872.2eV

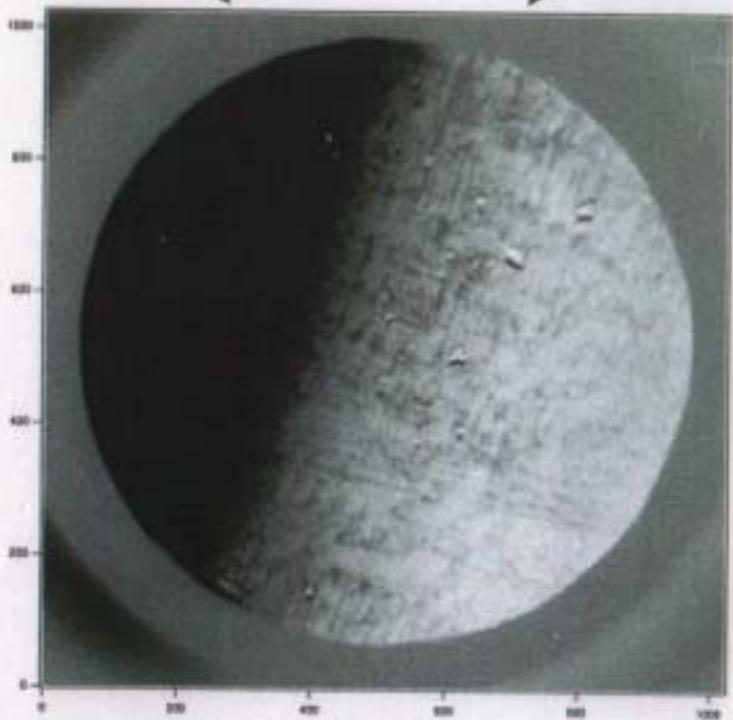
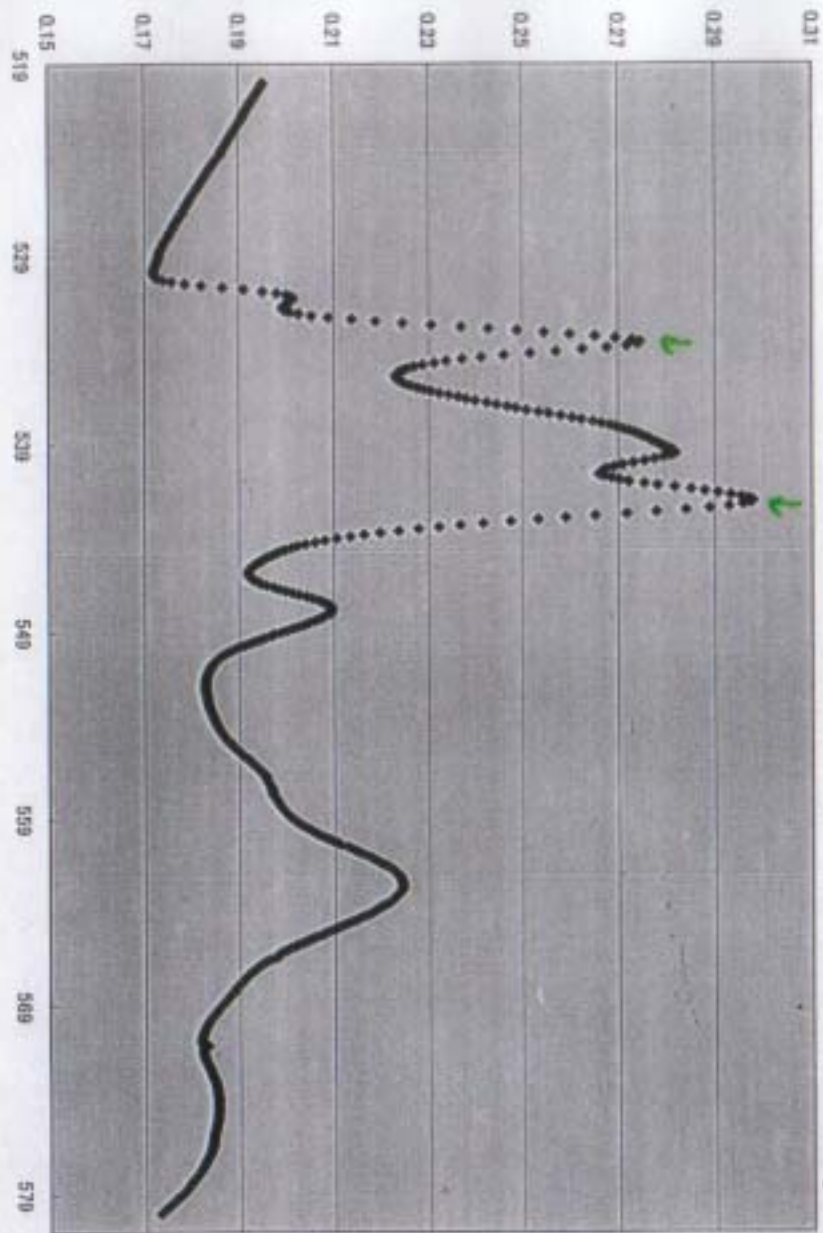


Fig. 3



• 系列 1

It is thought that the tetrahedral bo structure is a sign of covalent bonding, ionic bonds would tend as we shall see lat higher number of nearest neighbors (coord

SODIUM CHLORIDE STRUCTURE

The sodium chloride structure is show lattice is face-centered cubic, with a basis

NiO

⊙ O atom
 \Rightarrow No moment!

Origin of
 LD

Distortion
 or surface.

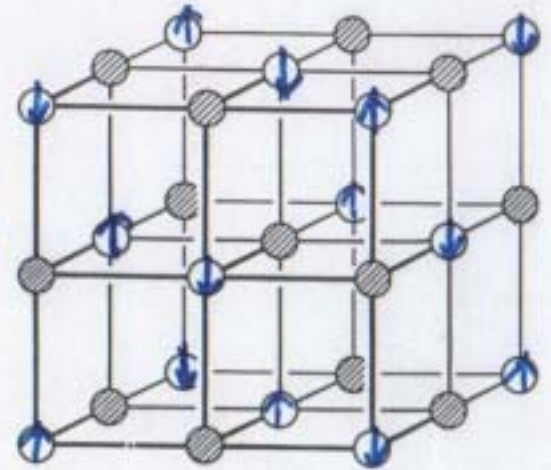


Fig. 1.29. The (a) sodium chloride and (b) ces atom separated by one-half the body diagon are four molecules in a unit cube, with at

$$\text{Na: } 000; \frac{1}{2} \frac{1}{2} 0; \frac{1}{2} 0 \frac{1}{2}$$

$$\text{Cl: } \frac{1}{2} \frac{1}{2} \frac{1}{2}; 0 0 \frac{1}{2}; 0 \frac{1}{2}$$

Each atom has as nearest neighbors six so that the coordination number is 6.

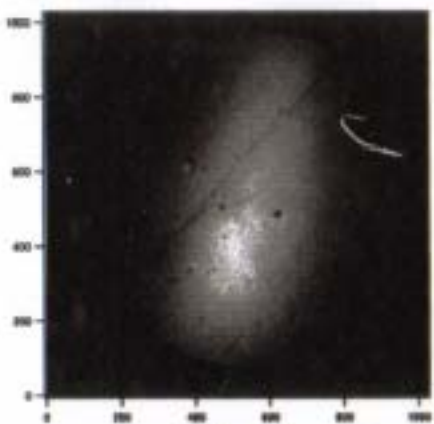


Fig. 3
the image at 542.1eV

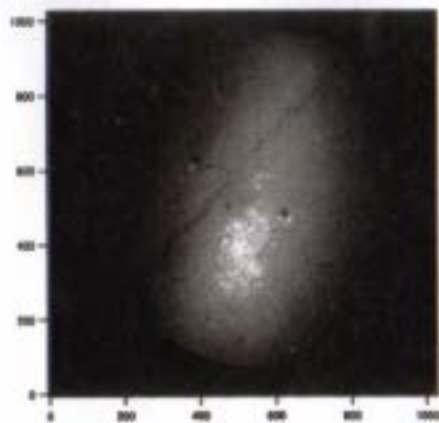


Fig. 4
the image at 533.7eV

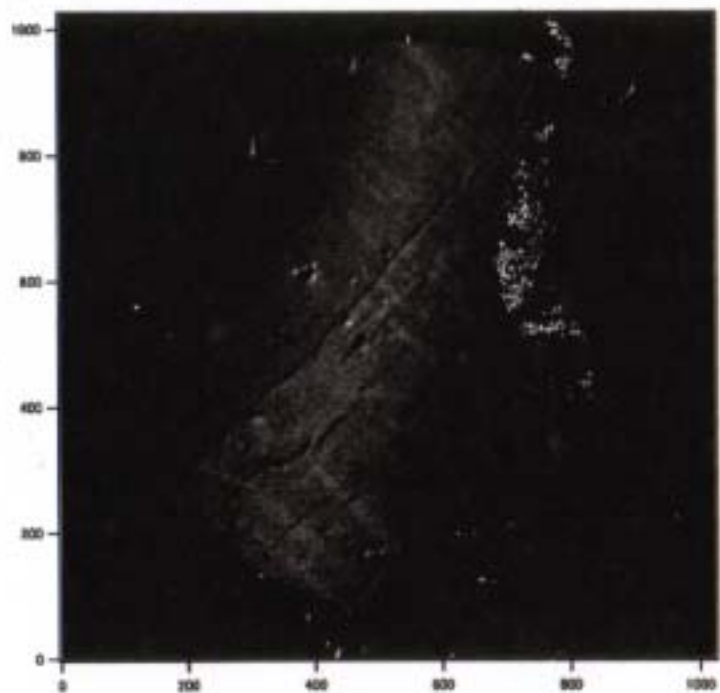
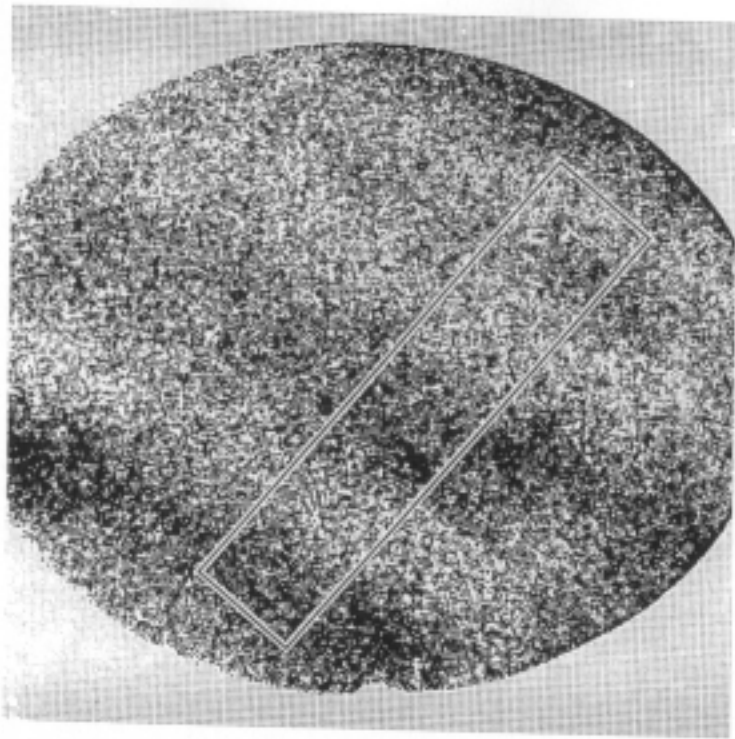
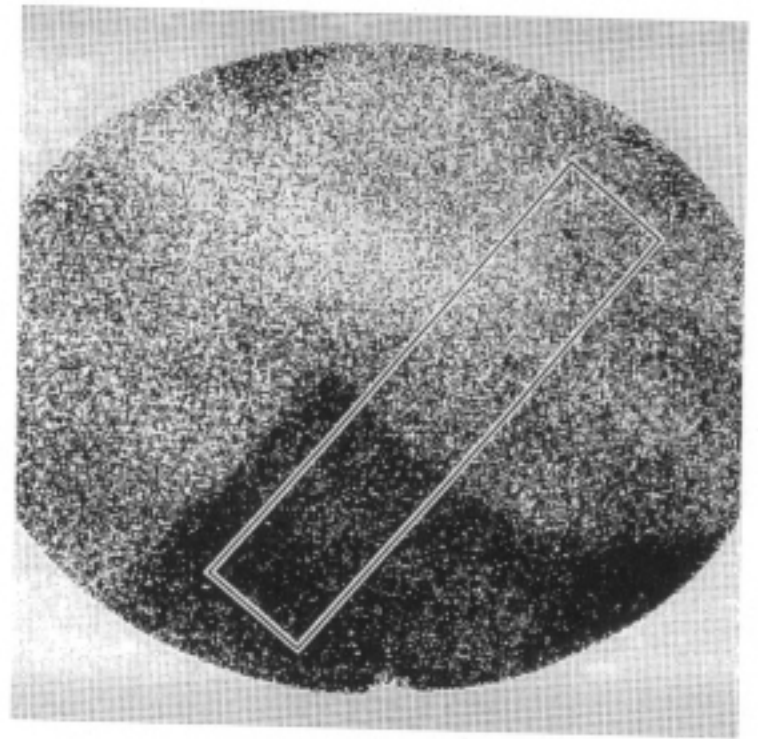


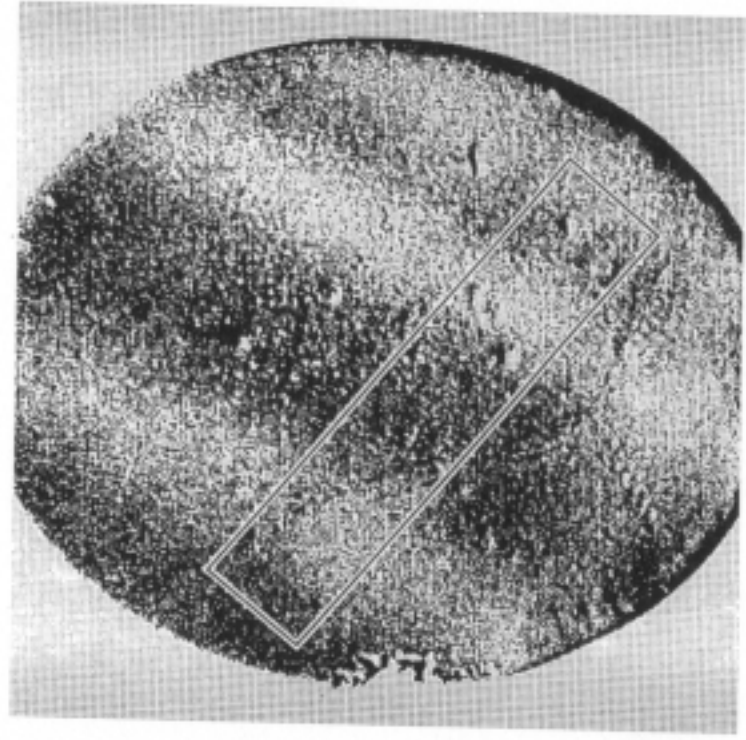
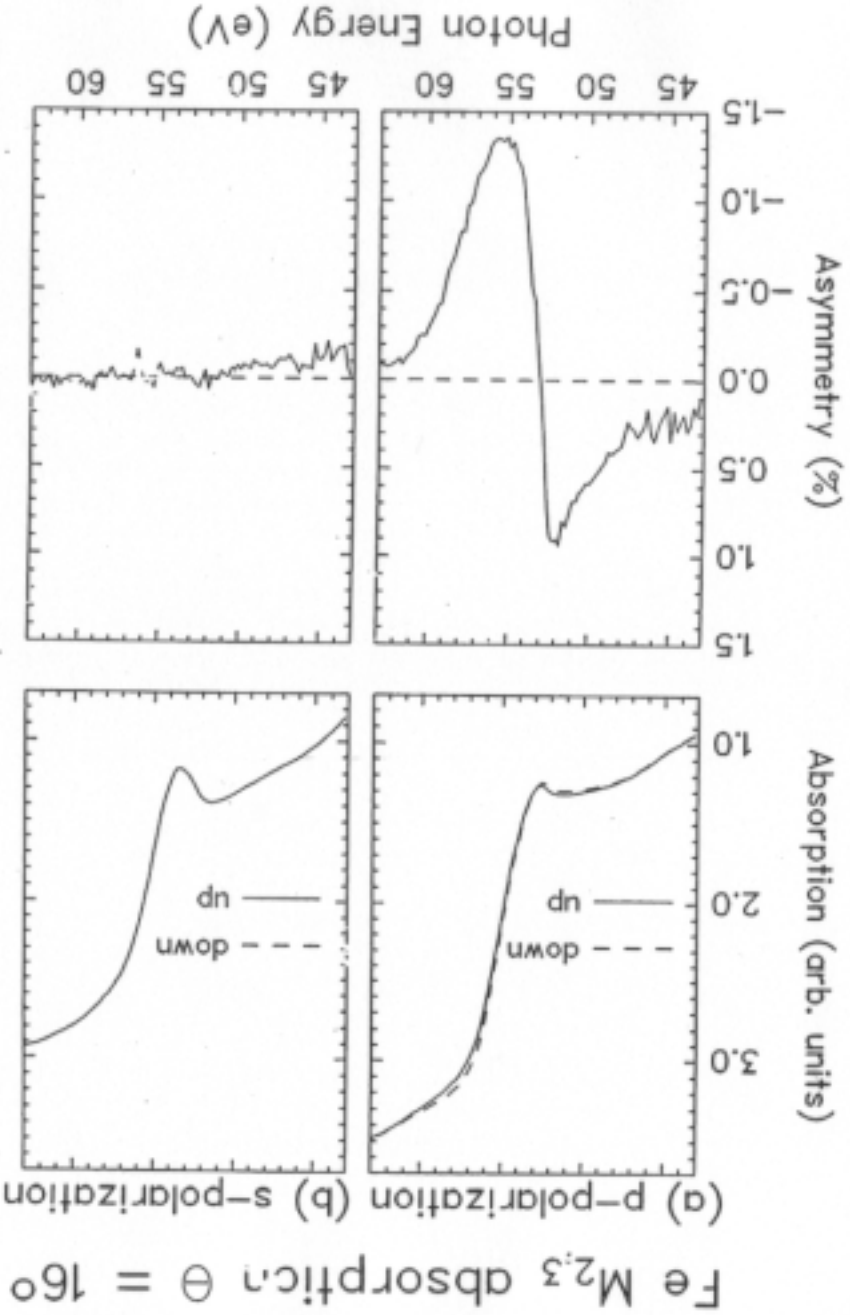
Fig. 5



Difference picture with s-polarized light



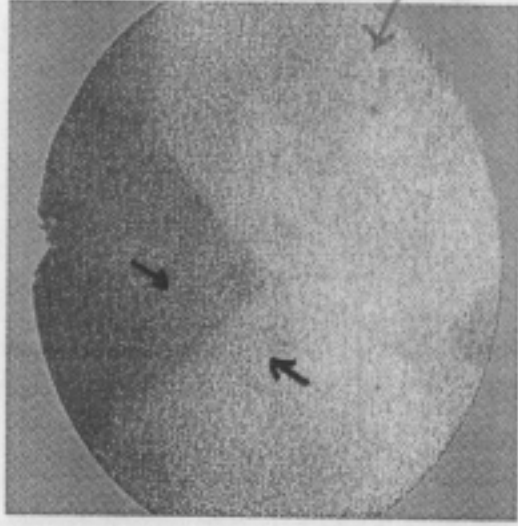
Difference picture with p-polarized light



Difference picture between σ^+ and σ^- light

$I(h\nu=52\text{eV}) - I(h\nu=55\text{eV})$

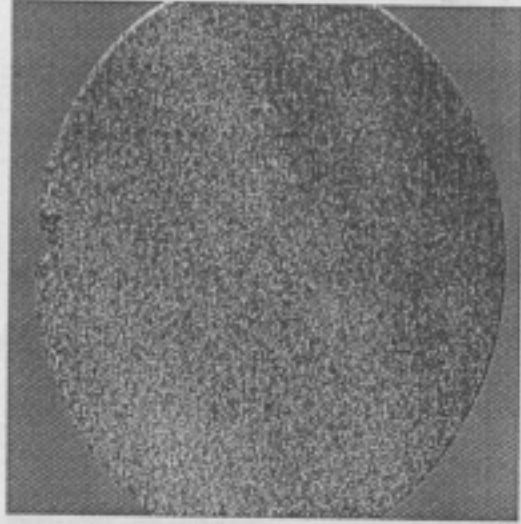
\Downarrow
d174_175.bmp



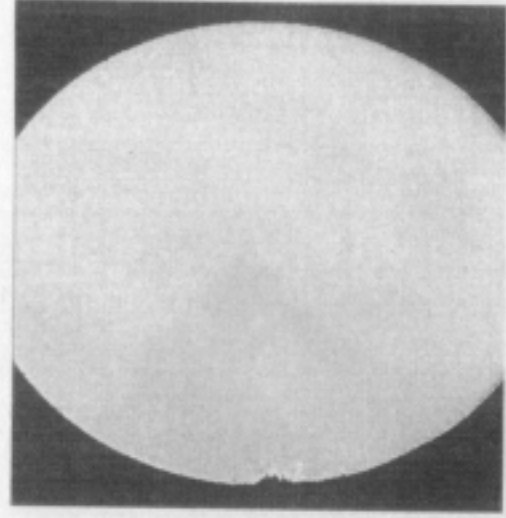
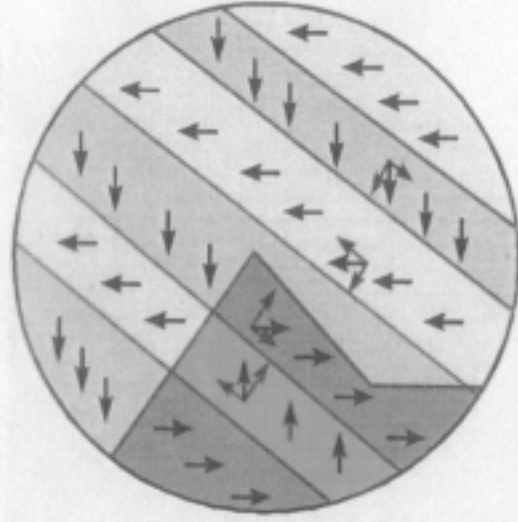
ϕ 0.2mm

4a

d188_189.bmp



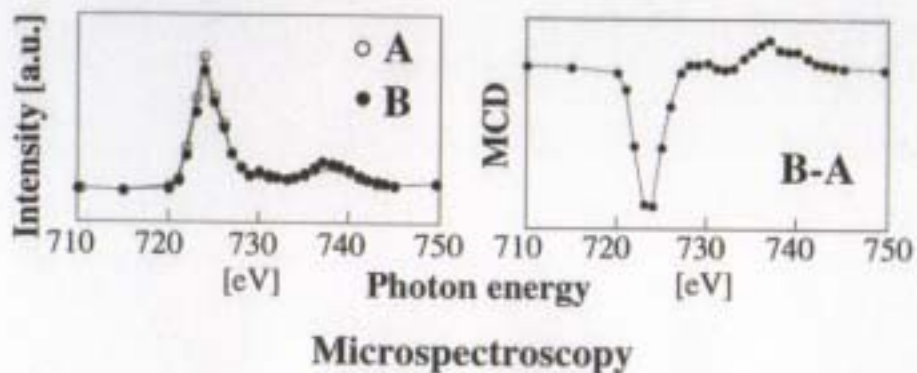
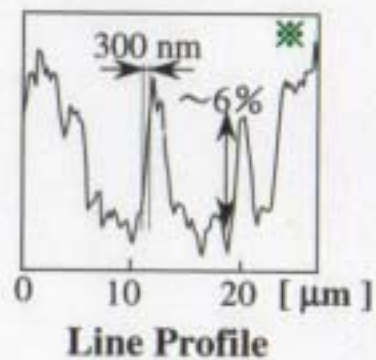
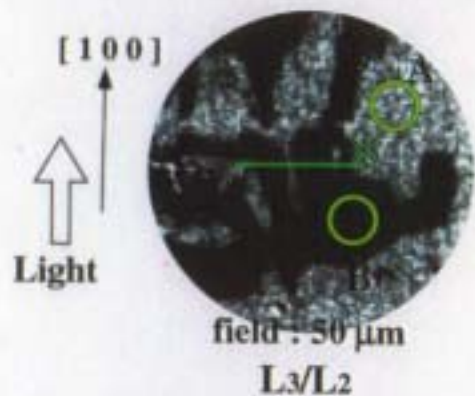
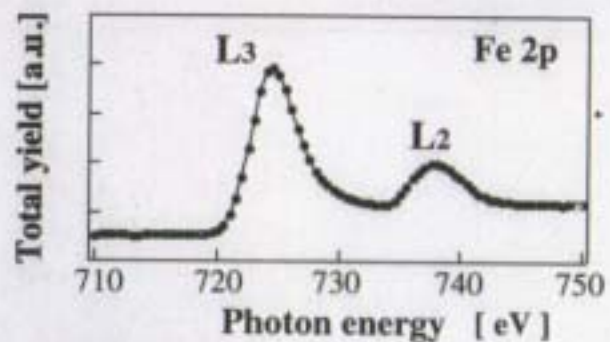
4b



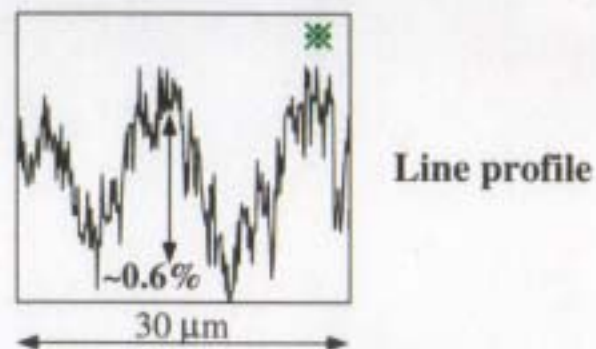
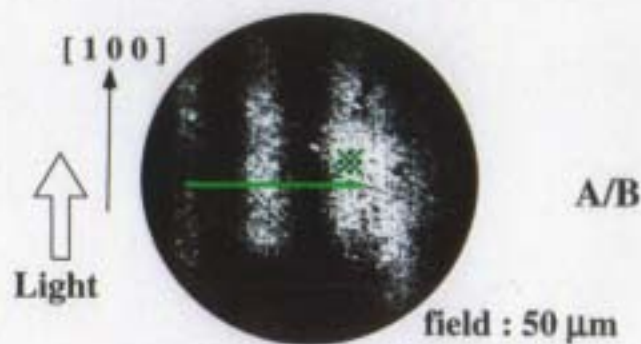
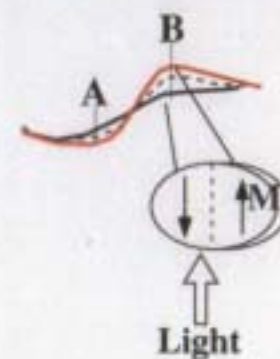
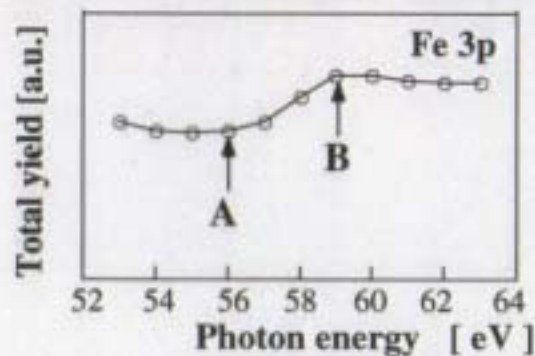
MLD + MCD

いろいろな偏光を組み合わせた
ことで磁化の向きが定まる。

Fe(100) 単結晶 L 吸収端磁気イメージ
(KEK-PF BL11A 円偏光)



Fe(100) 単結晶 M 吸収端磁気イメージ
(KEK-PF BL28A undulator 円偏光)

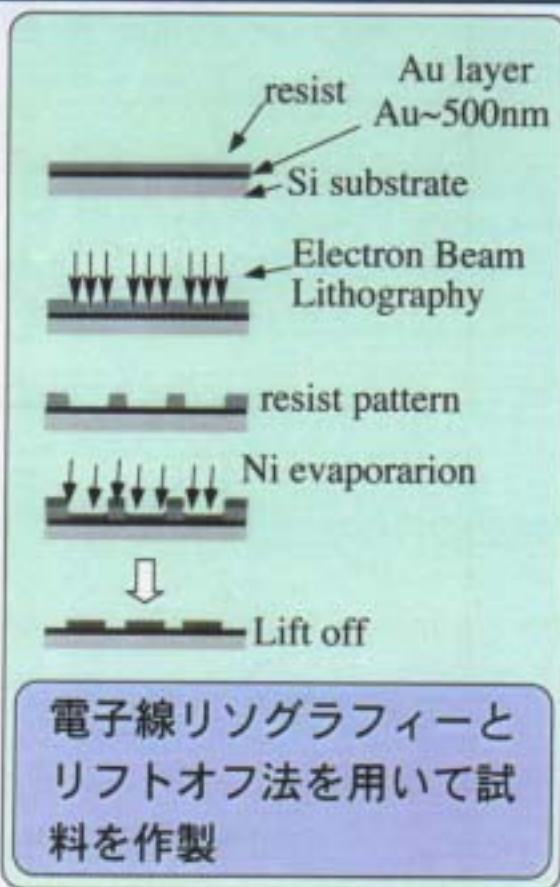


実験(1) 多結晶 Ni微小磁性体の作製



電子線露光装置

(試料提供: NTT物性科学基礎研究所, 横尾薫氏)



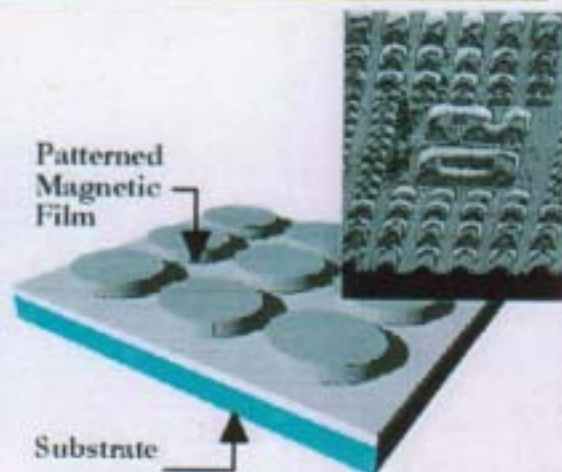
電子線リソグラフィーとリフトオフ法を用いて試料を作製

背景

・近年磁気記録媒体の記録密度は年率 100% と言う著しい速度で向上しているが、その記録密度は磁性薄膜の面内熱揺らぎにより1平方インチあたり100ギガビットが限界と考えられており、次世代高密度記録媒体の開発の必要性が高まっている。

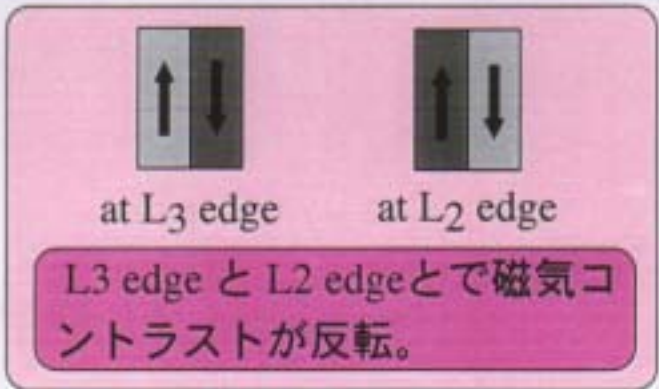
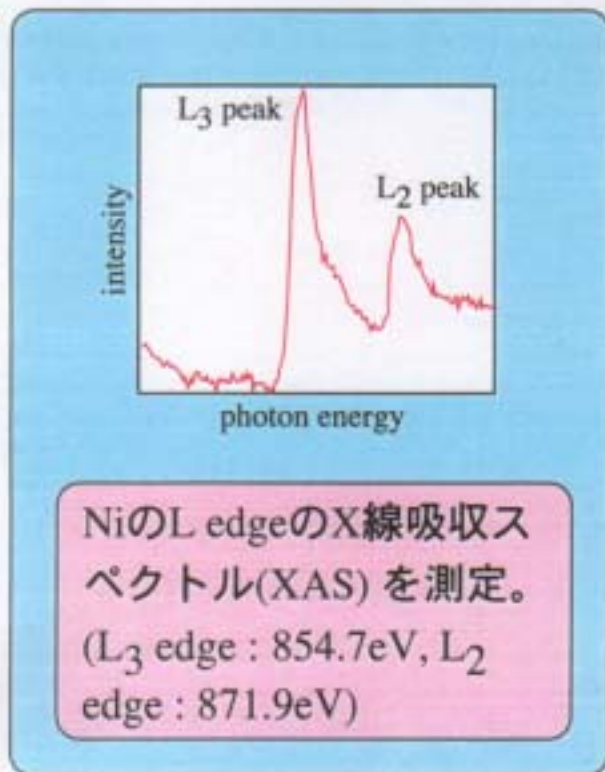


次世代高密度記録媒体として現在、形状や大きさを人工的にそろえた単一磁区の微粒子をアレイ状に並べ、この1微粒子を1ビットとして記録を行う「パターンドメディア」が期待されている。



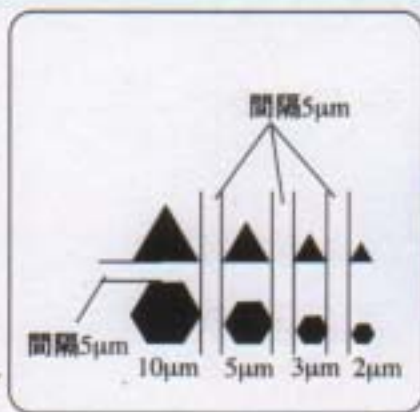
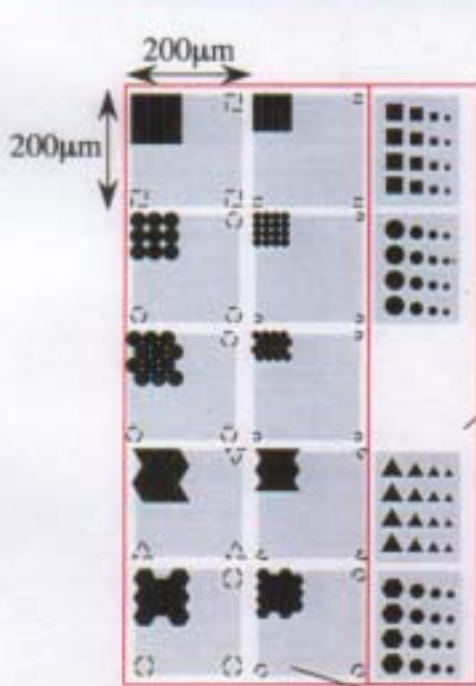
© IBM Almaden Research Center
<http://www.almaden.ibm.com/>

実験結果(1)



L₂ edge と L₃ edge の Photon energy で撮ったイメージを割り算することにより表面モフォロジーを打ち消し、磁気コントラストが強調された像を得ることが出来る。

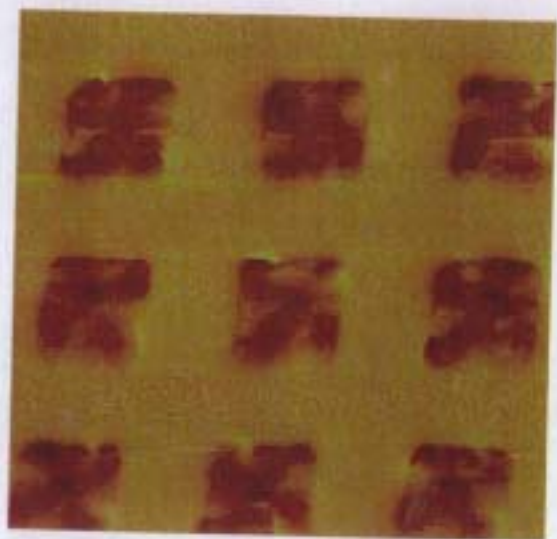
実験(2) 試料レイアウト



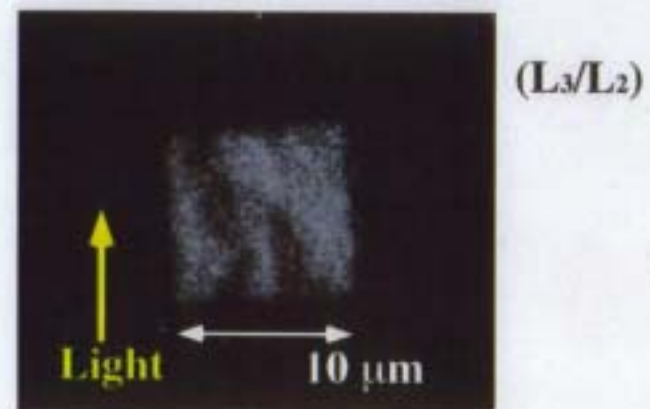
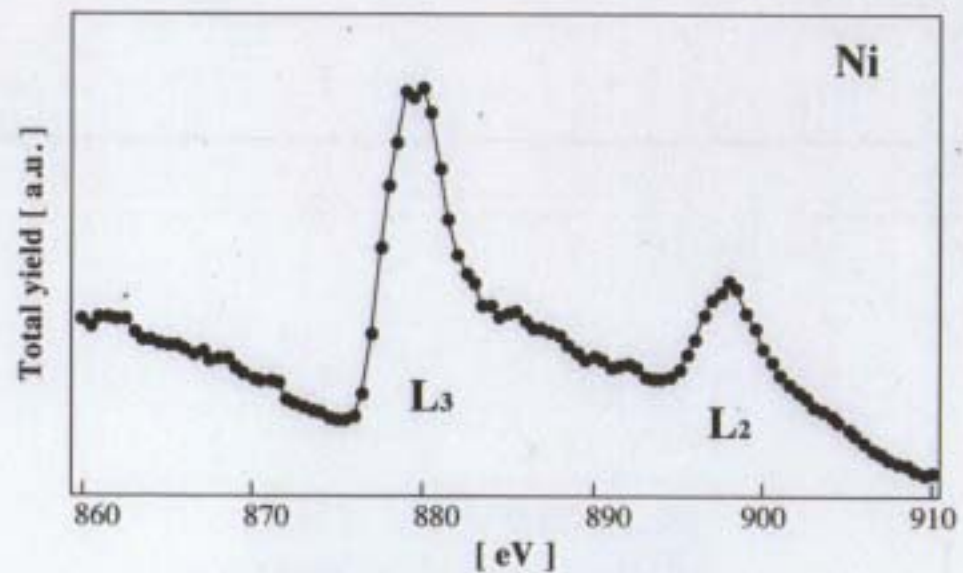
4種類の大きさを持つ4種類のドットを配列。



ドットアレイは各々0.1µmの間隔で配列



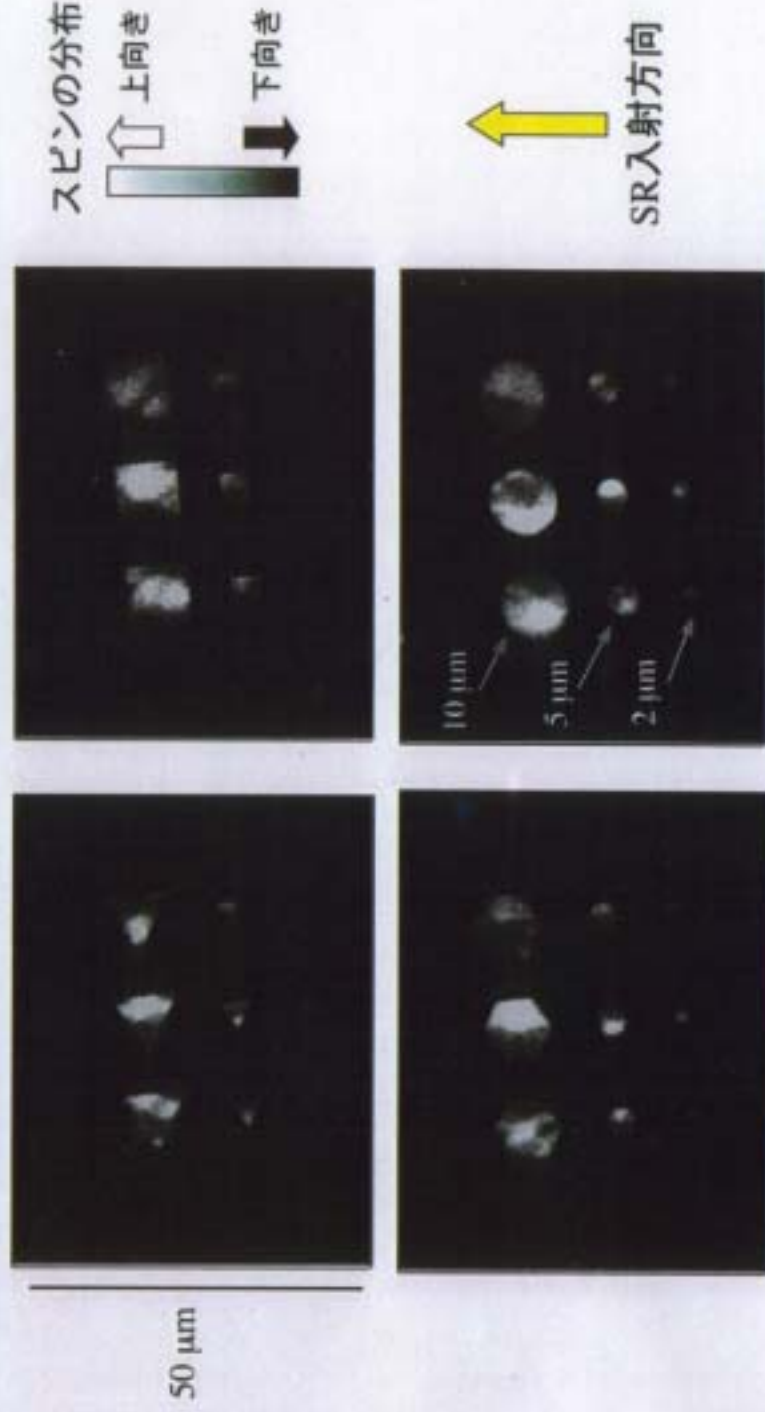
MFM image of Ni dots



Magnetic Image of Ni Dot

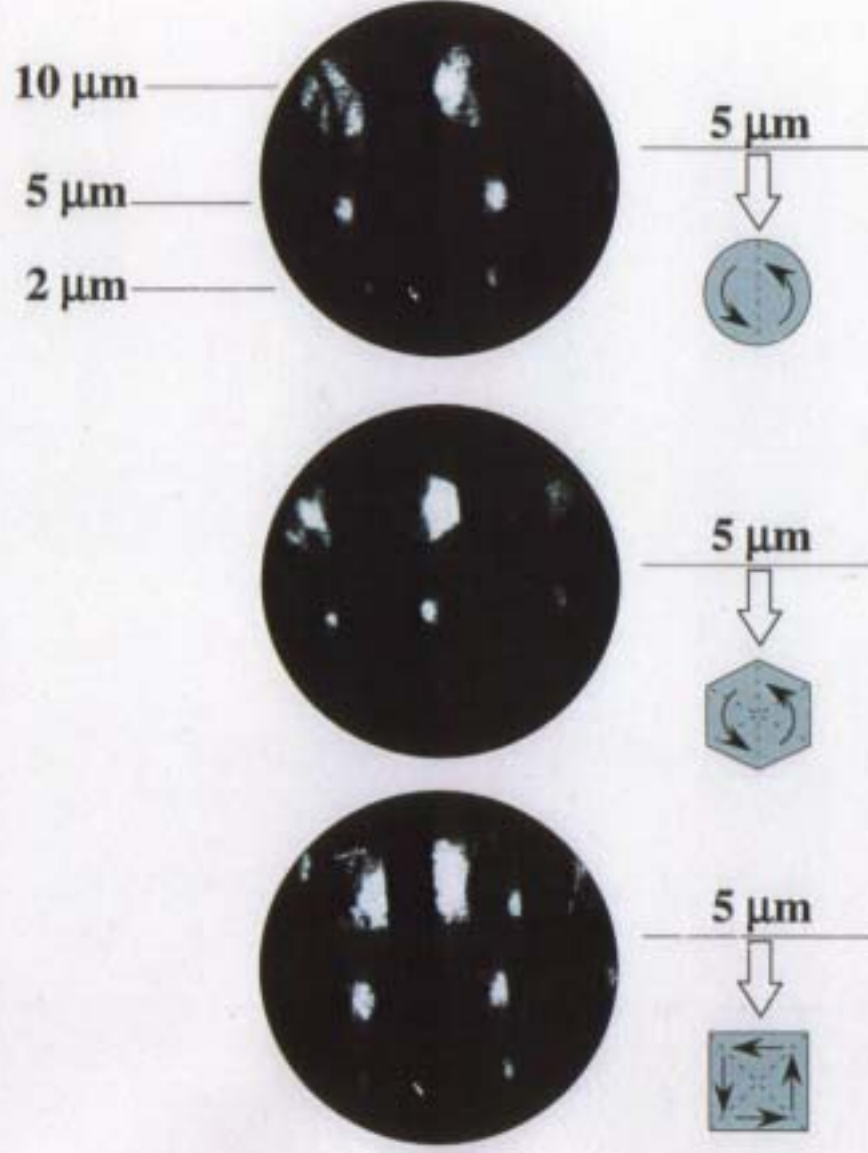
@KEK-PF BL-11A

実験結果(2)



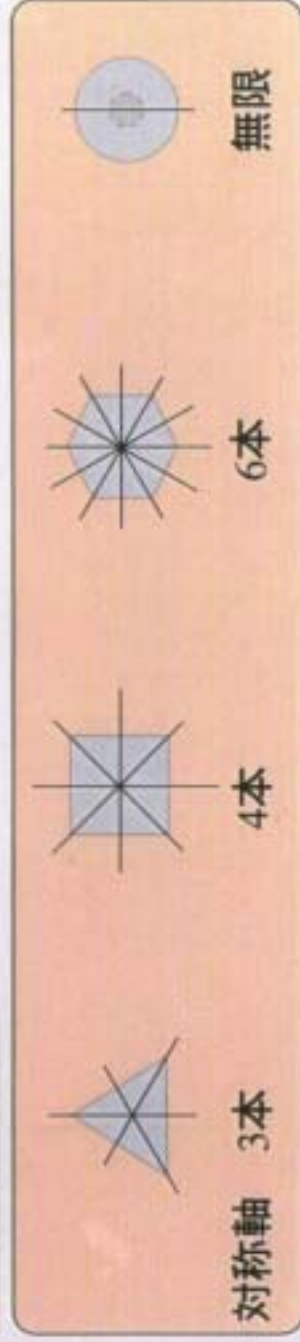
測定前に大気中で消磁器による消磁を行い観察。

Niドット磁気構造のサイズ依存性



考察(1)

形状による異方性



閉じた磁区構造を取れる



閉じた磁区構造を取れない

マイクロ磁気シミュレーション



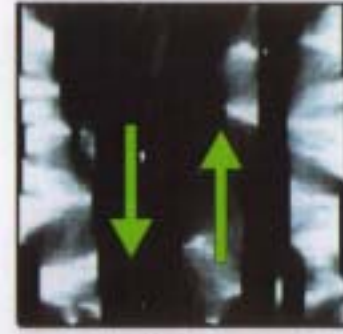
サイズ10 μm
(100×100セル分割)

Landau-Lifshitz equation

$$\frac{dM}{dt} = -\gamma M \times H_{eff} - \frac{\gamma\alpha}{M_s} M \times (M \times H_{eff})$$

γ : ジャイロ磁気定数
 α : ダンピング定数

多結晶Niドットのマイクロ磁気シミュレーション



10 μm



2 μm



1 μm



500 nm

サイズの減少と磁気構造の変化
(シミュレーション結果)

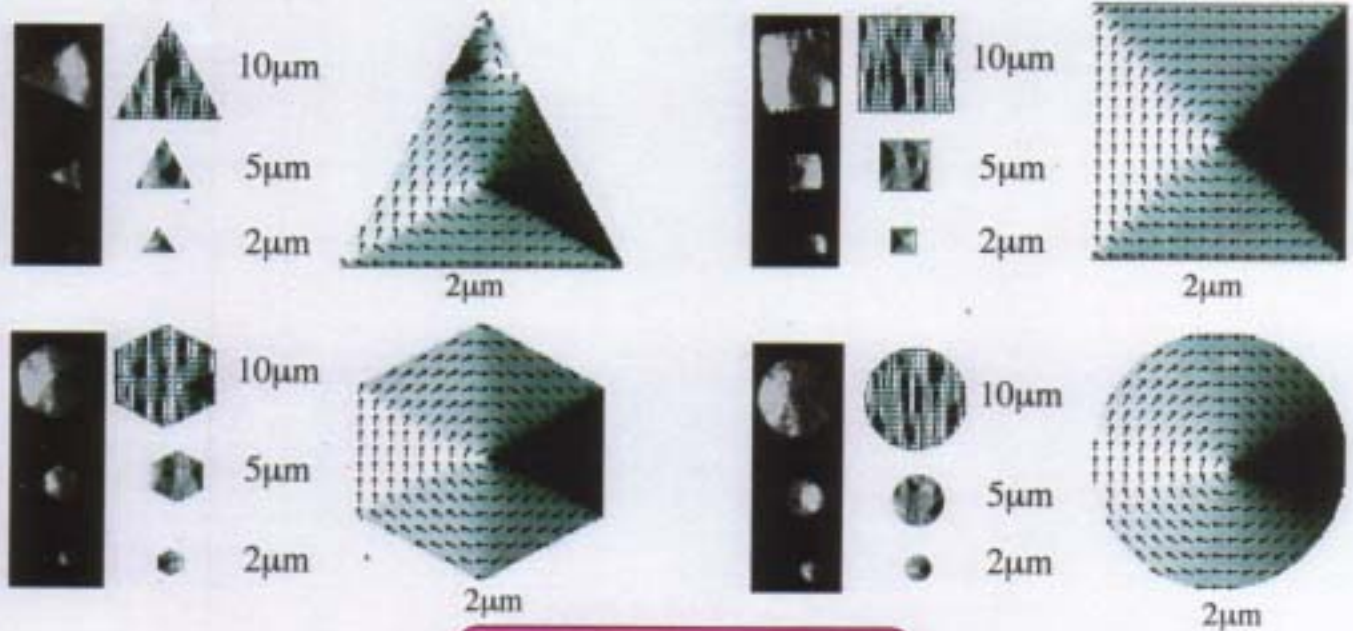


磁気イメージ

考察(2)

Landau-Lifshitz-Gilbert equation

$$dM/dt = -\gamma M \times H_{\text{eff}} - \gamma \alpha / M_s \cdot M \times (M \times H_{\text{eff}}) \quad (\alpha : \text{damping factor} \\ \gamma : \text{gyromagnetic factor})$$



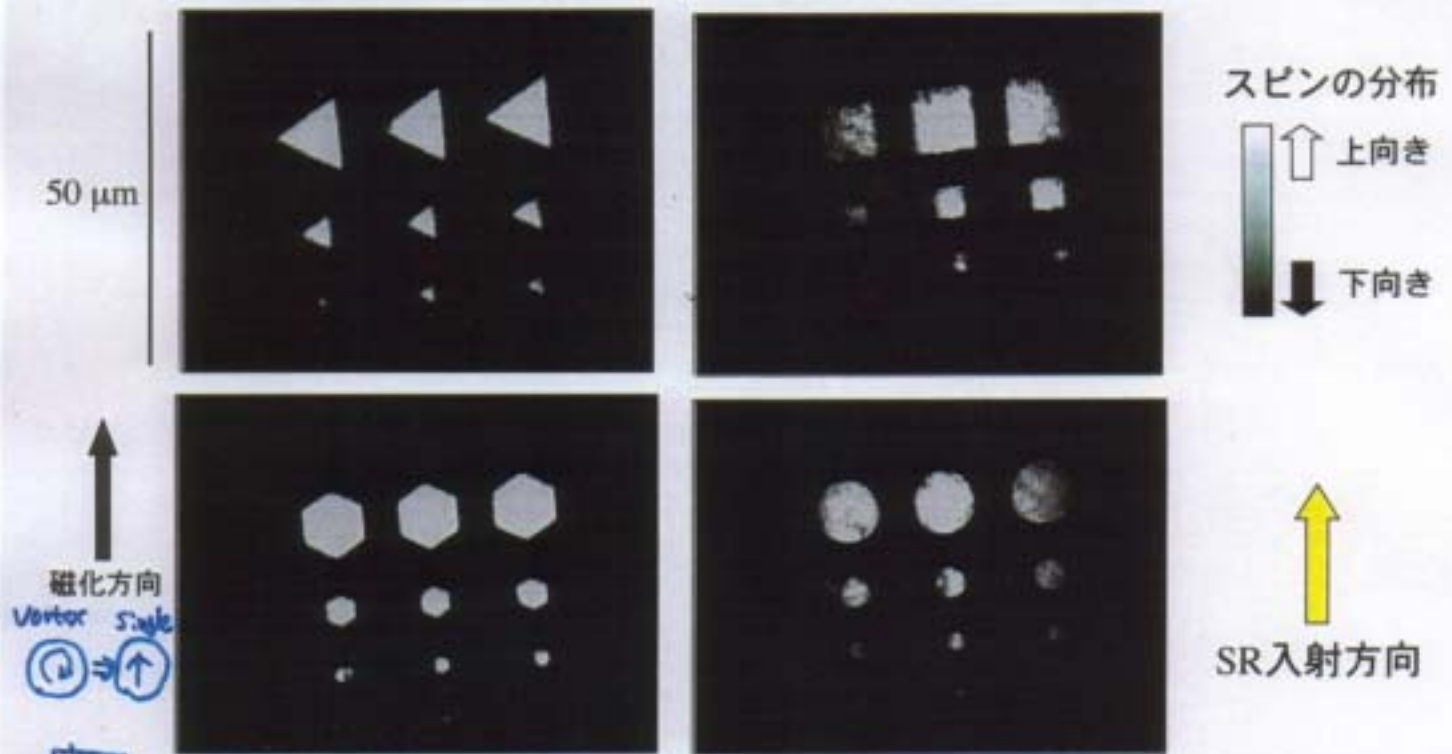
シミュレーションとの比較

Simulation ... OK.

small difference Defect in perfection of Dorr.

実験結果(3)

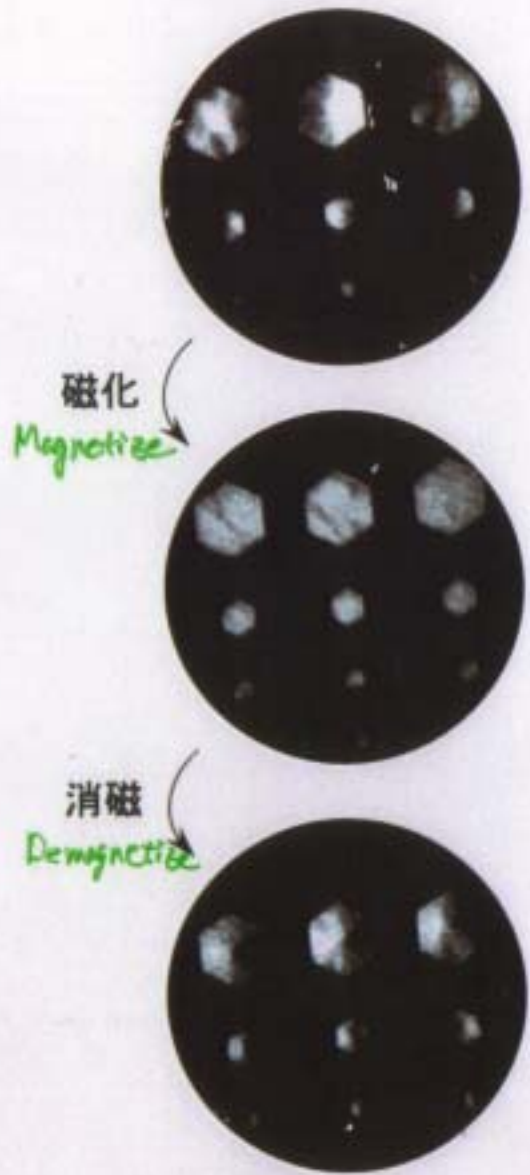
Magnetic field ~8K0e pulse



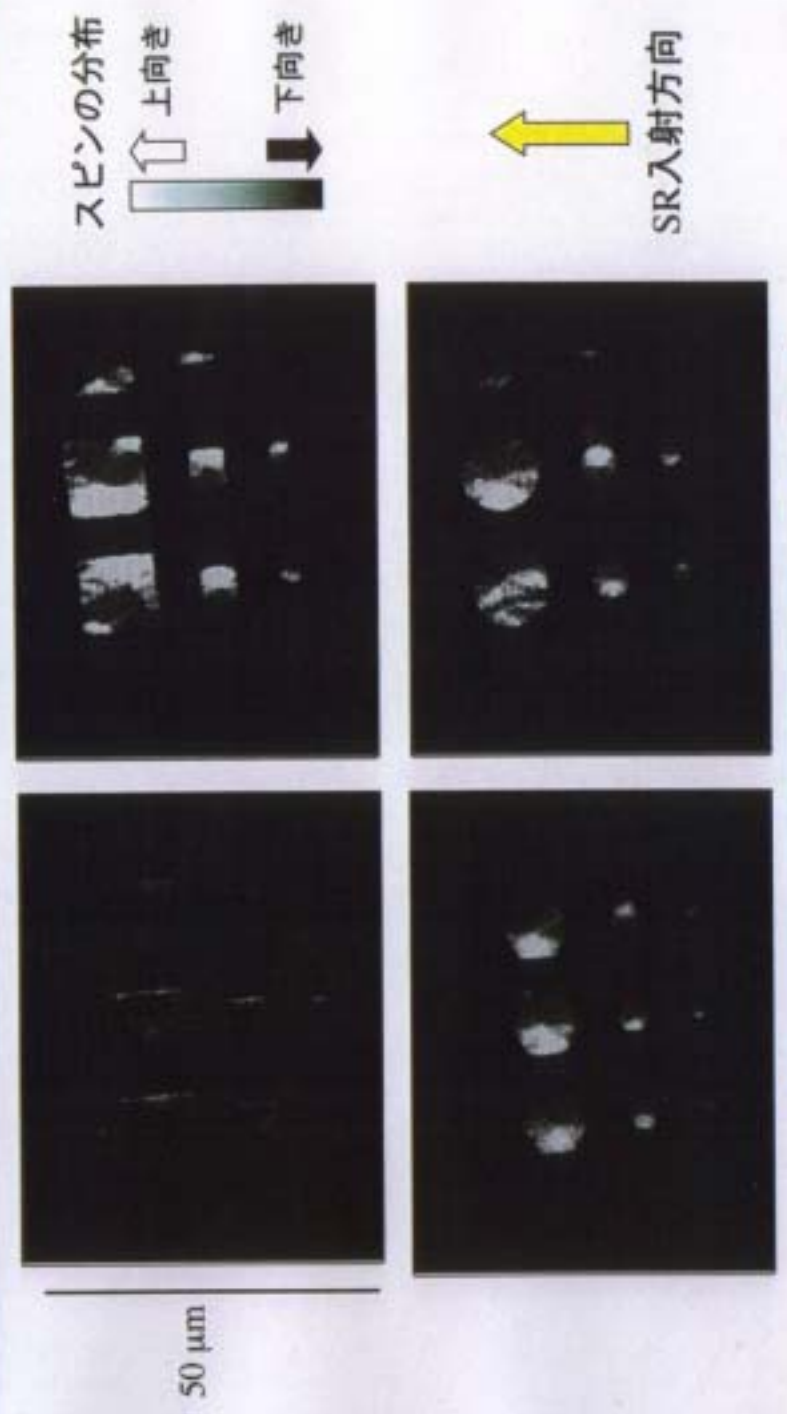
面内方向に磁化した後に測定。シングルドメインになることを確認。

Single Domain

磁気構造の変化



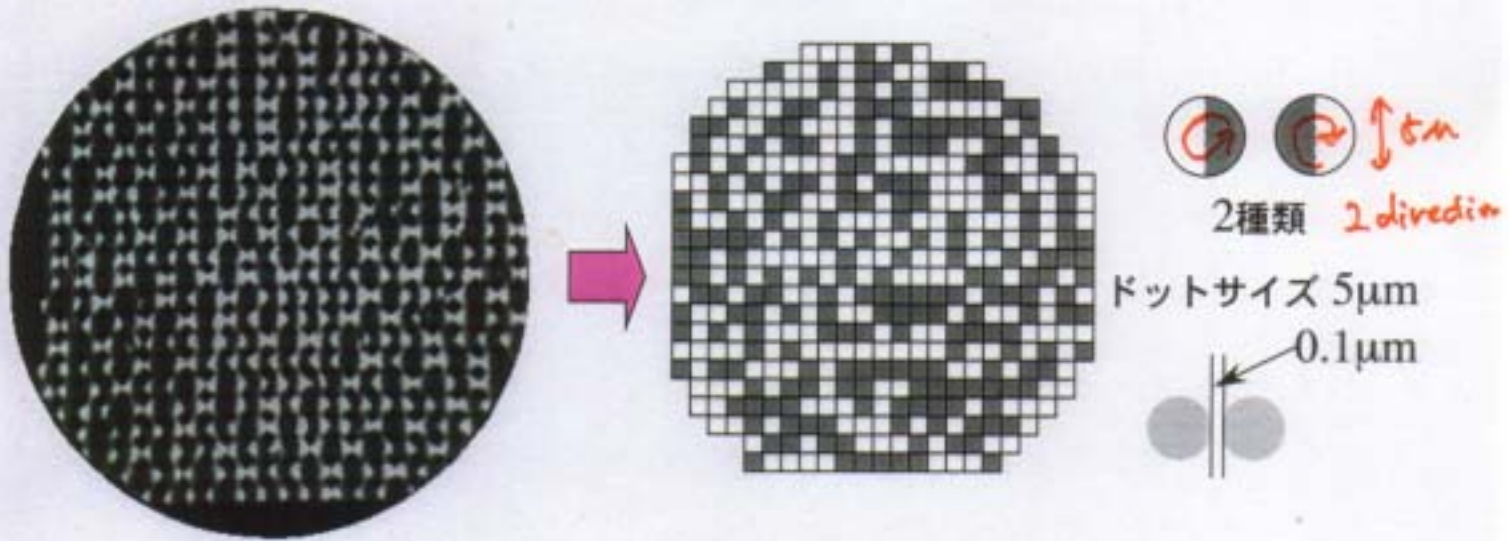
実験結果(4)



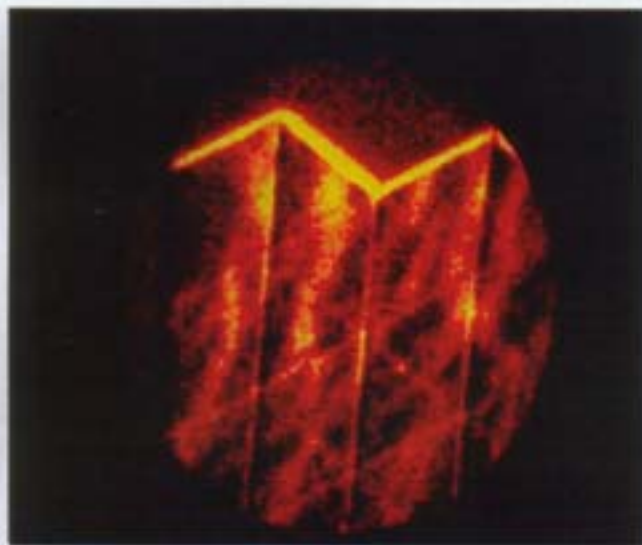
再び大気中にて消磁を行って後に測定。

実験結果(5)

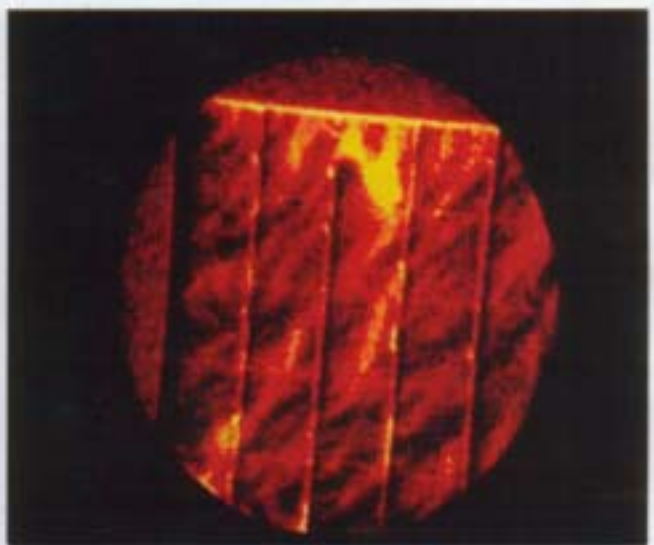
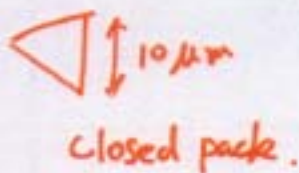
Niドット配列における磁氣的相互作用



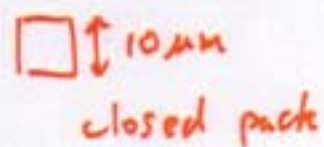
ドットの漏れ磁界が小さいため、
ドット間の相互作用はほとんどない。



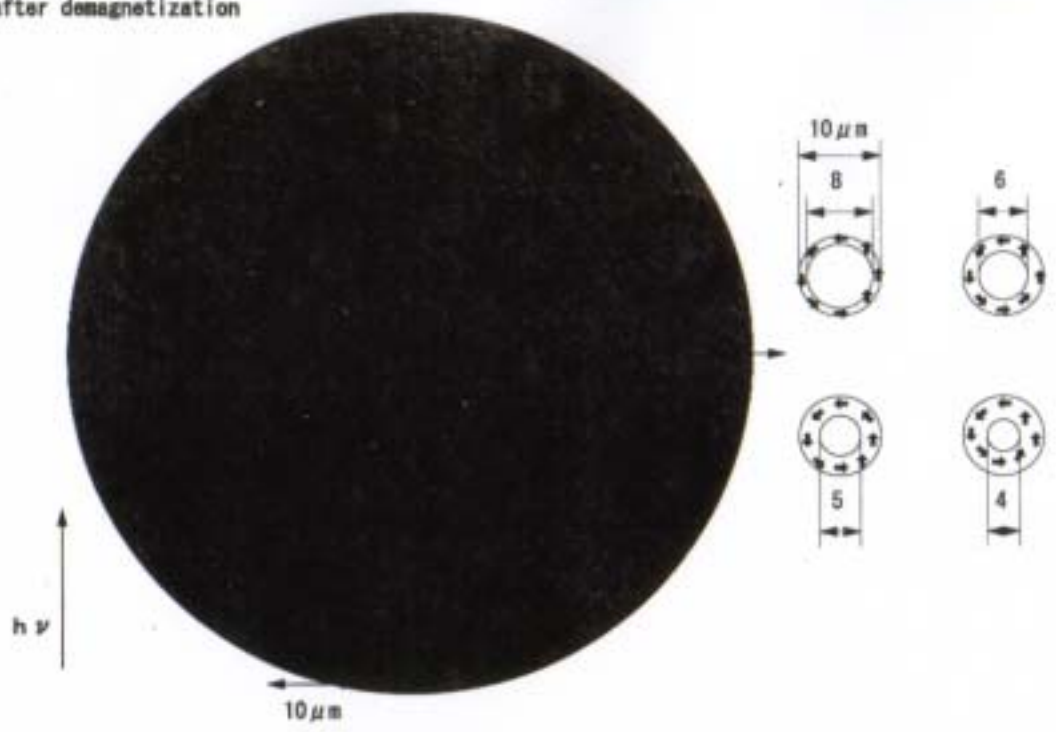
大きさ10μmの正三角形のドット



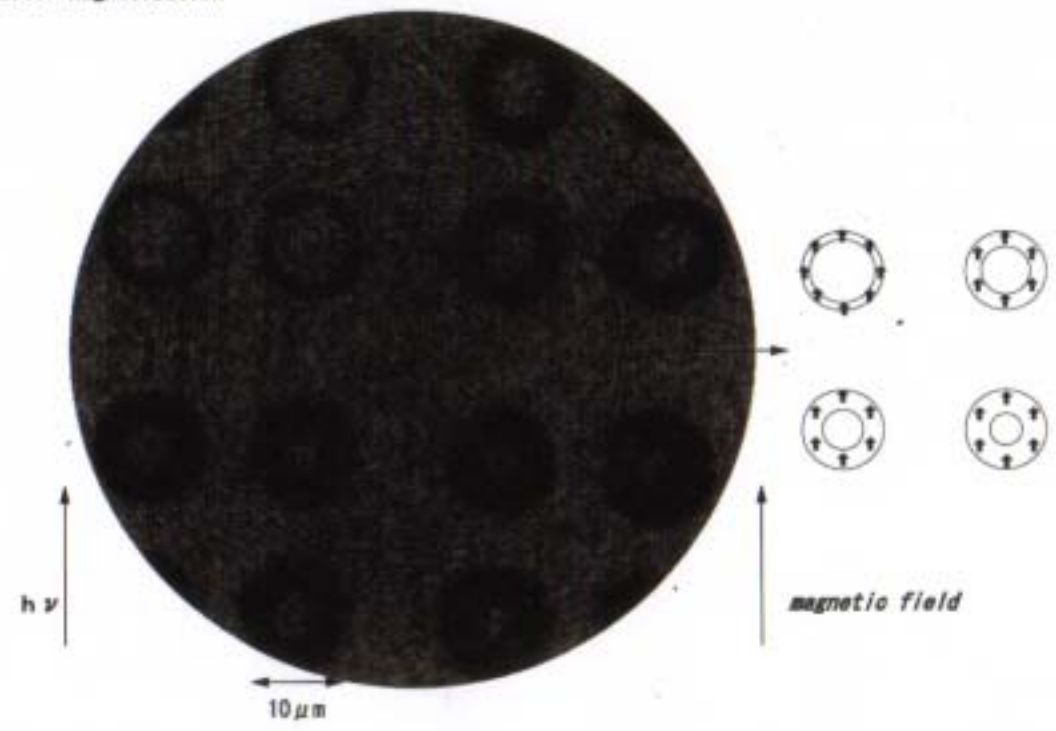
大きさ10μmの正方形のドット



after demagnetization



after magnetization



SMART ; BESSY II

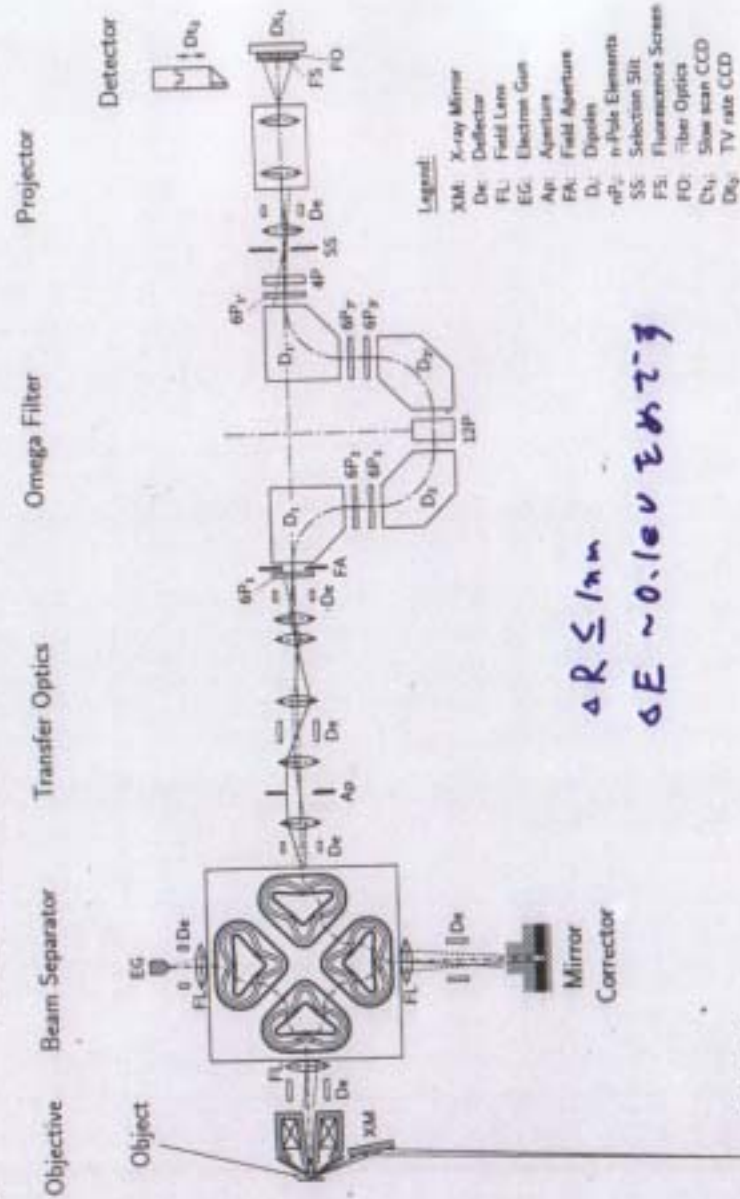


Fig. 1. Schematic layout of the new spectromicroscope SMART under construction for a BESSY II undulator beamline.

magnetic field, a stigmator and deflection elements.

the beam separator and the electrostatic mirror, since

$$0 < E_k < 1000 eV$$

$$1 < \Delta E < 5 eV$$

X-PEEM X-ray PhotoEmissionElectronMicroscope

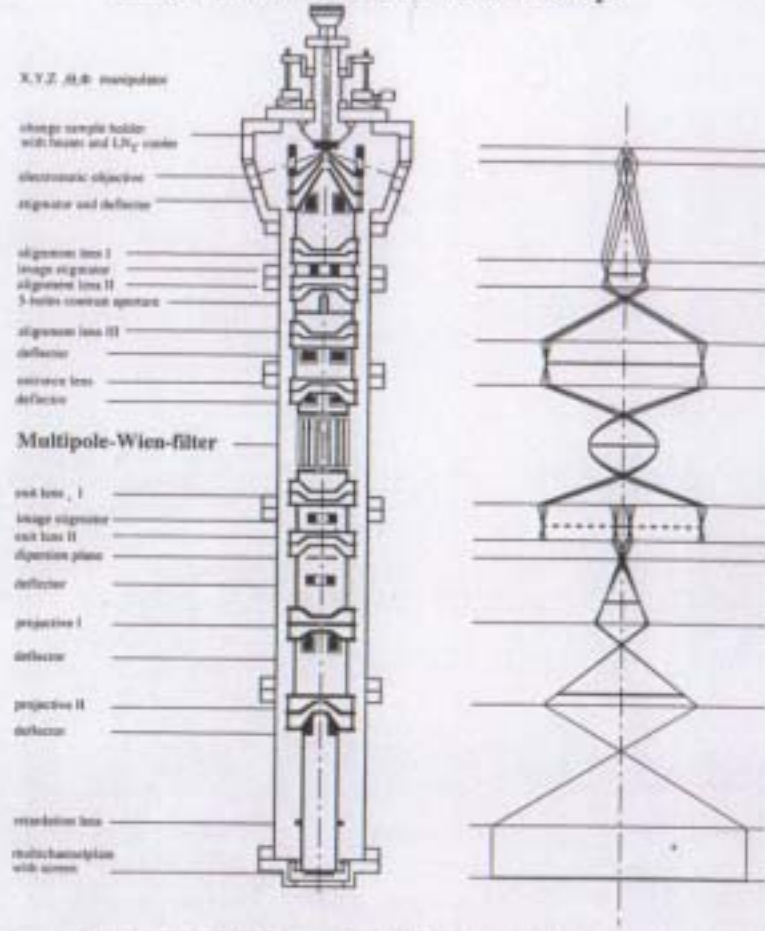


Fig. 1. Cross section of the microscope with multipole WIEN filter and fundamental rays (right).

With
SR
~20nm
~0.5eV

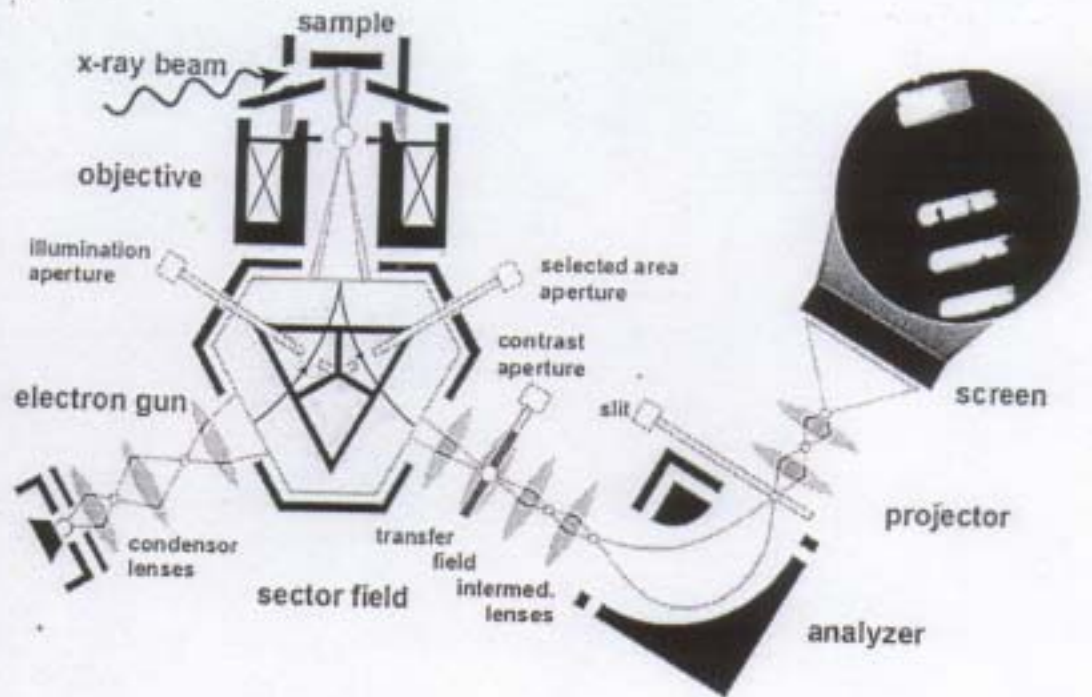
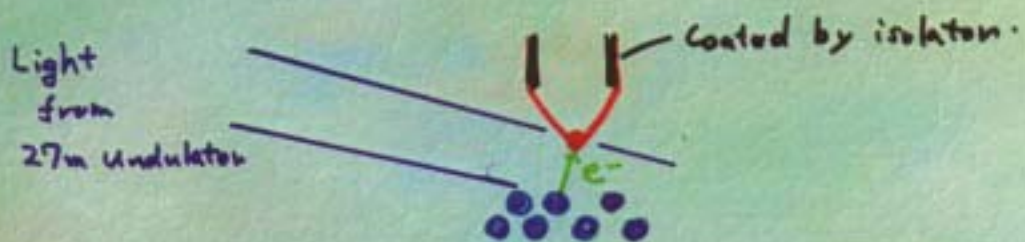
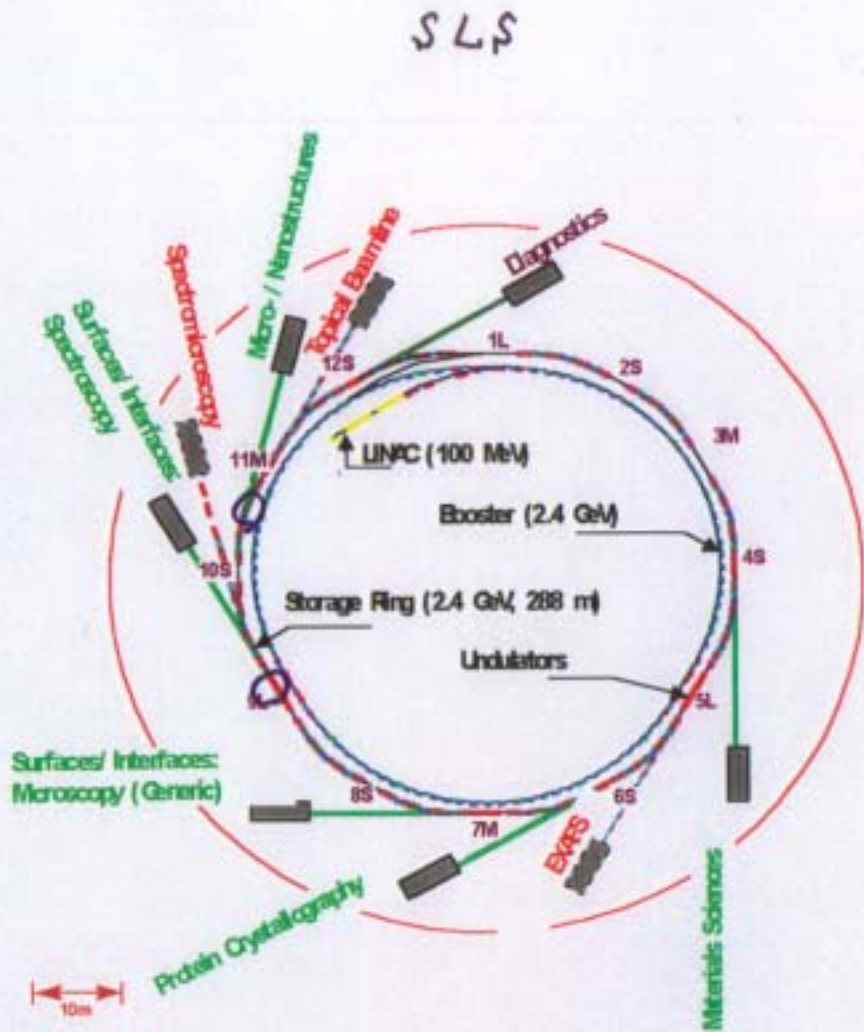
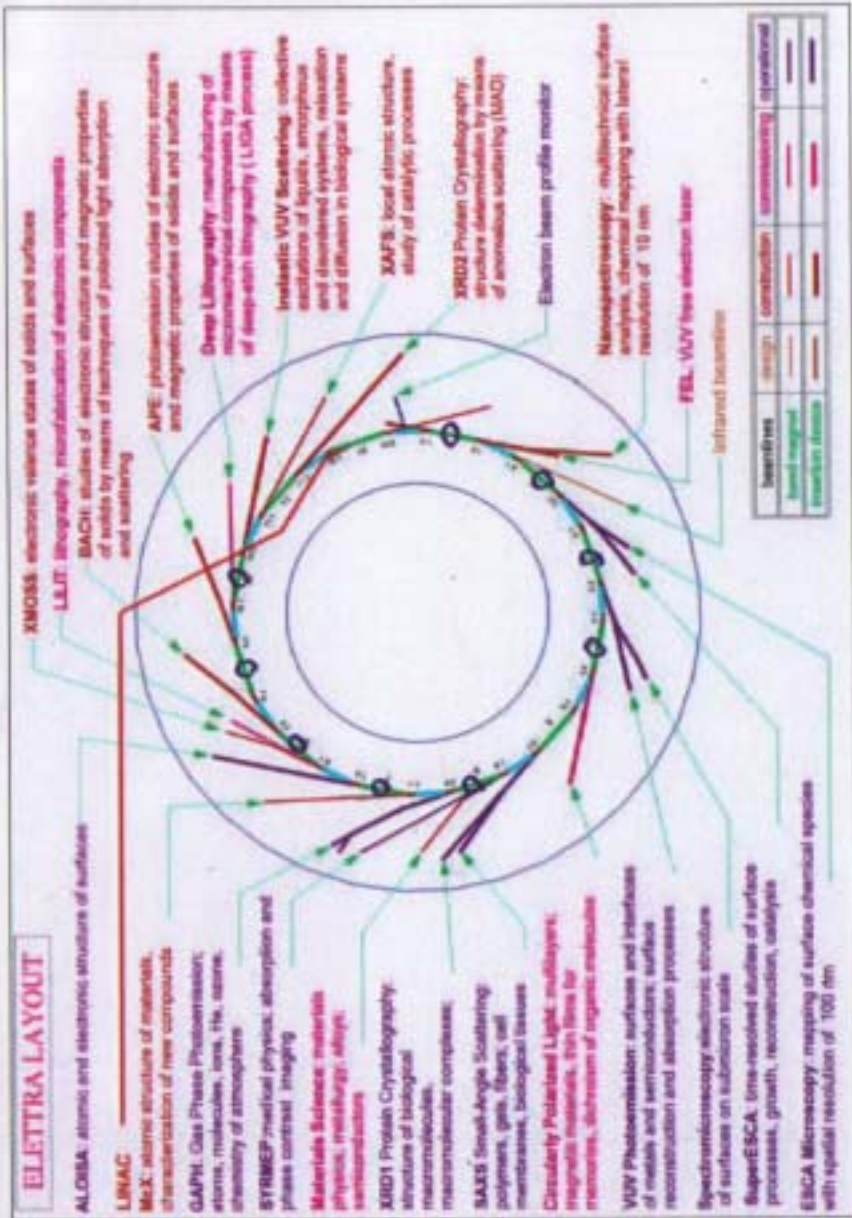


Fig. 2. Schematic drawing of the SPELEEM instrument.

Combined study of SR&STM towards element specific atomic imaging, and atomic scale photoemission spectroscopy



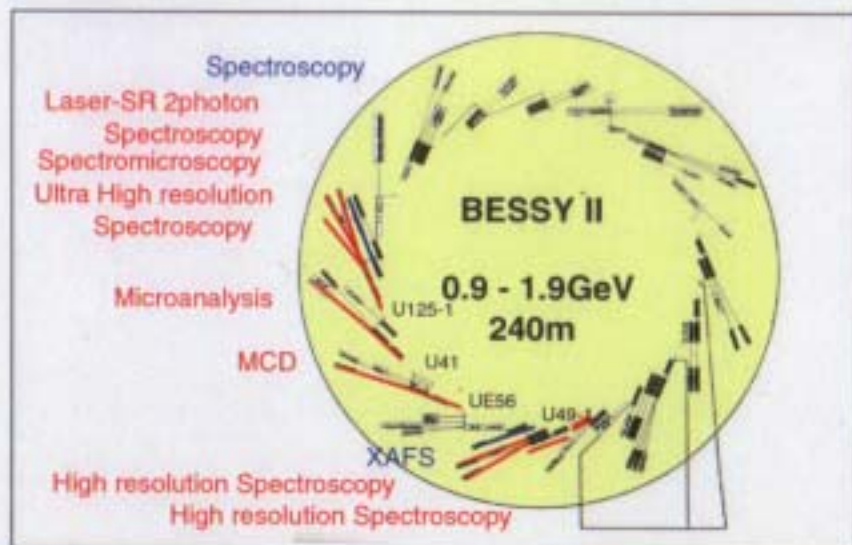
- Several proposals,
- Hasegawa (ISSP) et al., has already performed the work combined with X-ray tube.
- Nishitani et al.(Kyuushyu Inst. Tech.), has observed photoemission effect within 20nm by mercury lamp.
- Gray also observed the nano-meter length effect of photocurrent.(J.Electron Spectrosc.Relat. Phenom. 109(2000)183.)
- Order estimation
- 27m light source=>10 μ m, Si 2p, 10⁵ electrons/s/atom
- Probably, 1fA/atom



将来 VUV-SX
6本?

Beamline Overview 1998- **BESSY II** 6.1nm*rad(1.57GeV)

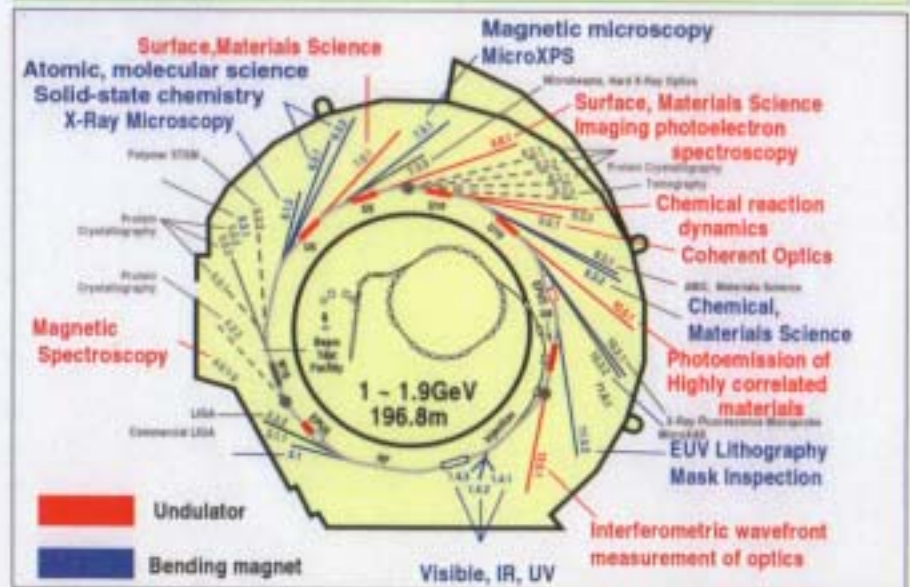
BEAMLINES	Bend-Magnet	Wiggler	Undulator
Now	7	2	6
Future	21	-	6
ENERGY RANGE	20 eV - 20 keV	hv > 2.0 keV	5 - 1500 eV



Beam Port	Source type	Energy Range	Experiments
ID-05-1,2	UE56-2	90 - 1300eV	MCD
ID-06-0(a,b)	4T-WLS/0.4T-LFD	>2000(a), >400(b)	X-ray LIGA, Soft X-ray LIGA
ID-07-0	U180	white	Compton Scattering
ID-07-2	U180	25 - 1300eV	Soft X-ray Radiometry
ID-09-2(a)	U49-1	130 - 1600eV	High resolution Spectroscopy
ID-09-2(b)	U49-1	90 - 1500eV	High resolution Spectroscopy*
ID-11-2	UE56-1	90 - 1300eV	MCD
ID-12-1	U41	150 - 1600eV	Microanalysis
ID-13-1	U125-1	20 - 1300eV	Ultra-high resolution Spectroscopy
ID-13-2	U125-1	200 - 800eV	Spectromicroscopy
ID-13-3	U125-1	1 - 440eV	Laser-SR 2-photon Spectroscopy
D-06-1-A	Bending	<15keV	LIGA
D-07010A	Bending	<15keV	Radiometry
D-07-1-B	Bending	1750 - 10keV	X-ray Radiometry
D-09-1-A1	Bending	<15keV	Diagnostic Beamline
D-09-1-A2	Bending	20 - 1900	XAFS
D-0902	Bending	2keV - 15keV	XAFS
D-13-1-B	Bending	200 - 700	Spectroscopy
D-13-2	Bending	2keV - 12keV	Energy Dispersive Reflectometry

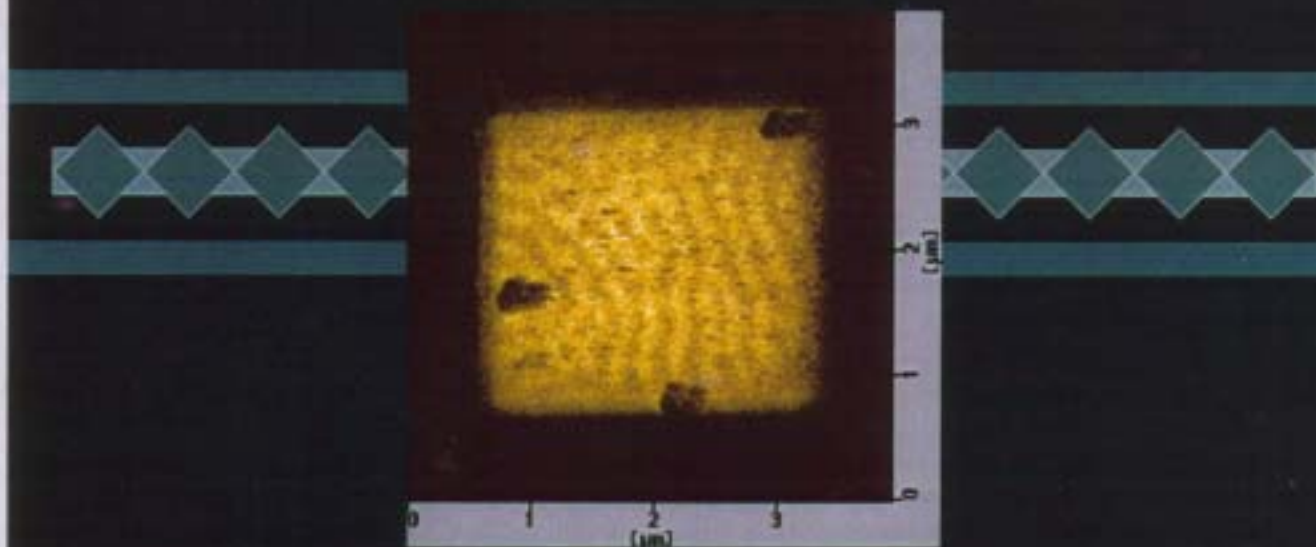
Beamline Overview 1993- **ALS** 3.5nm*rad(1.5GeV)

BEAMLINES	Bend-Magnet	Superbend	Wiggler	Undulator	IR/VIS/UV
Now	13	0	1	7	3
2002	14	5	3	8	3
ENERGY RANGE	30 eV - 20 keV	6 - 18 keV	3.5 - 14 keV	5 - 1800 eV	0.005 - 6.2 eV



Beam Port	Source type	Energy Range	Experiments
1.4.1	Bend	1.6-6.2 eV	Ultraviolet photoluminescence
1.4.2	Bend	0.002-3 eV	Visible and infrared Fourier transform spectroscopy (FTIR)
1.4.3	Bend	0.05-1 eV	Infrared spectromicroscopy
4.0.1-2	EPUS	60-1800 eV	Magnetic spectroscopy
6.1.2	Bend	300-800 eV	High-resolution zone-plate X-Ray Microscopy
6.3.1	Bend	500-2000 eV	Solid-state chemistry
6.3.2	Bend	50-1300 eV	Atomic, molecular, and materials science
7.0.1	U5	50-1200 eV	Surface, Materials Science Photon-polarization dichroism
7.3.1.1	Bend	175-1500 eV	Magnetic microscopy, spectromicroscopy
7.3.1.2	Bend	175-1500 eV	Micro x-ray photoelectron spectroscopy
8.0.1	U5	65-1400 eV	Surface and materials science, imaging photoelectron spectroscopy
9.0.1	U10	200-800 eV	Coherent optics experiments
9.0.2	U10	5-30 eV	Chemical reaction dynamics
9.3.2	Bend	30-1400 eV	Chemical and materials science
10.0.1	U10	17-340 eV	Photoemission of highly correlated materials
11.3.2	Bend	50-1000 eV	Inspection of EUV lithography masks

展望 軟磁性体への応用



La_{0.7}Sr_{0.3}MnO₃(001)薄膜
 面内**CMR or GMR** : 磁区ドメイン?
 MFM(面直磁区観察例:京大化研)

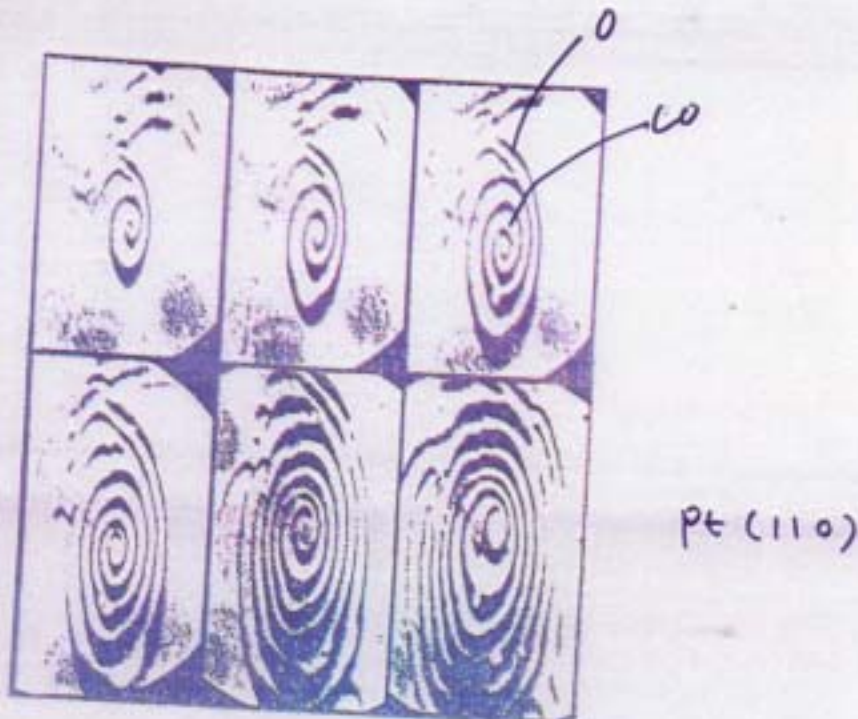


図3 Pt上でのCOの酸化反応の光電子放射顕微画像。結晶の方位は図の縦方向が[110]、横方向が[001]。短い辺が0.2mm。左上から右下までの経過時間74秒。

S. Jakobik et al.
 2011 15 (11) 730-737