TEM of Epitaxial Thin Films Controlled by Planes Extending (near) Normal to Interface;

with Application to 2 Methods to Reduce Crystal Orientations in Polycrystalline Longitudinal Magnetic Media;

Reducing 2-D-Oriented to 1-1/2-D-Oriented, & Reducing 3-D-Oriented to 2-1/4-D-Oriented.

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@ Komag: M. Zhang, L. He, M. Lu, W. Cao, C. Chen, L.H. Chan, B. Lairson
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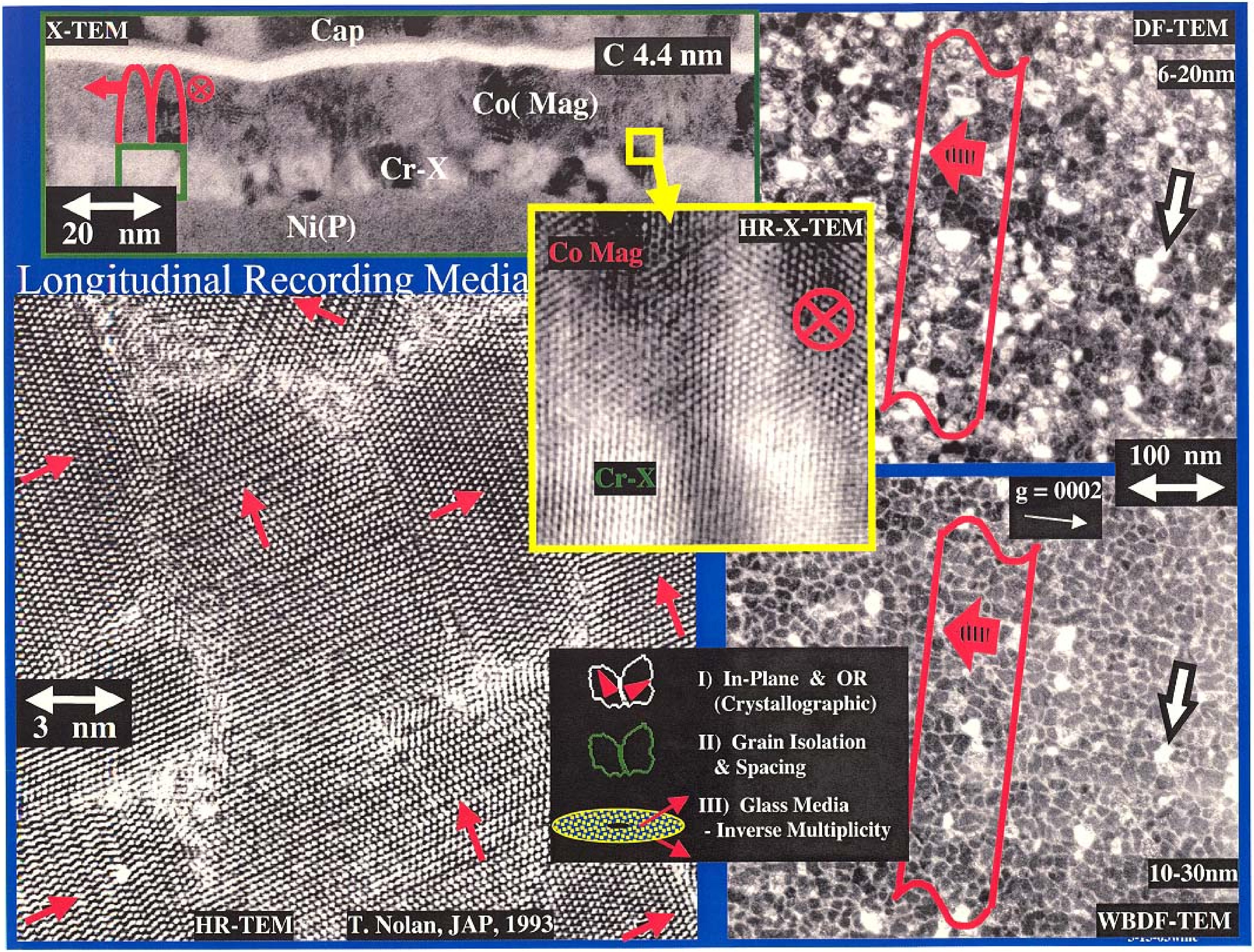
@ National Center for Electron Microscopy, LBL: J. Turner, R. Kilaas, U. Dahmen

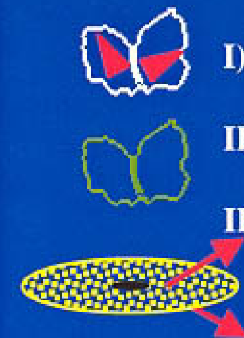
3-15-03wmc


X-TEM
Cap
C 4.4 nm
Co(Mag)
Cr-X
Ni(P)
20 nm
Longitudinal Recording Media
HR-X-TEM
Co Mag
Cr-X
DF-TEM
6-20nm
100 nm
g = 0002
3 nm
I) In-Plane & OR
(Crystallographic)
II) Grain Isolation
& Spacing
III) Glass Media
- Inverse Multiplicity
10-30nm
HR-TEM
T. Nolan, JAP, 1993
WBDF-TEM

Circumferential Texture Scratches

1) M. Mirzamaani, et al, IEEE V26 (5) 2457 (1990).
2) K. Johnson, et al, IEEE V31 (6) 2721 (1995).

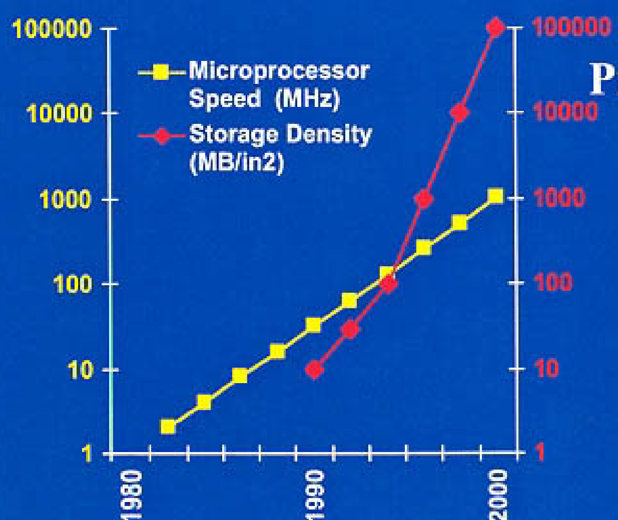
Longitudinal Winchester Media

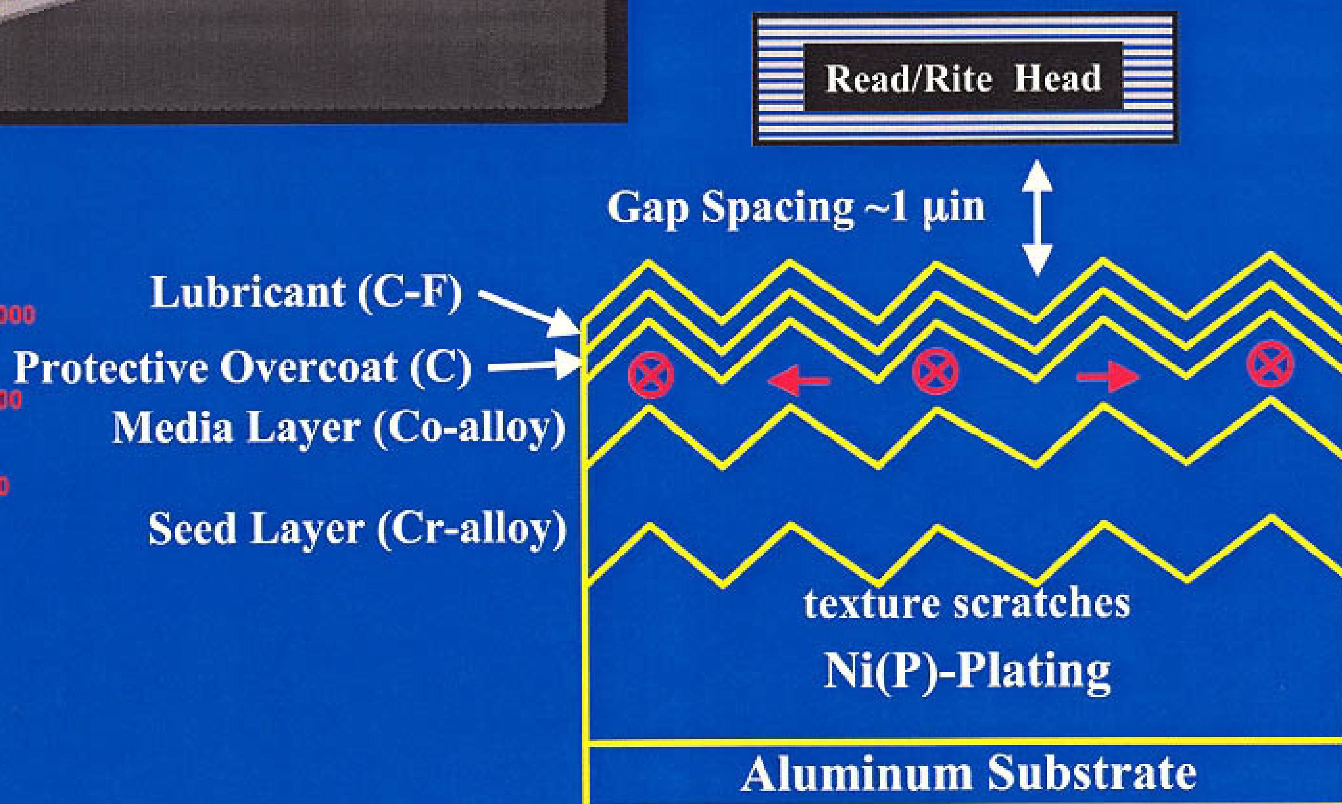


I) "Crystallographic" OR needs scratches (maybe) & ?

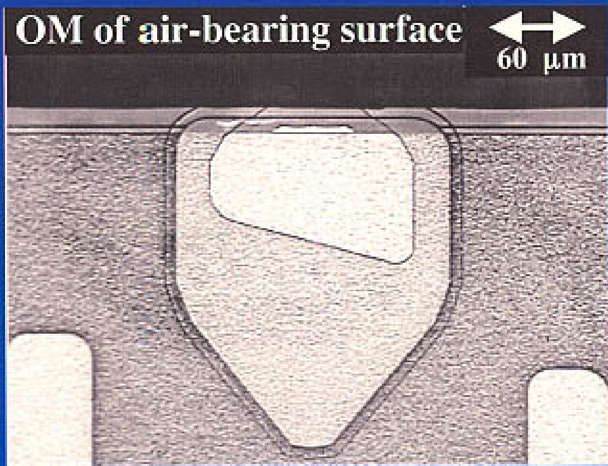
II) Grain Isolation & Spacing may be influenced by scratches

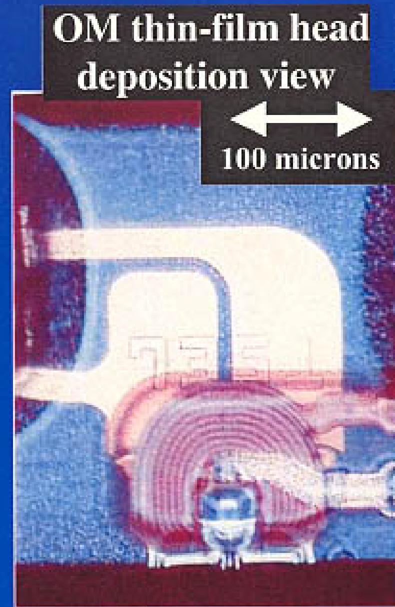
III) Glass Media & NiAl Crystallography scratches may be irrelevant

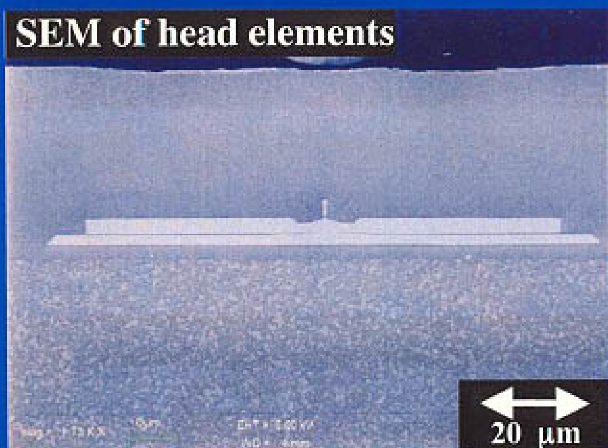




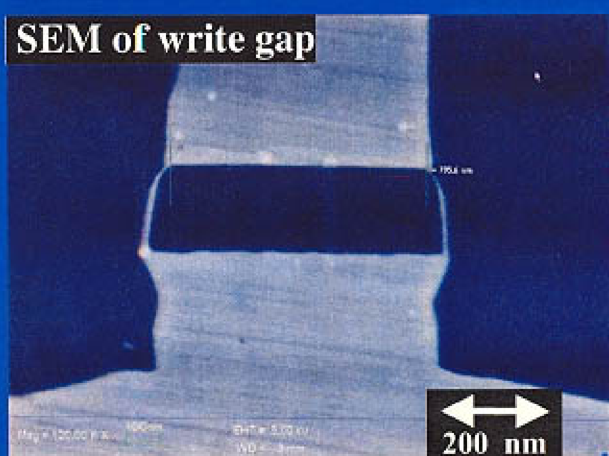
3-15-03wmc

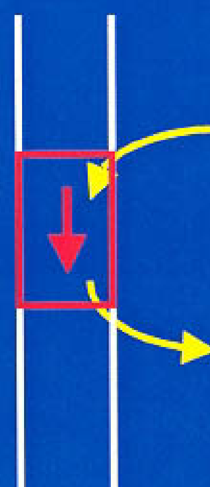


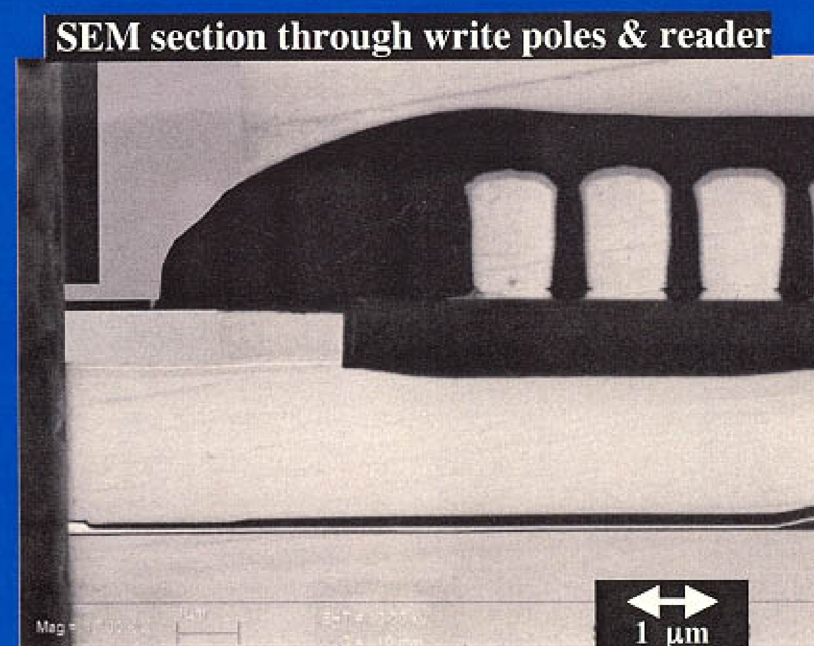


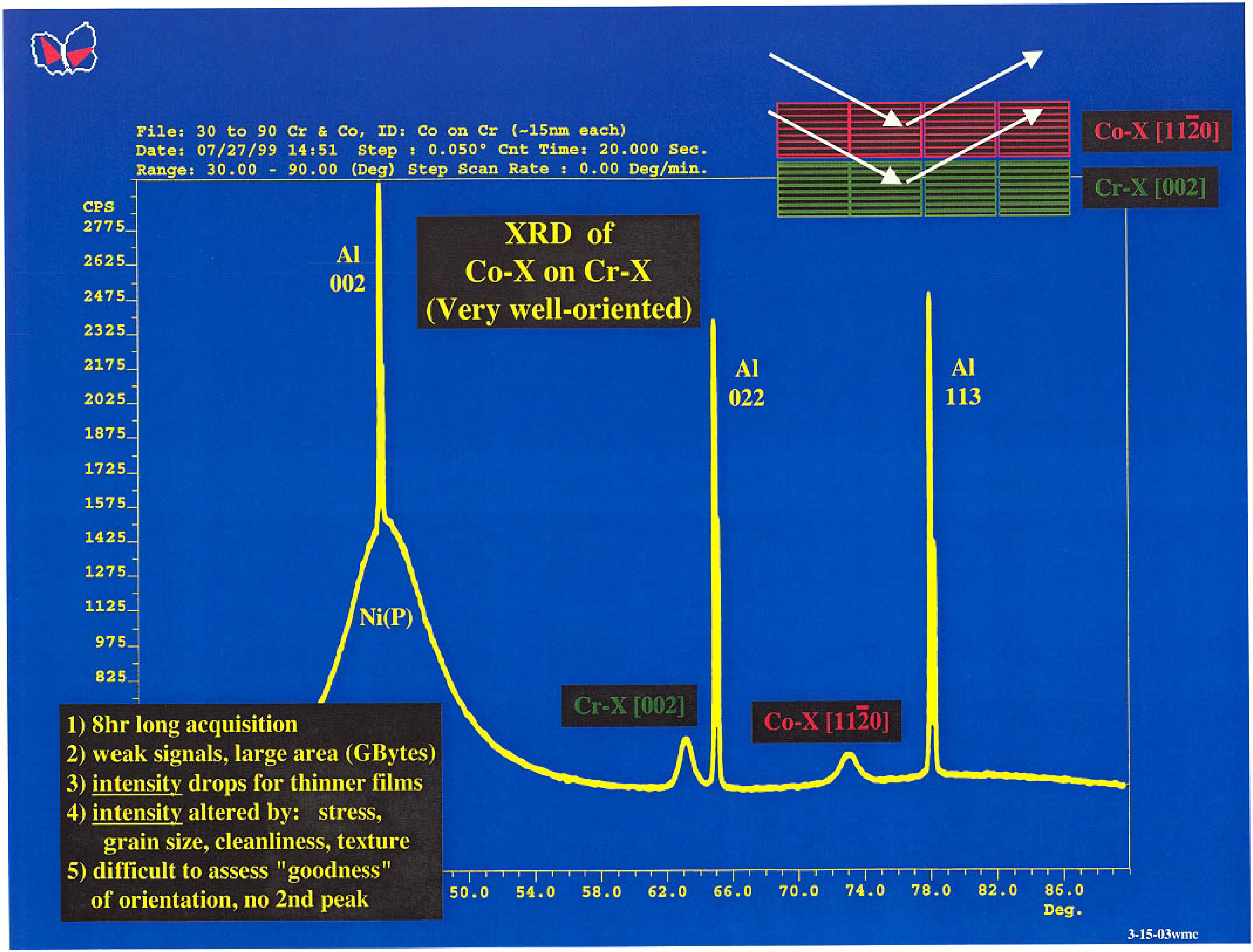


Thin Film Head

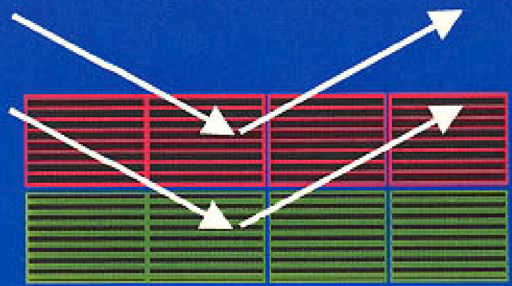


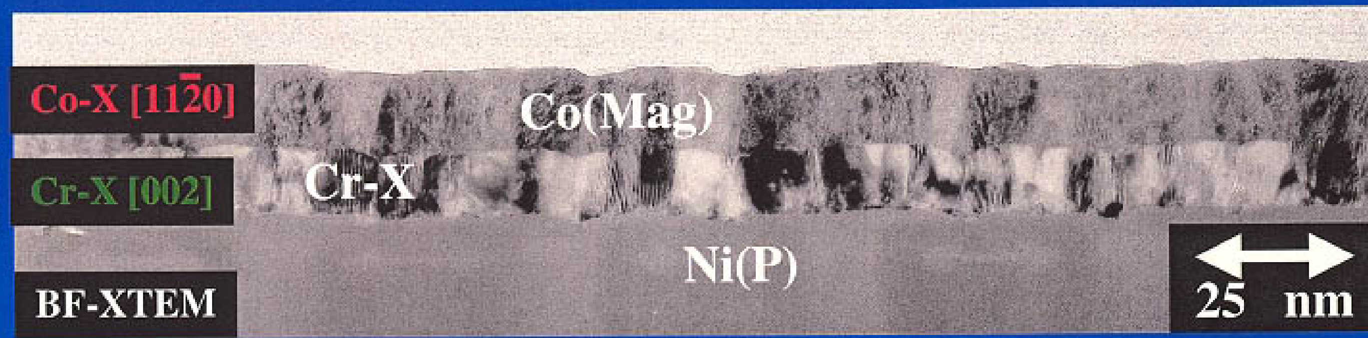


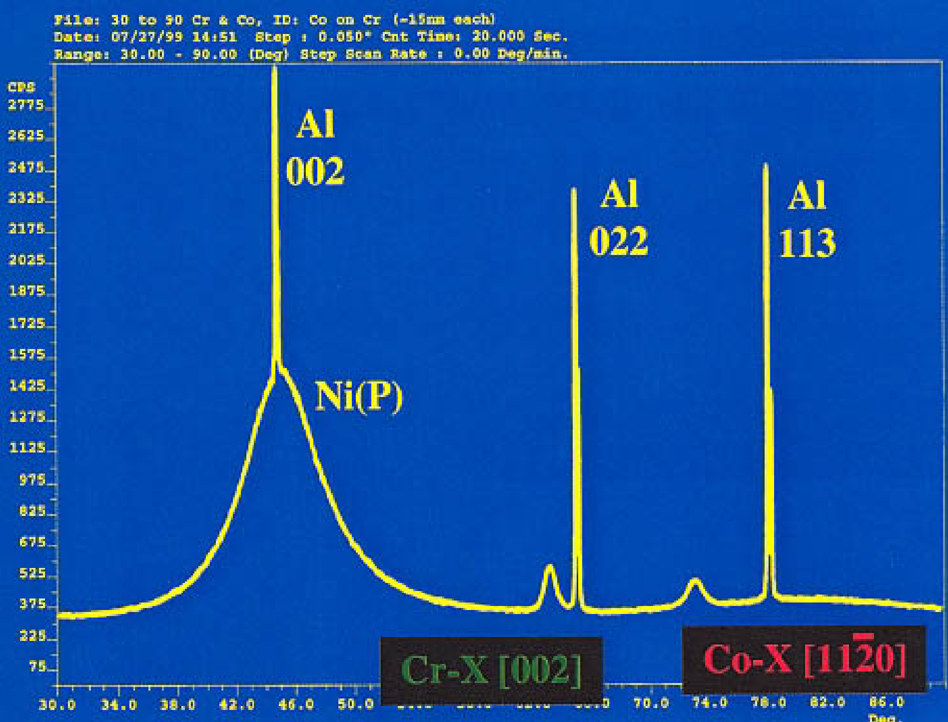


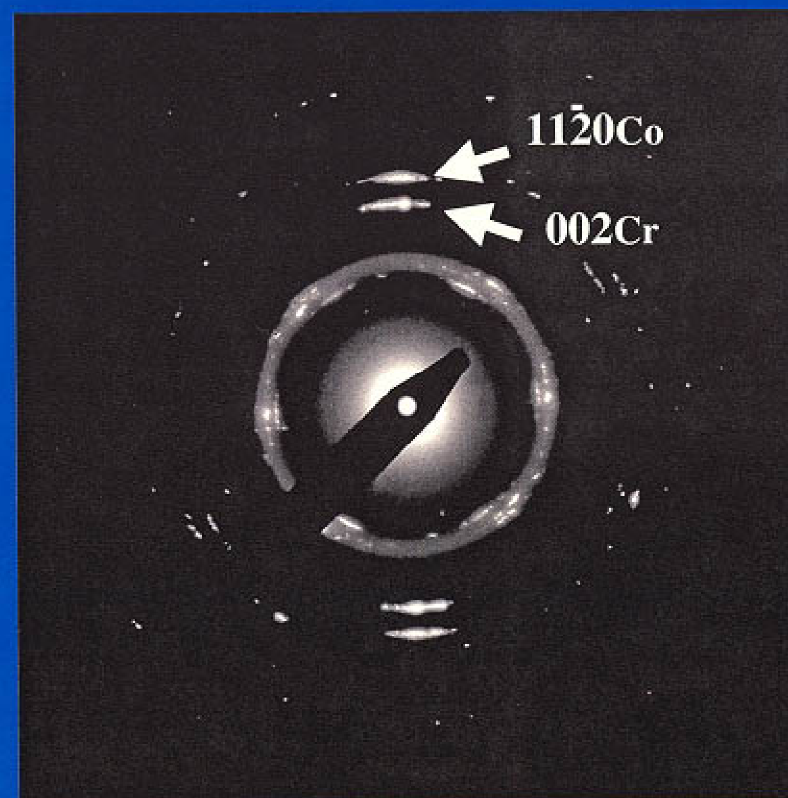

File: 30 to 90 Cr & Co, ID: Co on Cr (~15nm each)
Date: 07/27/99 14:51 Step : 0.050° Cnt Time: 20.000 Sec.
Range: 30.00 - 90.00 (Deg) Step Scan Rate : 0.00 Deg/min.
Co-X [11$\bar{2}$0]
Cr-X [002]
XRD of
Co-X on Cr-X
(Very well-oriented)
CPS
2775
2625
2475
2325
2175
2025
1875
1725
1575
1425
1275
1125
975
825
Al 002
Al 022
Al 113
Ni(P)
Cr-X [002]
Co-X [11$\bar{2}$0]
1) 8hr long acquisition
2) weak signals, large area (GBytes)
3) intensity drops for thinner films
4) intensity altered by: stress, grain size, cleanliness, texture
5) difficult to assess "goodness" of orientation, no 2nd peak
50.0
54.0
58.0
62.0
66.0
70.0
74.0
78.0
82.0
86.0
Deg.
3-15-03wmc

Out-of-Plane Diffraction of Well-Oriented [11$\bar{2}$0] Co-Alloy on [002] Cr





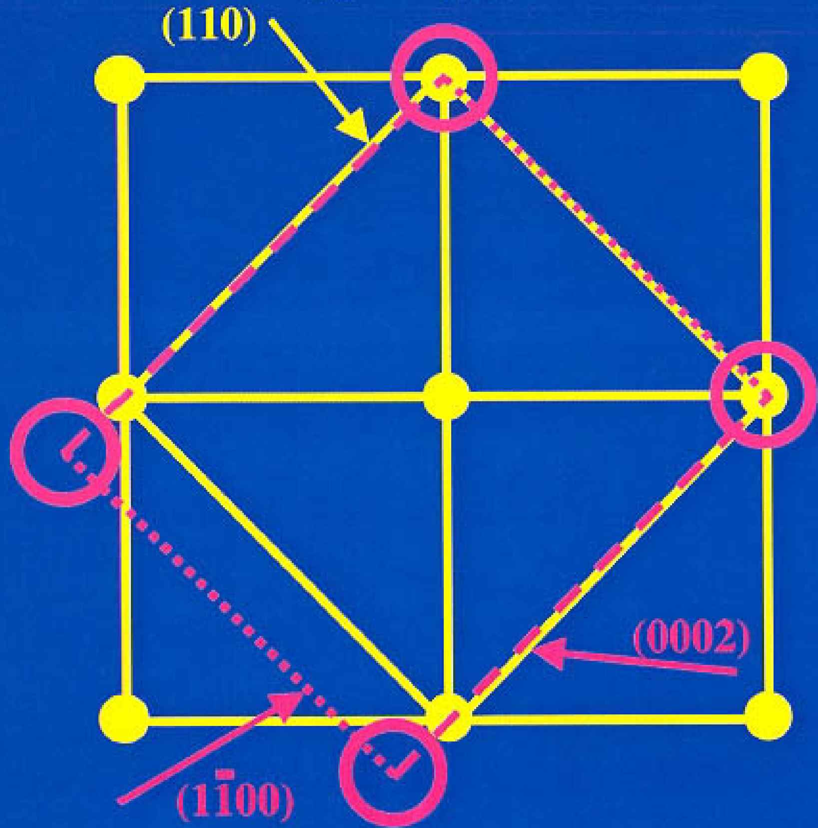




3-15-03wmc

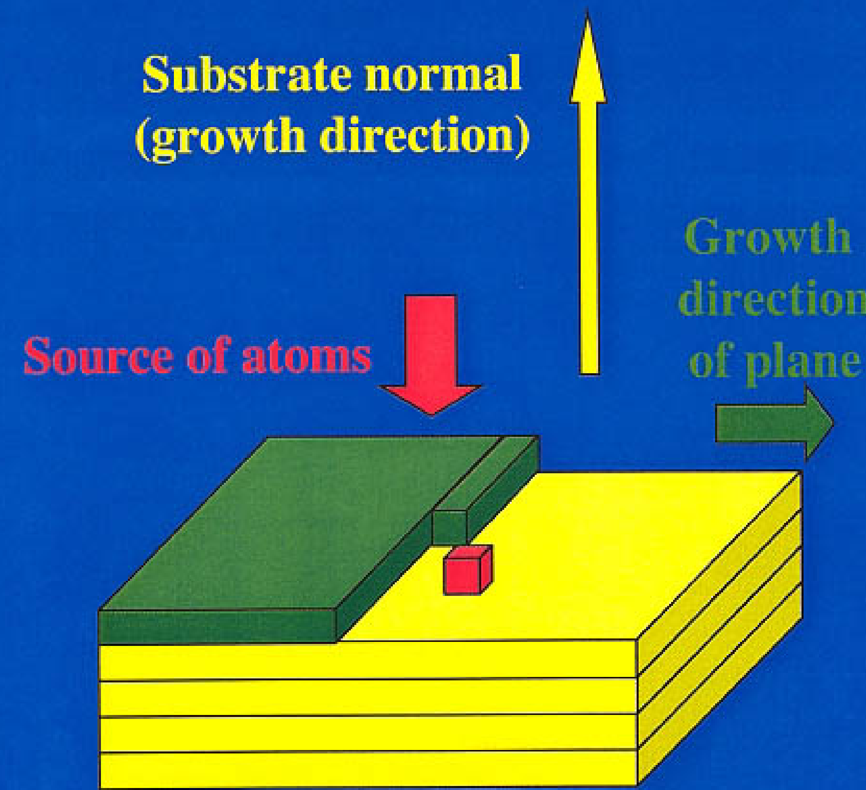
[11$\bar{2}$0] - Co // [002] - Cr
Hetero-epitactical Growth

$d_{110} \approx d_{0002} \approx 0.204\text{nm}$
$d_{1\bar{1}0} < d_{1\bar{1}00} \approx 0.220\text{nm}$



Alloy Cr to increase d_{110}
Alloy Co (Cr, Pt, Ta, B) to alter c/a
& magnetic properties: coercivity, noise
Multiplicity leads to Co - Bicrystals

TLK Terrace-Ledge-Kink
Growth Model



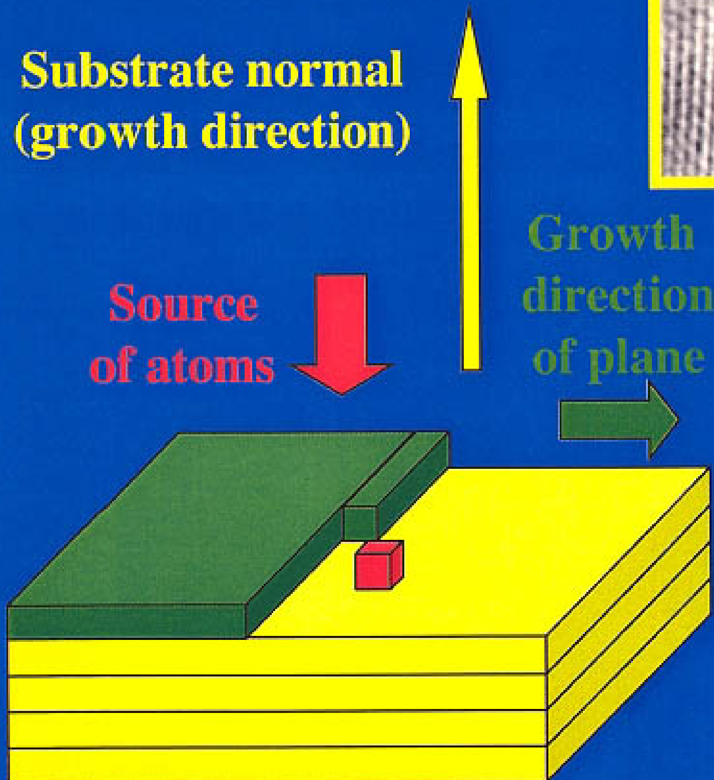
3-15-03wmc

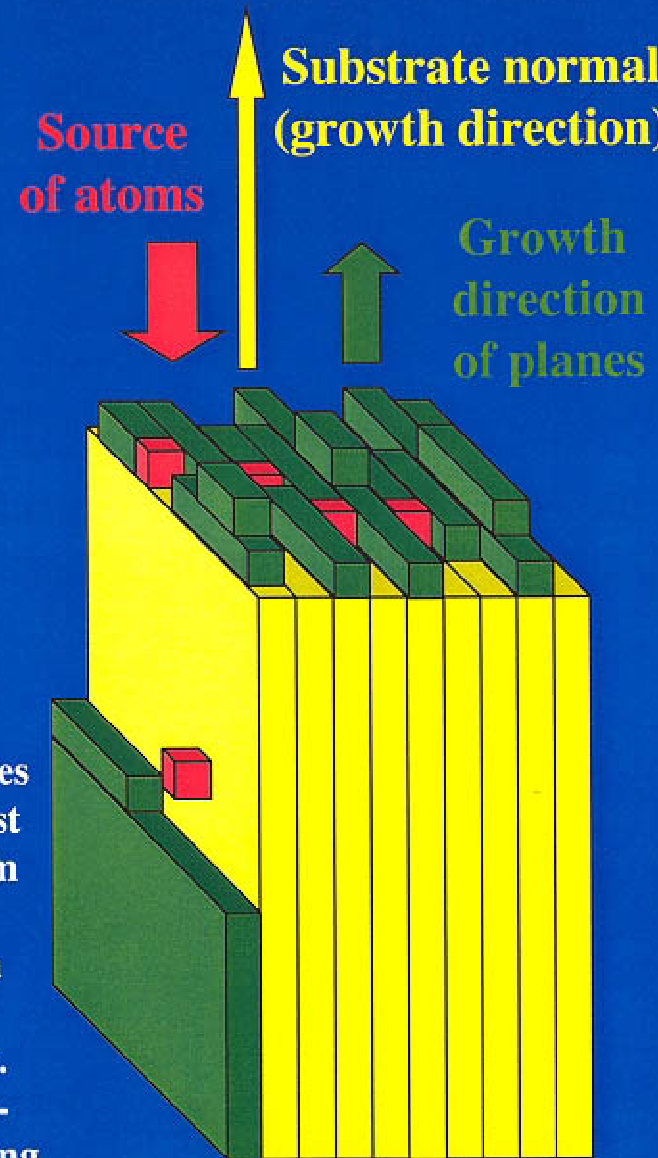
[11$\bar{2}$0] - Co // [002] - Cr

Hetero-epitactical Growth

2) During fast growth of films, surface is not atomically flat; Planes normal to surface grow, extending from one film to next.

1) Epitaxy is often considered as a well-controlled, system with slow kinetics and very flat surfaces. In a TLK (Terrace-Ledge-Kink) growth model, the "growing planes" grow in a direction parallel to the substrate.

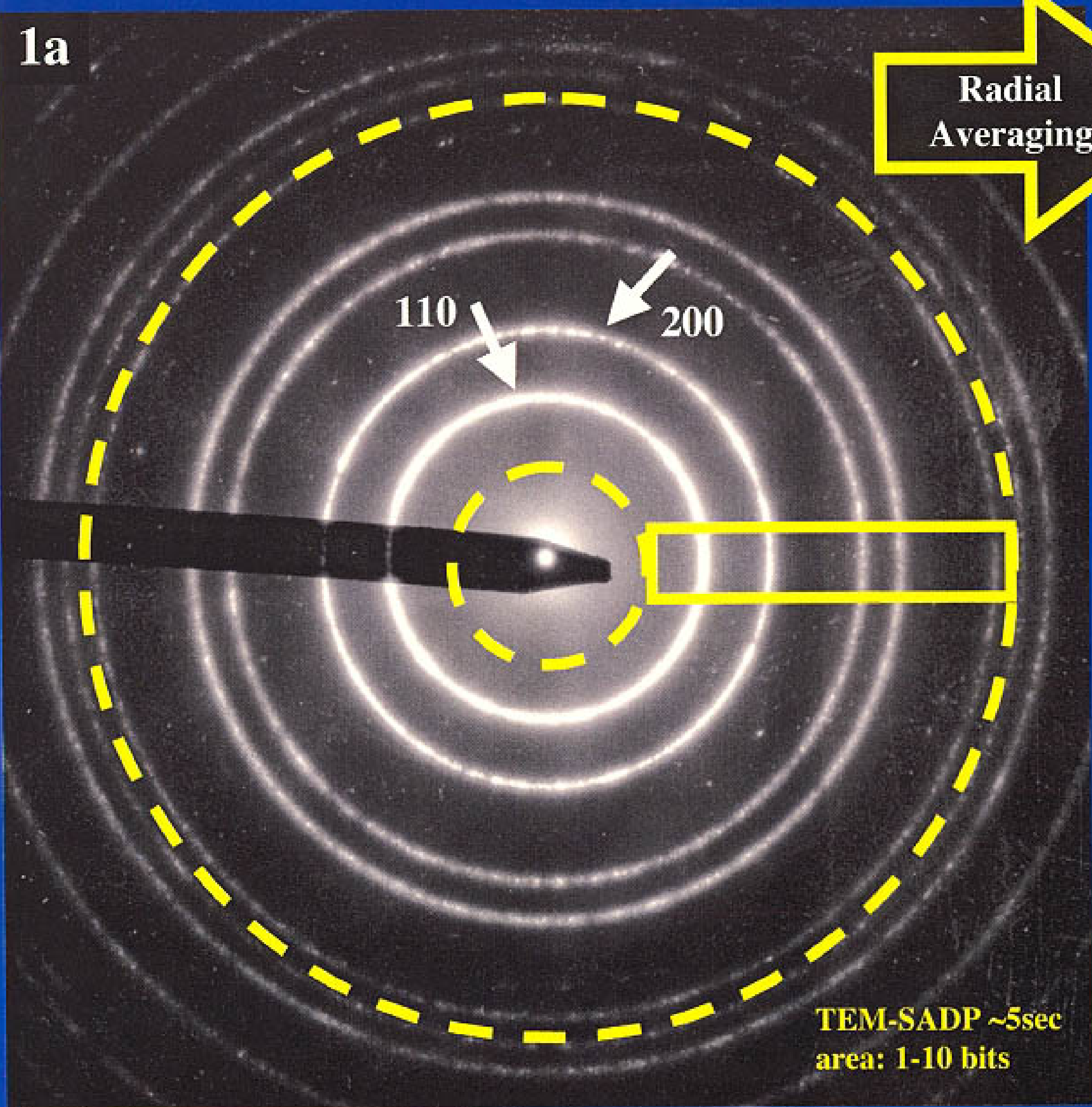


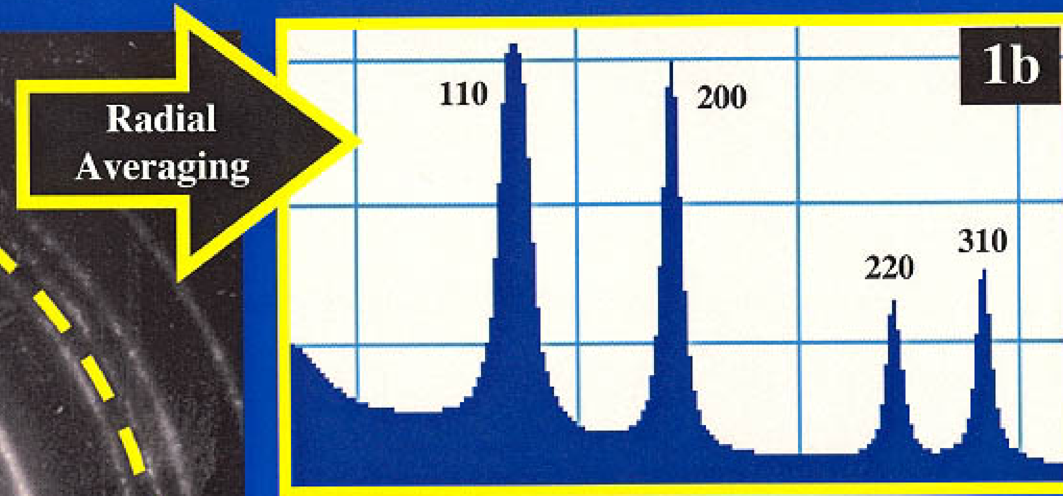


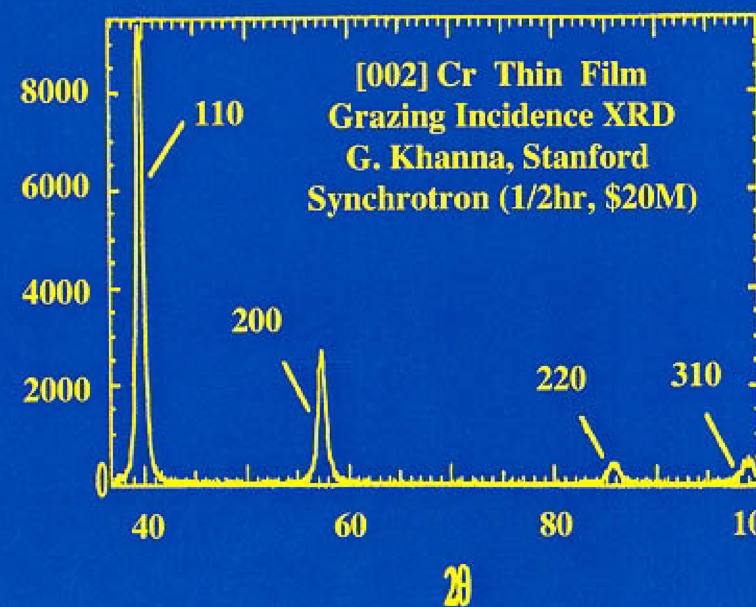
3) The lattice matching becomes controlled by one set of (closest packed) planes extending from one layer to next. And less concern depends on how lattices template the substrate in a top-down view. Electron Diffraction of plane-view-samples enables monitoring this lattice matching in 3D.

3-15-03wmc

Well - Oriented [002] Cr Polycrystalline Thin Film



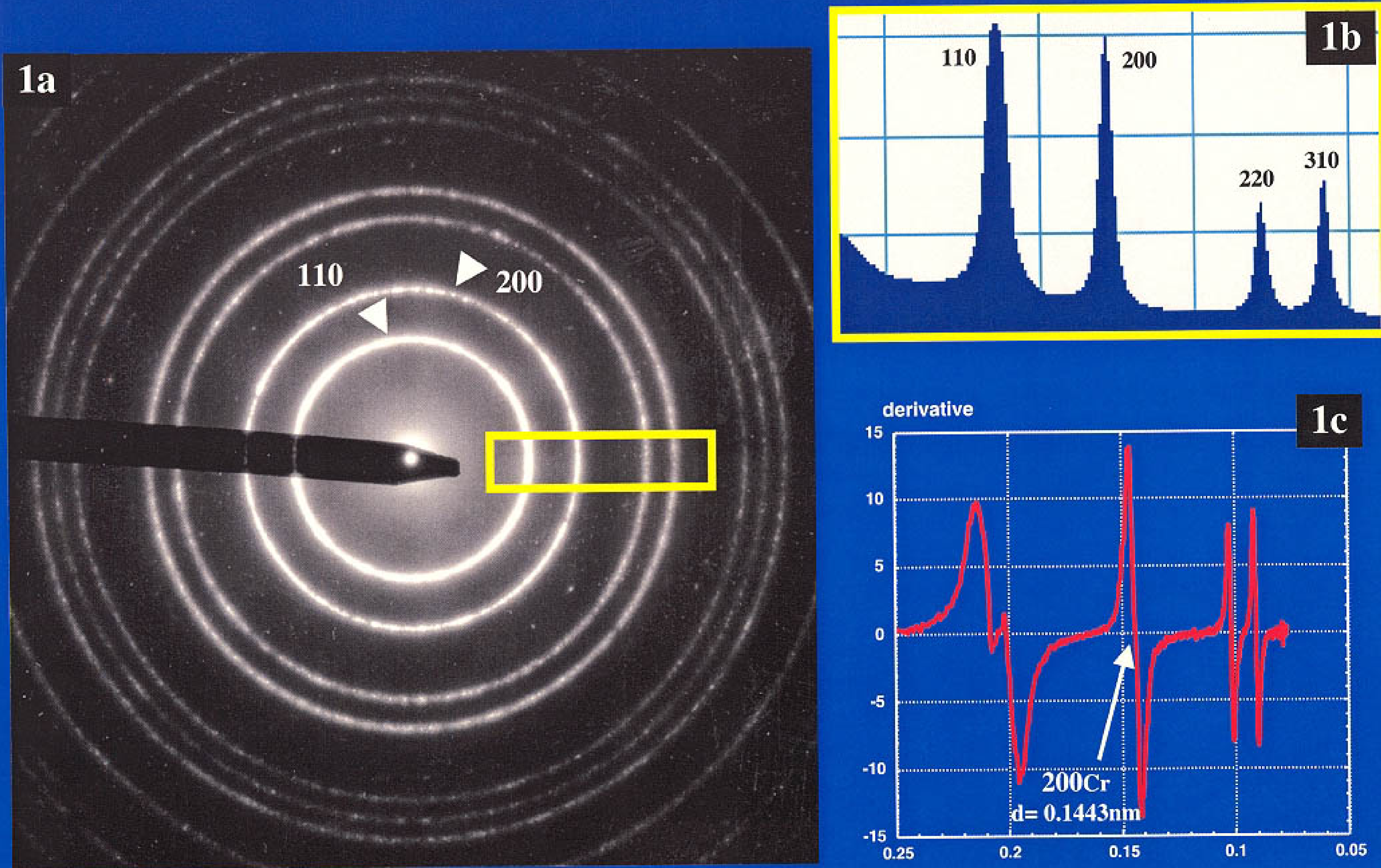




Microscopy, V4(2), 1998; & V4(2), 2000

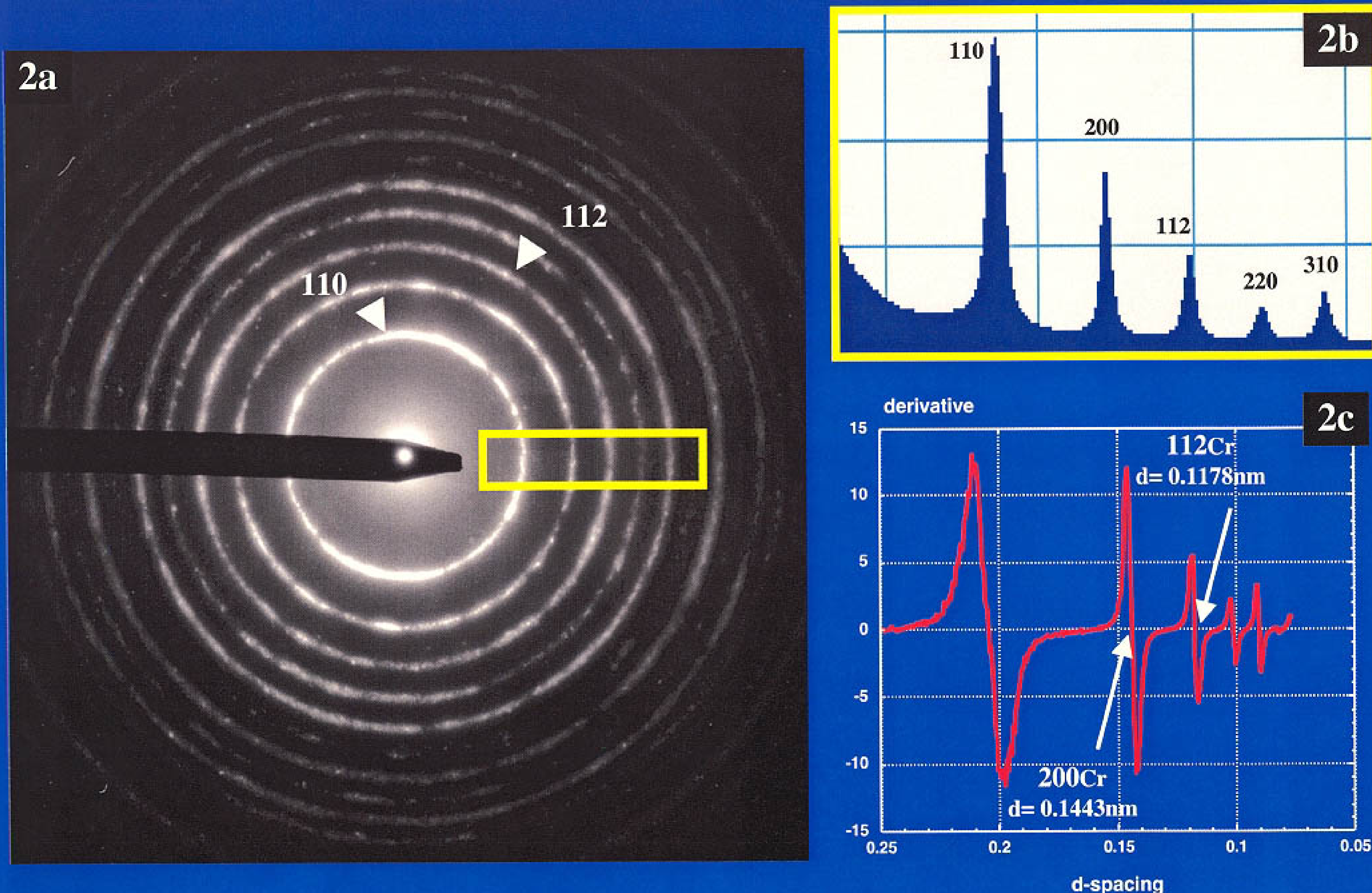
3-15-03wmc

Well - Oriented [002] Cr Polycrystalline Thin Film

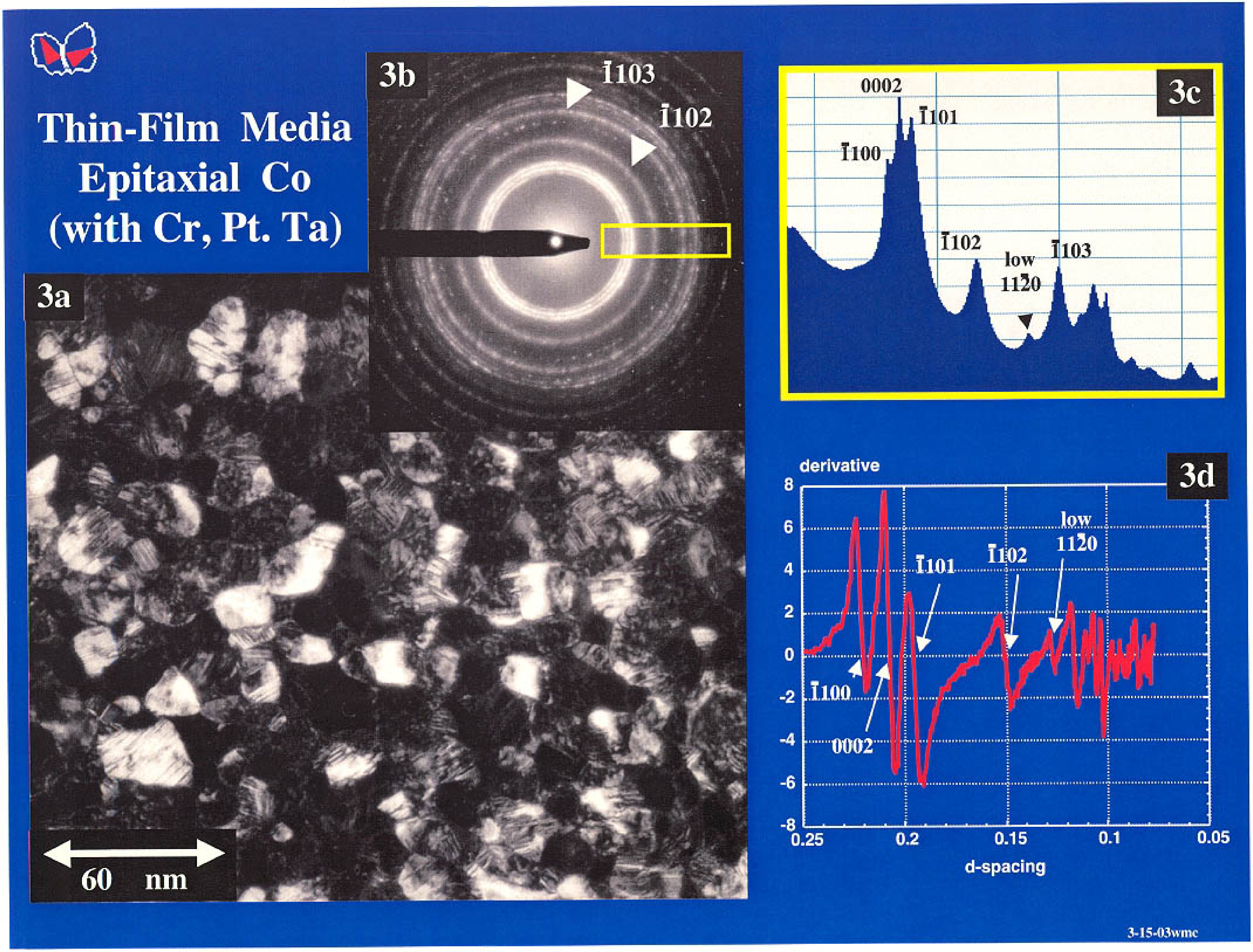


3-15-03wmc

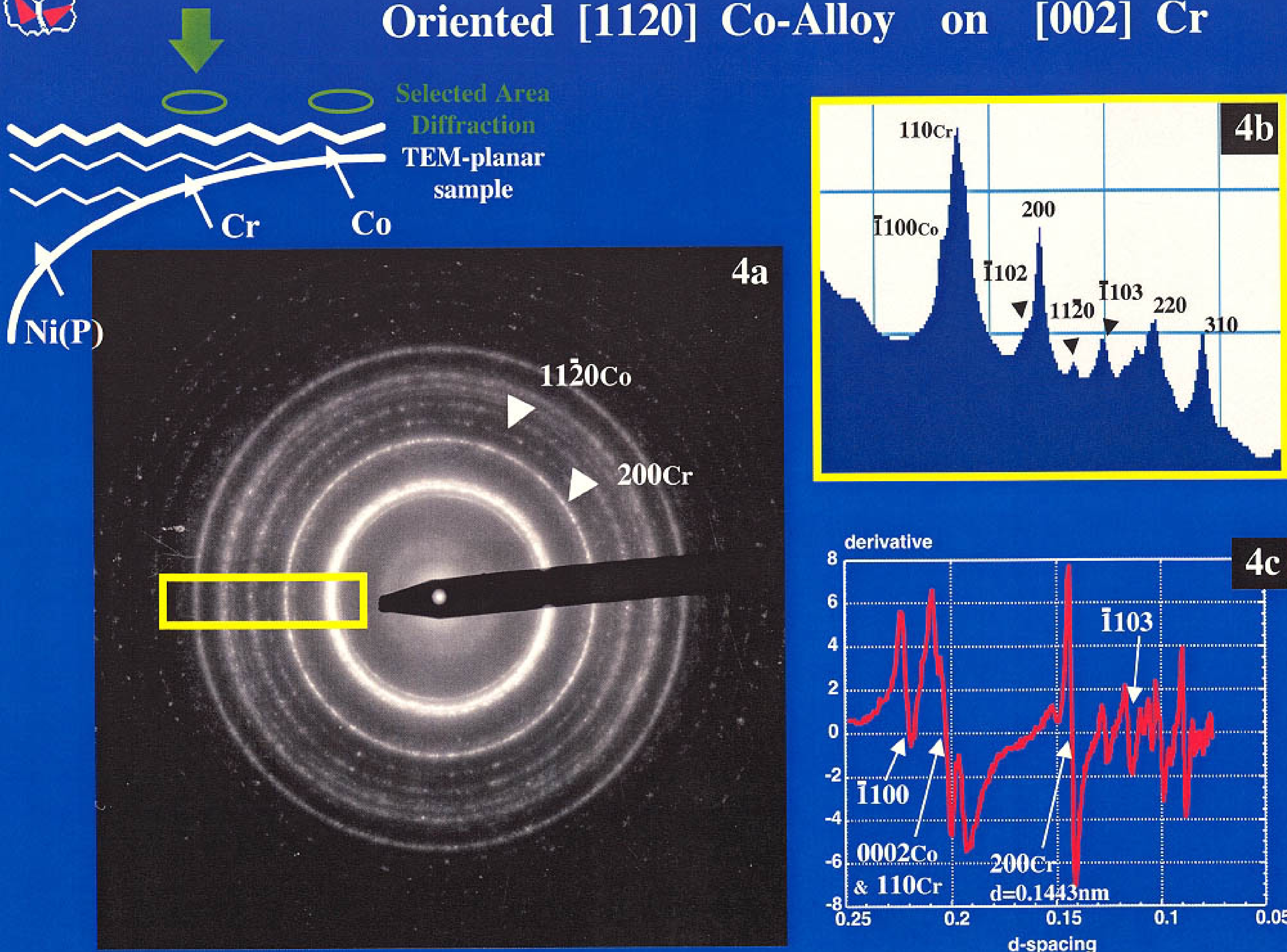
Randomly - Oriented Cr Polycrystalline Thin Film



3-15-03wmc

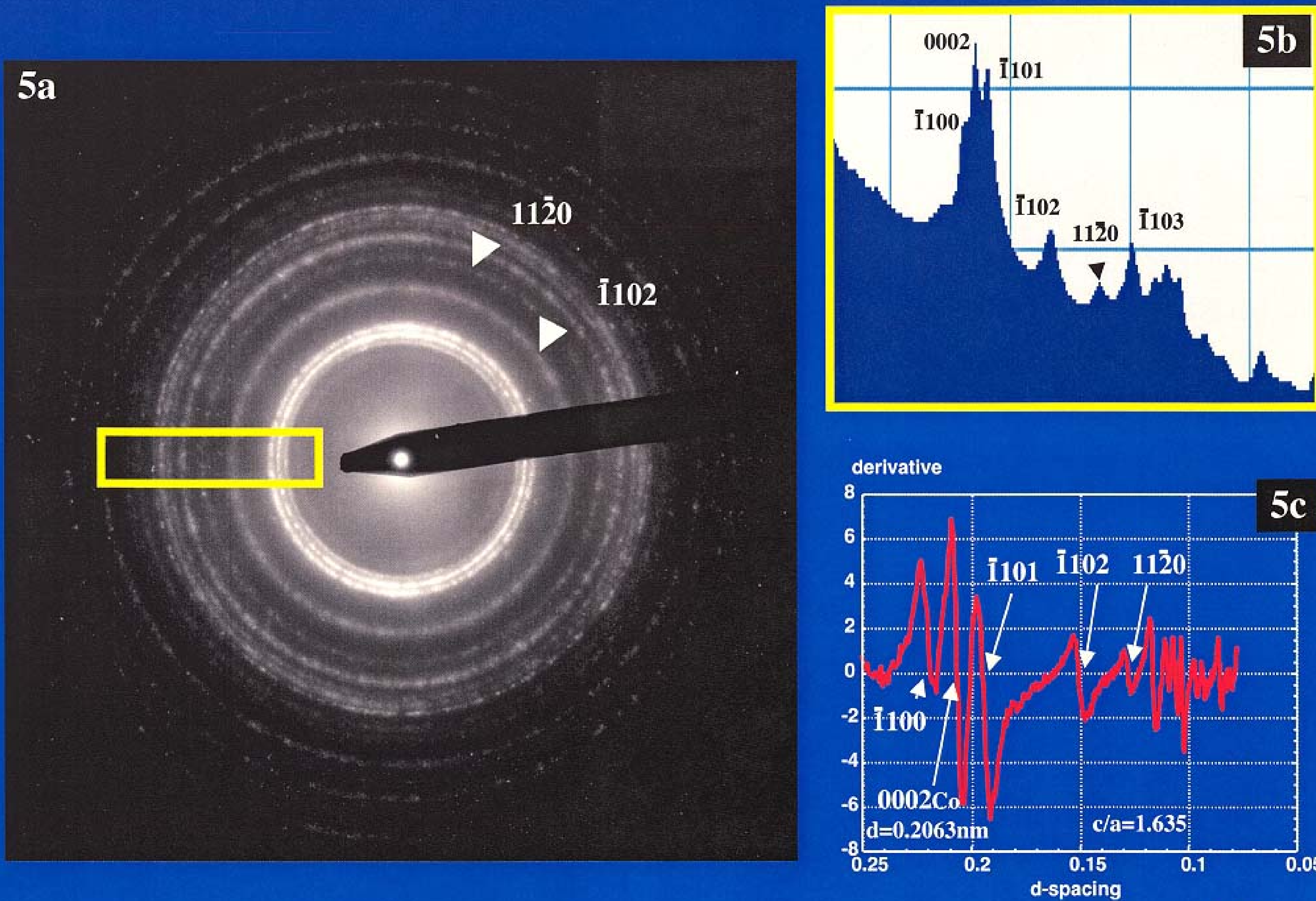

Thin-Film Media
Epitaxial Co
(with Cr, Pt. Ta)
3a
60 nm
3b
1̄103
1̄102
3c
0002
1̄101
1̄100
1̄102
low
112̄0
1̄103
3d
derivative
8
6
4
2
0
-2
-4
-6
-8
0.25
0.2
0.15
0.1
0.05
d-spacing
1̄101
1̄102
low
112̄0
1̄100
0002
3-15-03wmc

Oriented [11$\bar{2}$0] Co-Alloy on [002] Cr



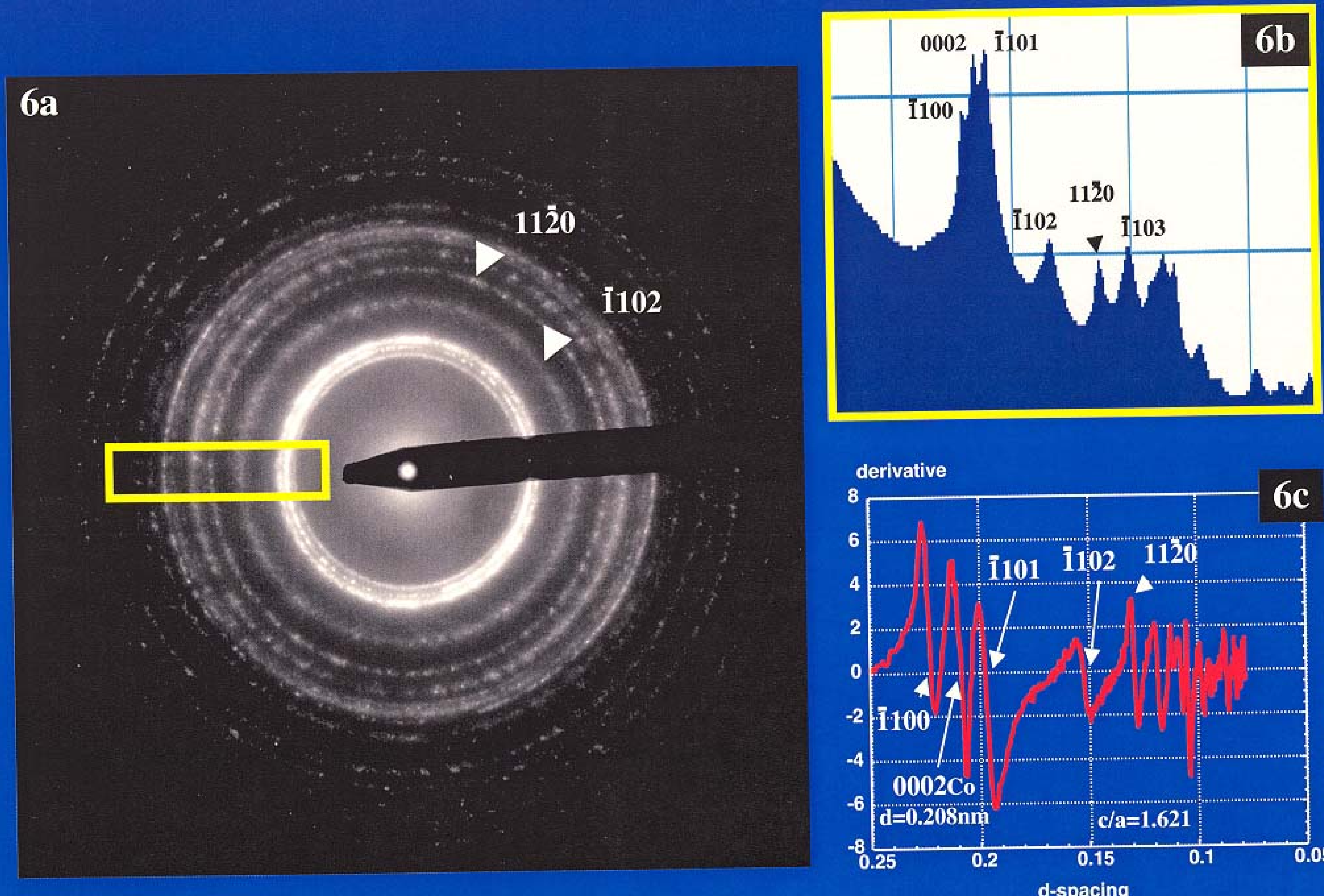
3-15-03wmc

Oriented [11$\bar{2}$0] Co - Alloy with 0% Pt

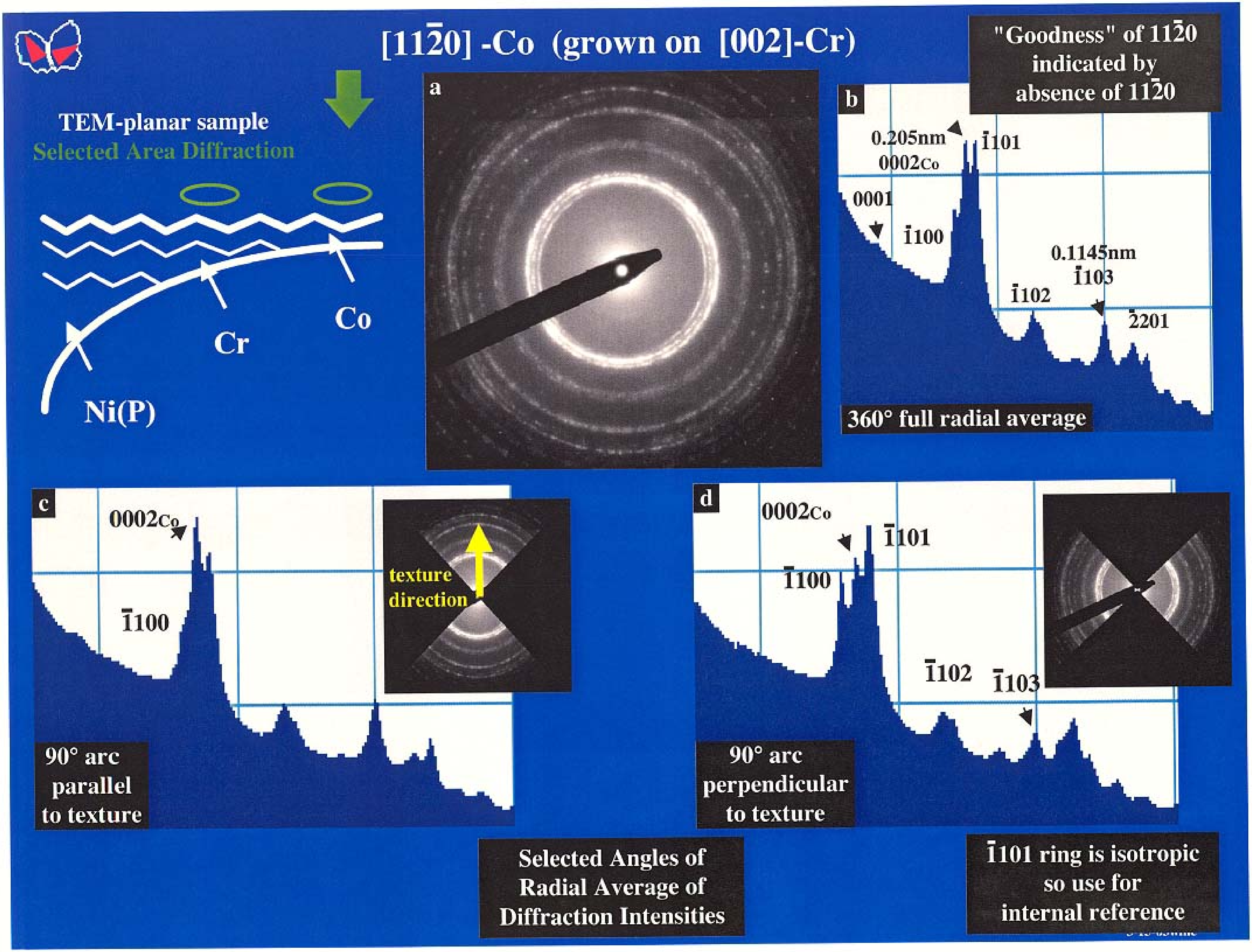


3-15-03wmc

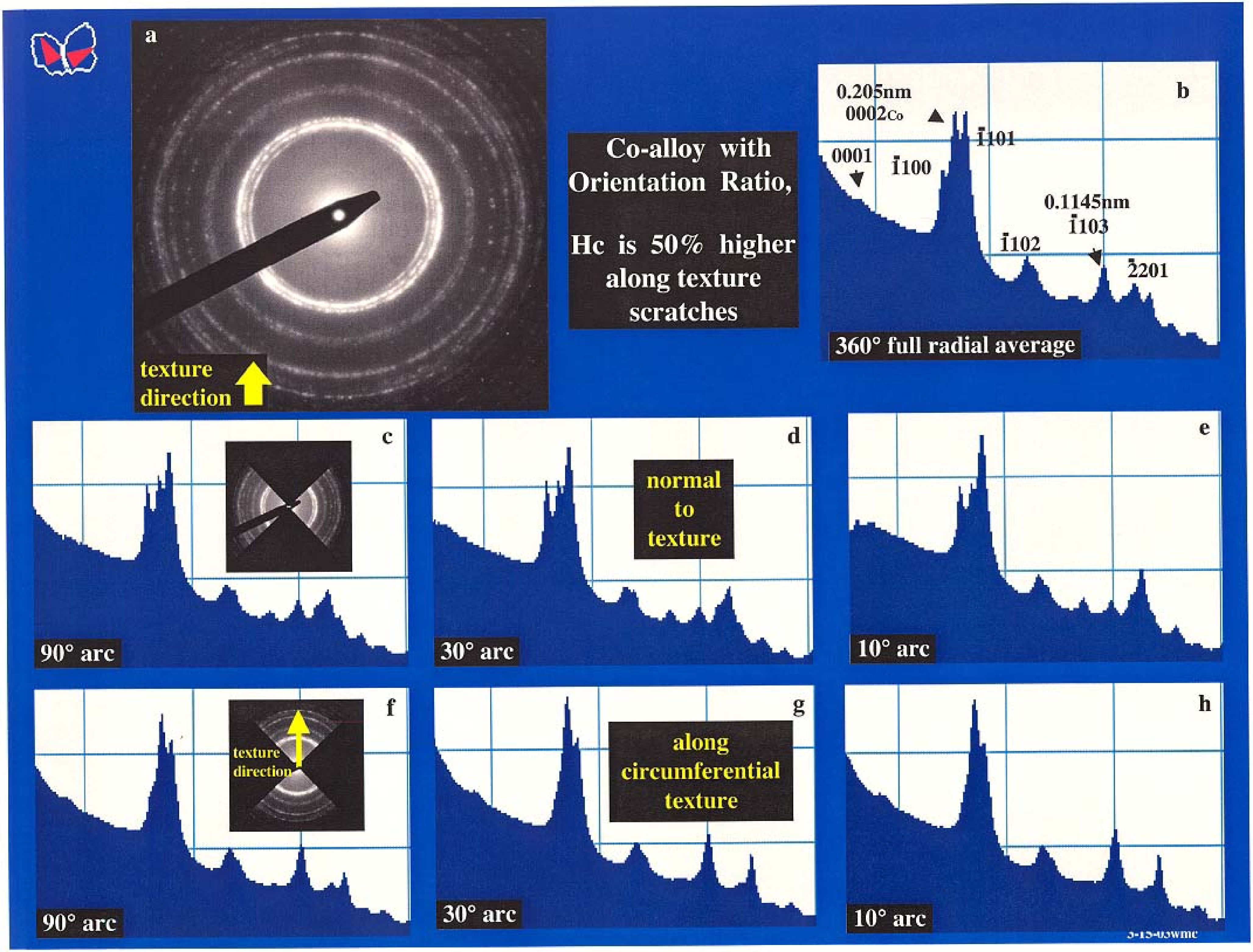
Oriented [11$\bar{2}$0] Co - Alloy with 8% Pt



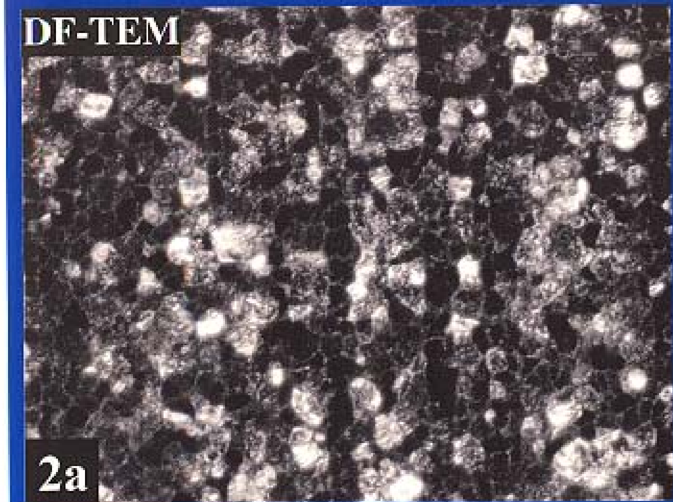
3-15-03wmc

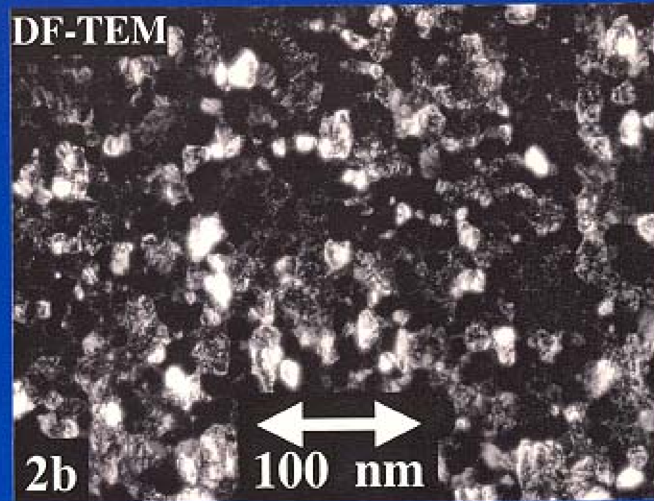


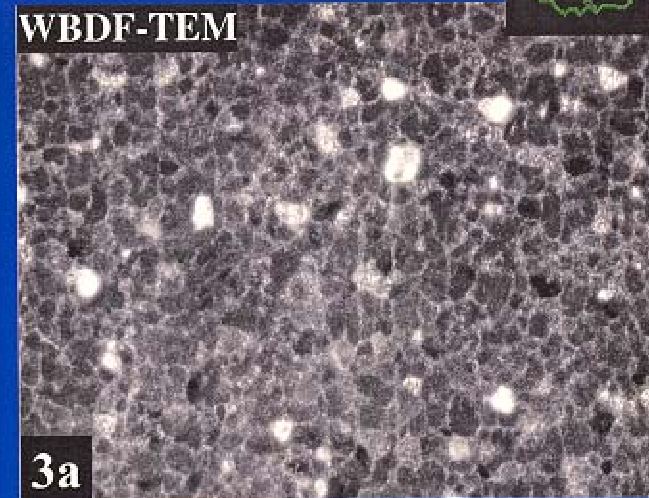
[11$\bar{2}$0] -Co (grown on [002]-Cr)
"Goodness" of 11$\bar{2}$0 indicated by absence of 11$\bar{2}$0
TEM-planar sample
Selected Area Diffraction
Co
Cr
Ni(P)
a
b
0.205nm
0002Co
$\bar{1}$101
0001
$\bar{1}$100
0.1145nm
$\bar{1}$103
$\bar{1}$102
$\bar{2}$201
360° full radial average
c
0002Co
$\bar{1}$100
texture direction
90° arc parallel to texture
d
0002Co
$\bar{1}$101
$\bar{1}$100
$\bar{1}$102
$\bar{1}$103
90° arc perpendicular to texture
Selected Angles of Radial Average of Diffraction Intensities
$\bar{1}$101 ring is isotropic so use for internal reference

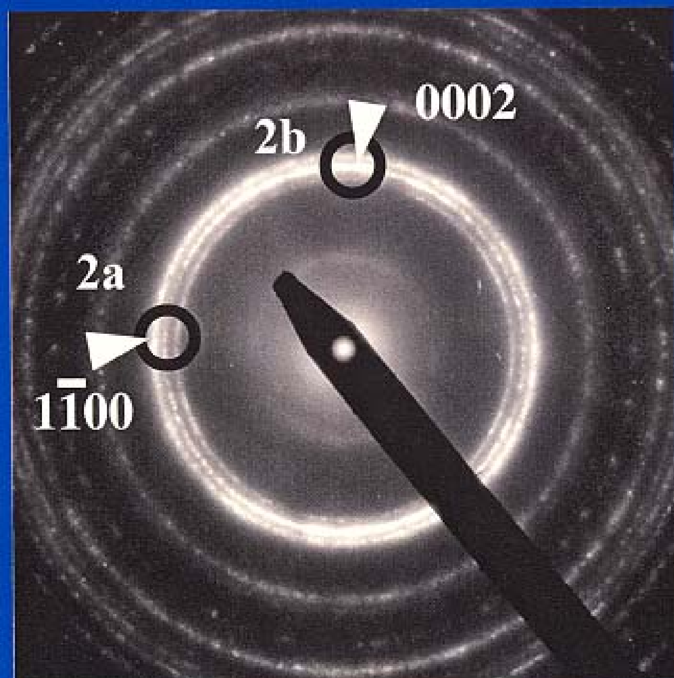

a
texture
direction
Co-alloy with
Orientation Ratio,
Hc is 50% higher
along texture
scratches
b
0.205nm
0002Co
0001
1̄100
1̄101
0.1145nm
1̄103
1̄102
2̄201
360° full radial average
c
90° arc
d
normal
to
texture
30° arc
e
10° arc
f
texture
direction
90° arc
g
along
circumferential
texture
30° arc
h
10° arc

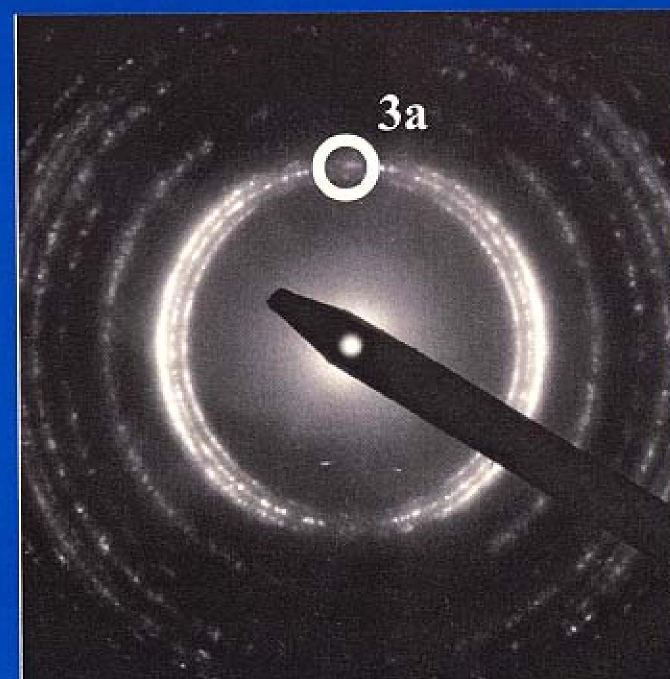
Dark Field Imaging









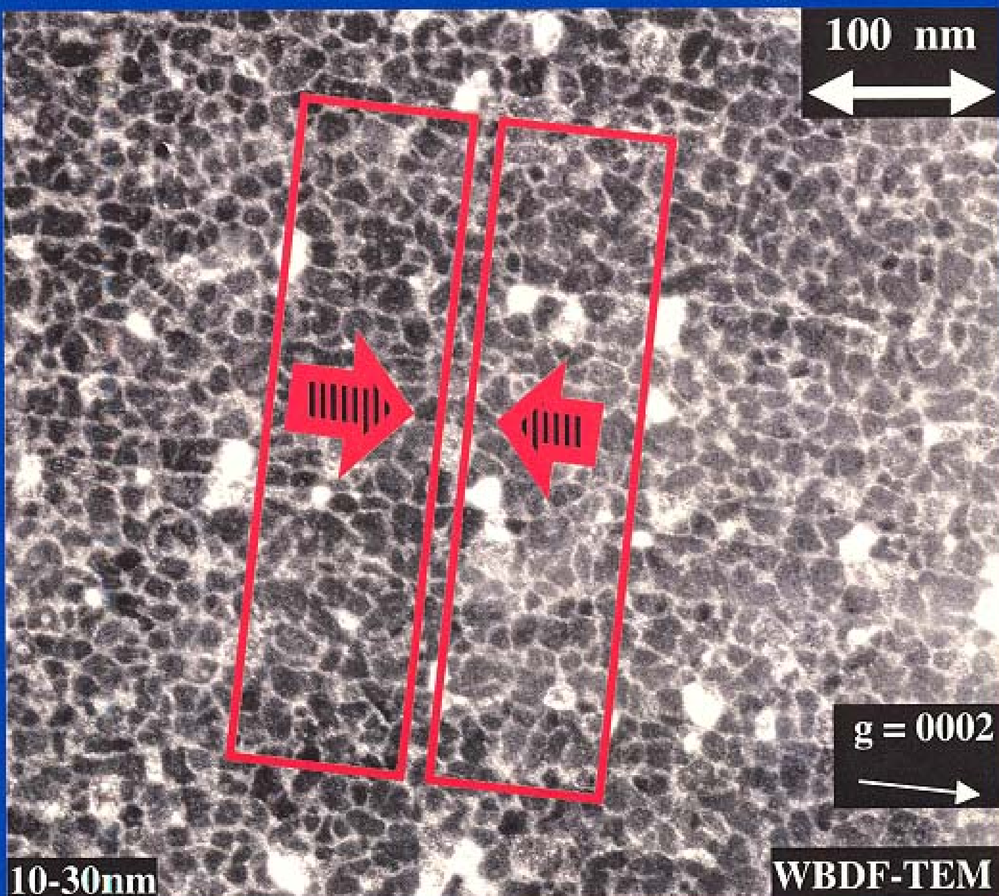


Dark Field Image can be Acquired Either Parallel to Texture Scratches (2a) or Perpendicular (2b - make texture invisible)

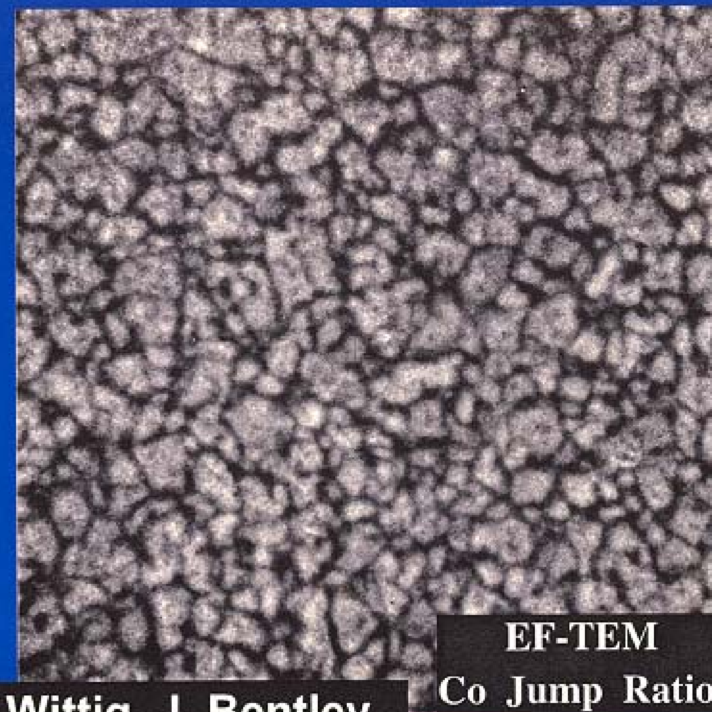
Small Tilt to Non-Diffracting Condition Produces "Weak Beam" Dark Field Image Making Grain Boundary Phase Visible (3a)

3-15-03wmc

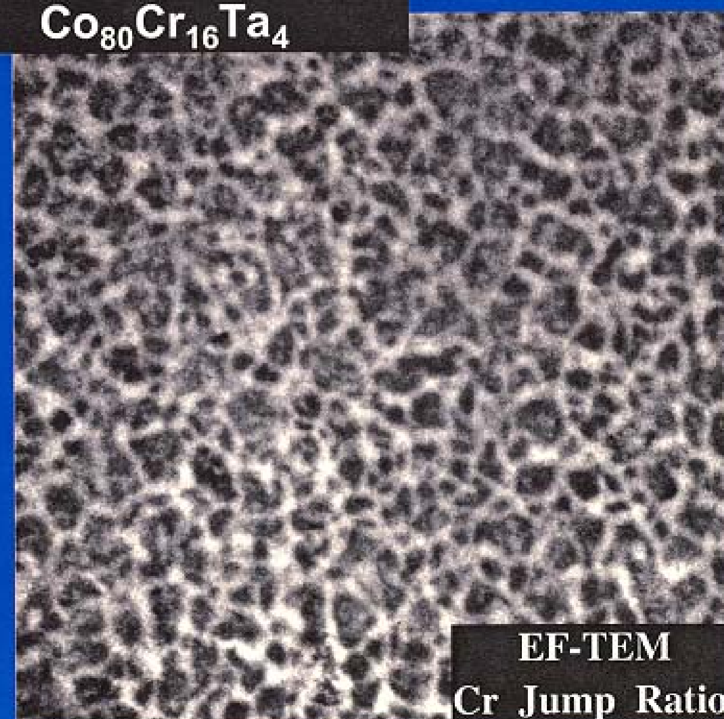
Structural Imaging (Dark Field due to diffraction)



Chemical Imaging (due to inelastic scattering)

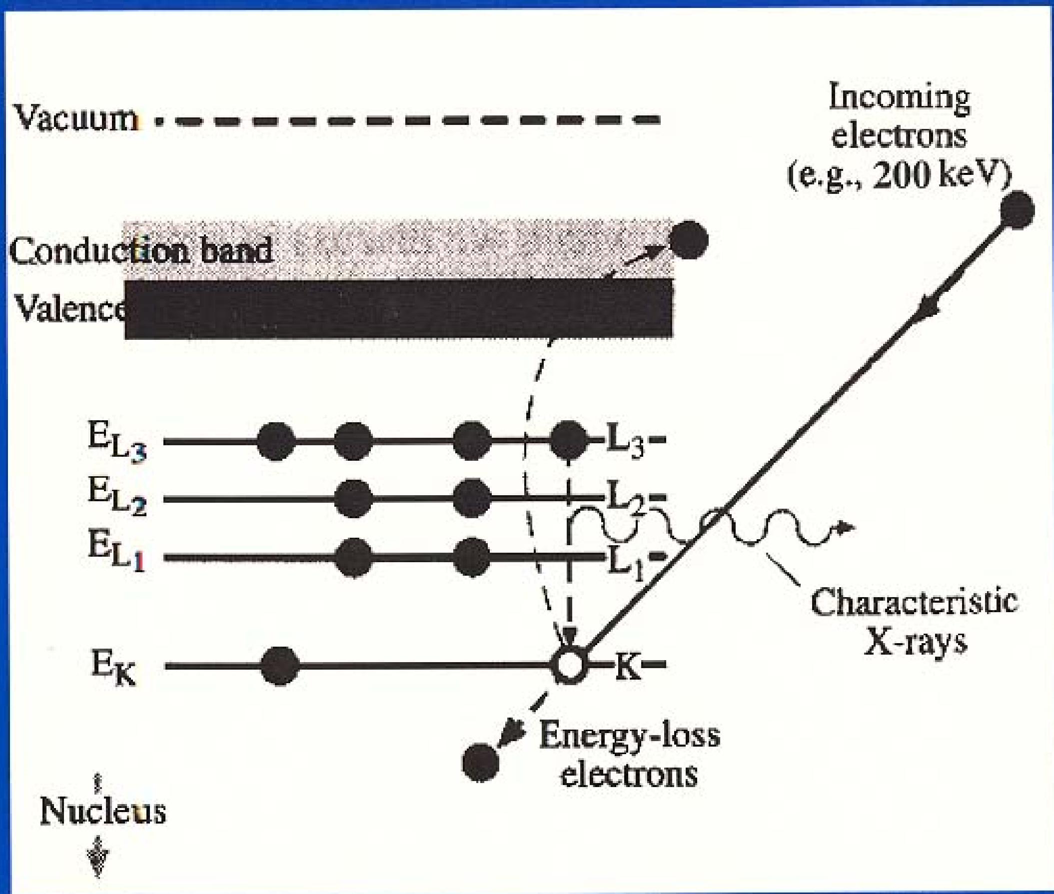


J. Wittig, J. Bentley
$Co_{80}Cr_{16}Ta_4$



3-15-03wmc

Elemental Information by Analytical Electron Microscopy



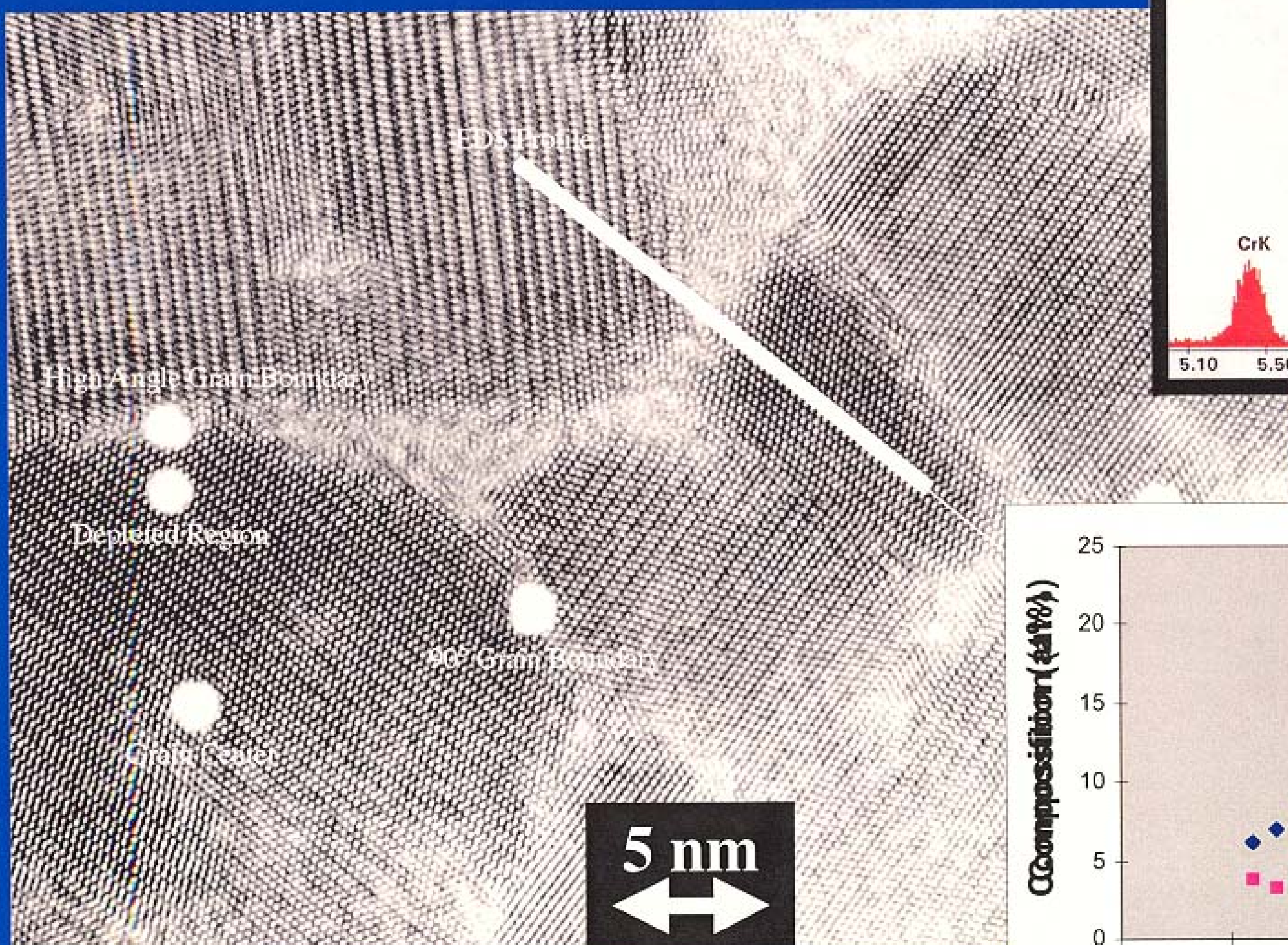
Inelastic Interactions Between Electrons and Specimen Lead to Two Spectroscopies

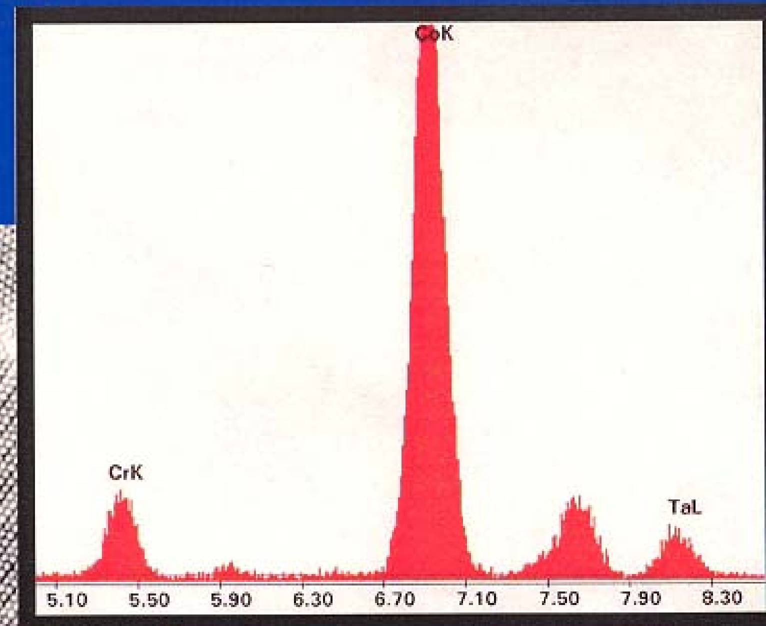
Incoming electron loses energy, Measure the transmitting energy: "Electron Energy Loss Spectroscopy" (EELS, PEELS, EF-TEM)
TEM samples must be thin to prevent double losses

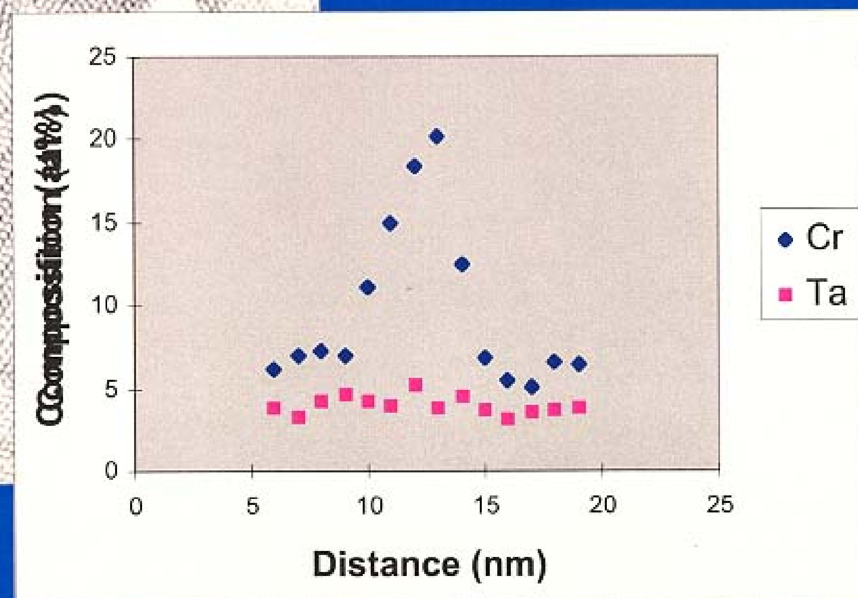
Inner shell ionization relaxes by Emitting characteristic x-ray, Measure x-ray energy: "Energy Dispersive X-Ray Spectroscopy" (EDS, EDX, WDS, X-Ray Mapping)

EDS X-ray Analysis

Use small beam to analyze local region, "nanoprobe"
Scan probe to provide chemical "line profile" (or mapping)

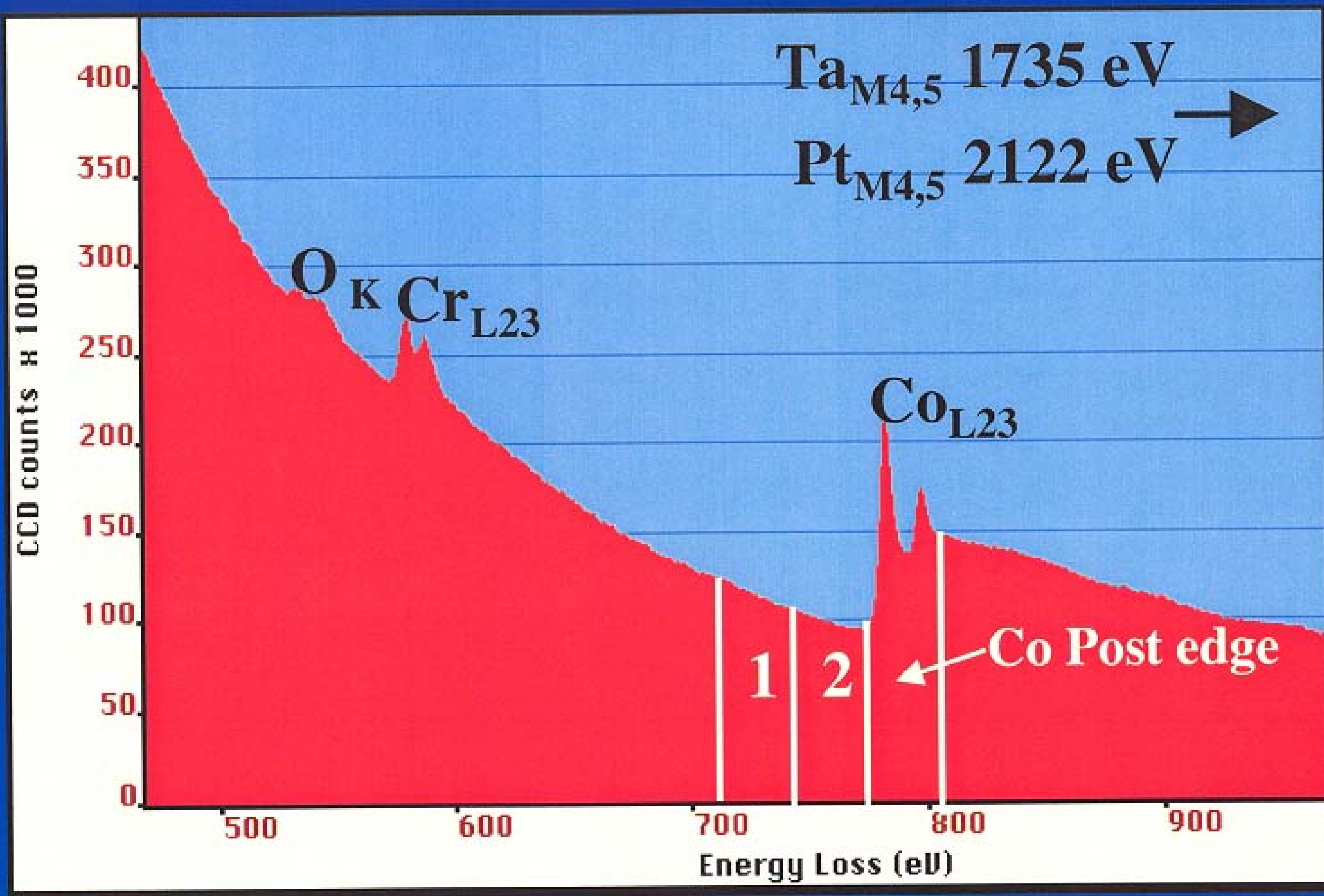






Jim Wittig, Vanderbilt
Jim Bentley, ORNL

PEELS (Energy Loss) Analysis

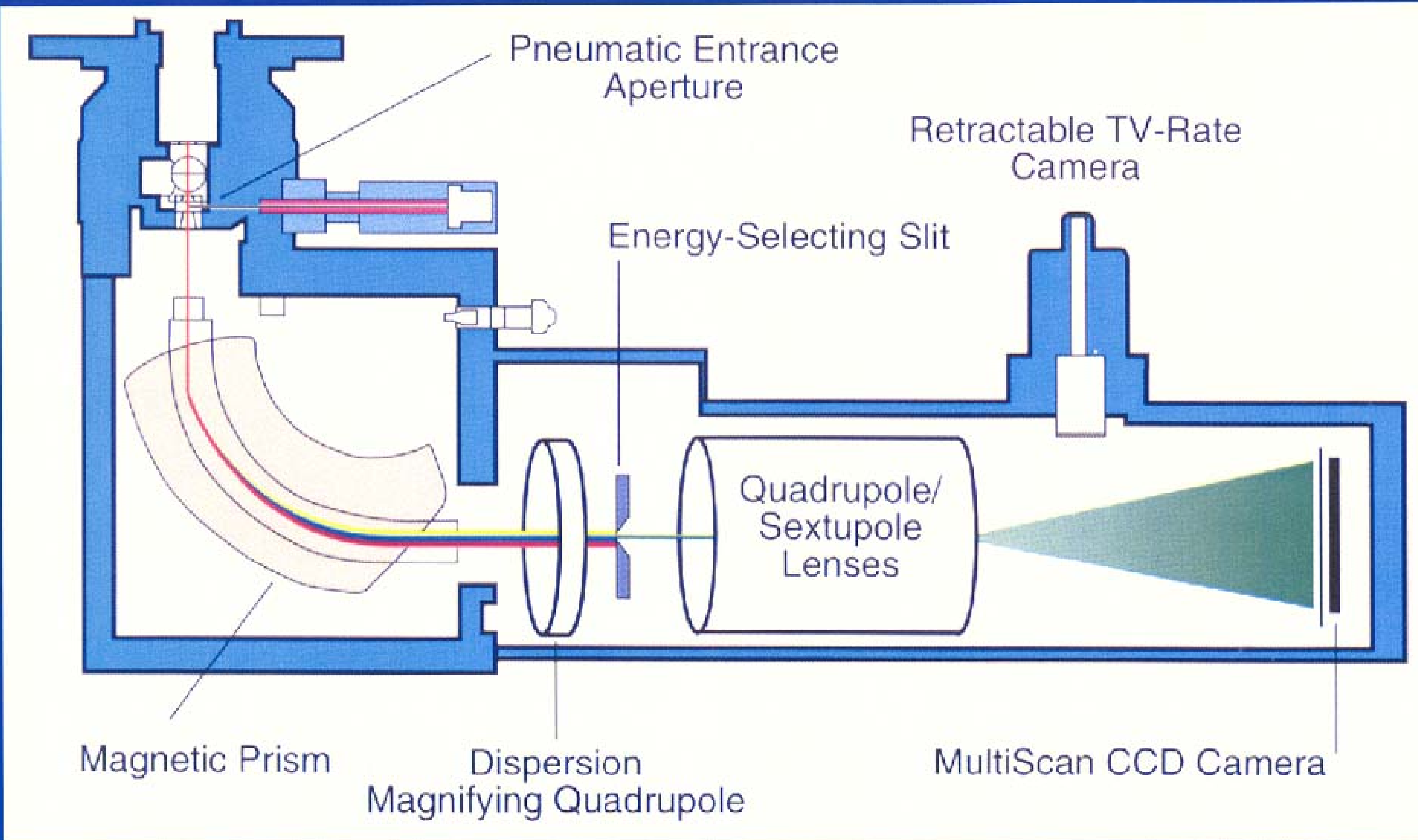


Jim Wittig, Vanderbilt
Jim Bentley, ORNL

EELS Spectrum detects core-loss edges
More sensitive than EDS to low-atomic-number elements
(energies comparable to Auger, with large background)
Model background with pre-edge windows
Collect Images of "pre" and "post" edge and subtract (or ratio)

3-15-03wmc

EF-TEM (Energy Filtered) / GIF (Gatan Imaging Filter)



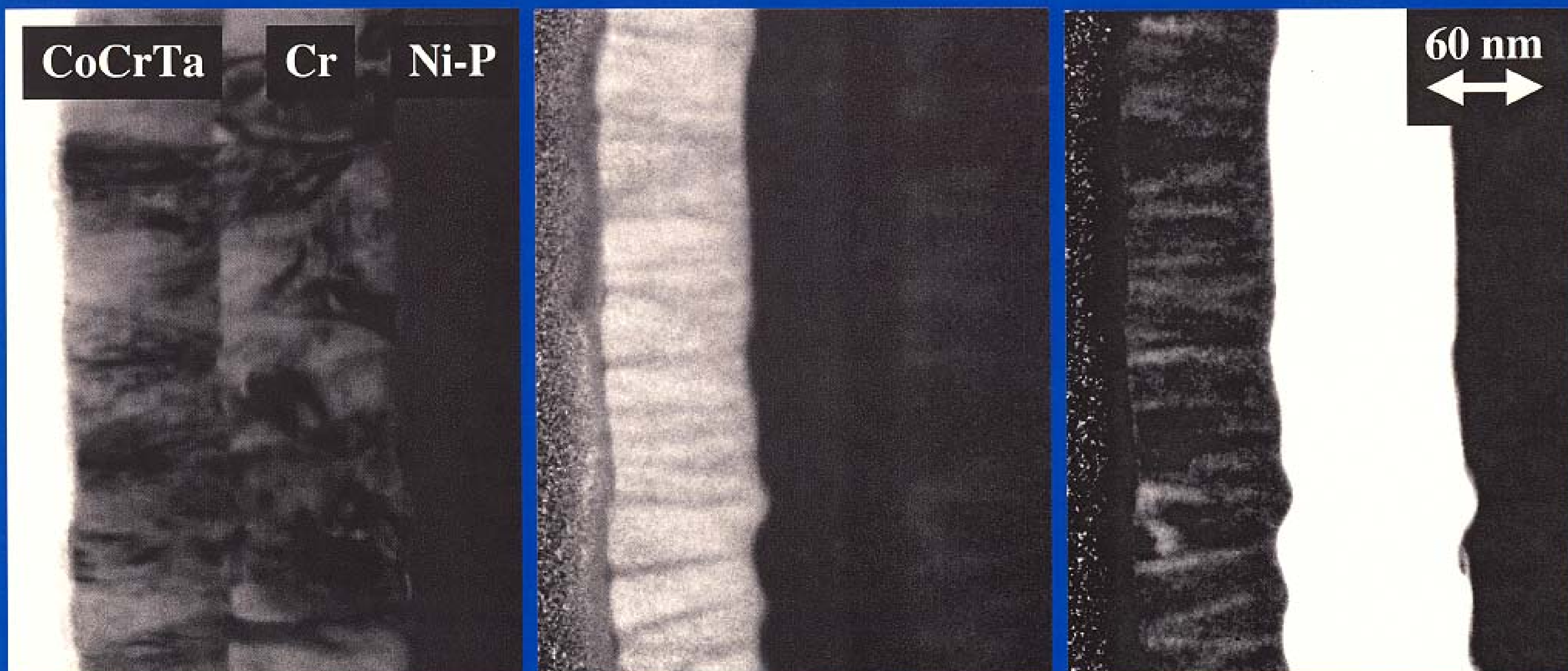
Energy losses small (<3KeV), so transmitted by lenses in lower TEM
GIF disperses and then filters the different energies
And lenses in GIF reform (chemical) image onto CCD

3-15-03wmc

Cross-sectioned magnetic recording media

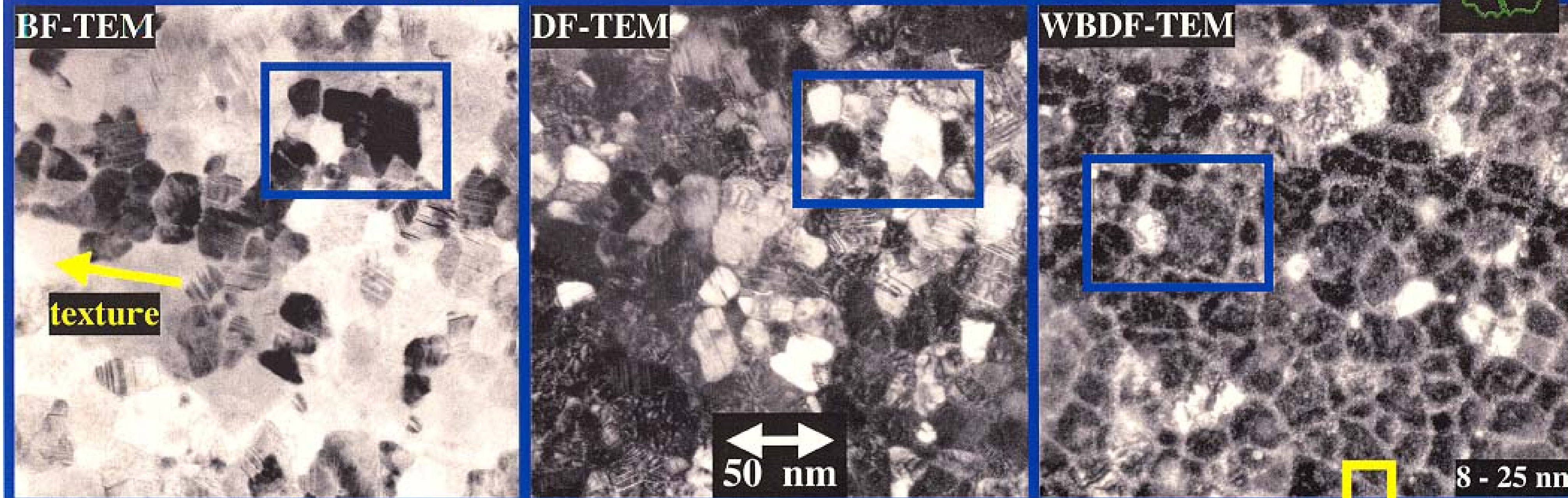
EF-TEM reveals intergranular segregation between columnar grains

zero loss | Co jump ratio | Cr jump ratio



Jim Wittig, Vanderbilt
Jim Bentley, ORNL
3-15-03wmc

Dark Field Imaging & Cross Correlation to Measure Grain Size



0.4

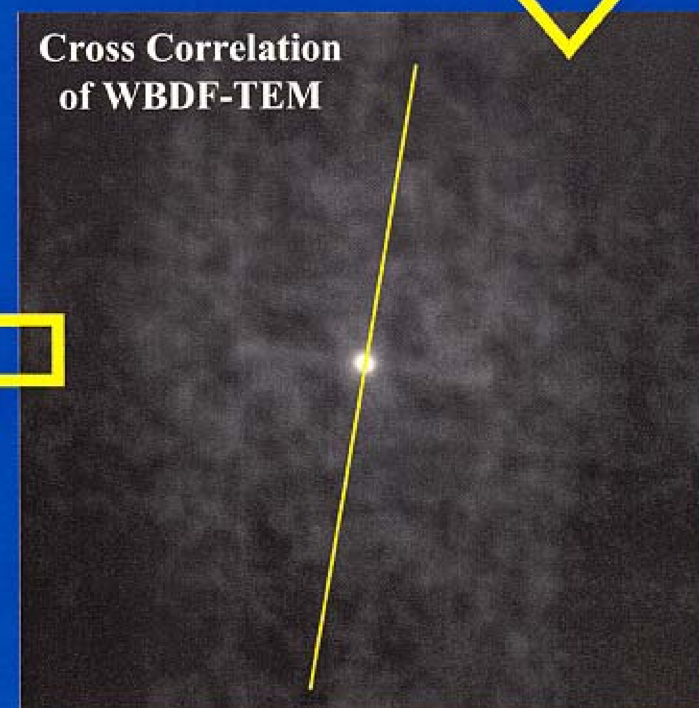
central peak
truncated

central peak width
α segregation width

fine peak spacing
α grain size

large peak spacing
α texture spaces

0

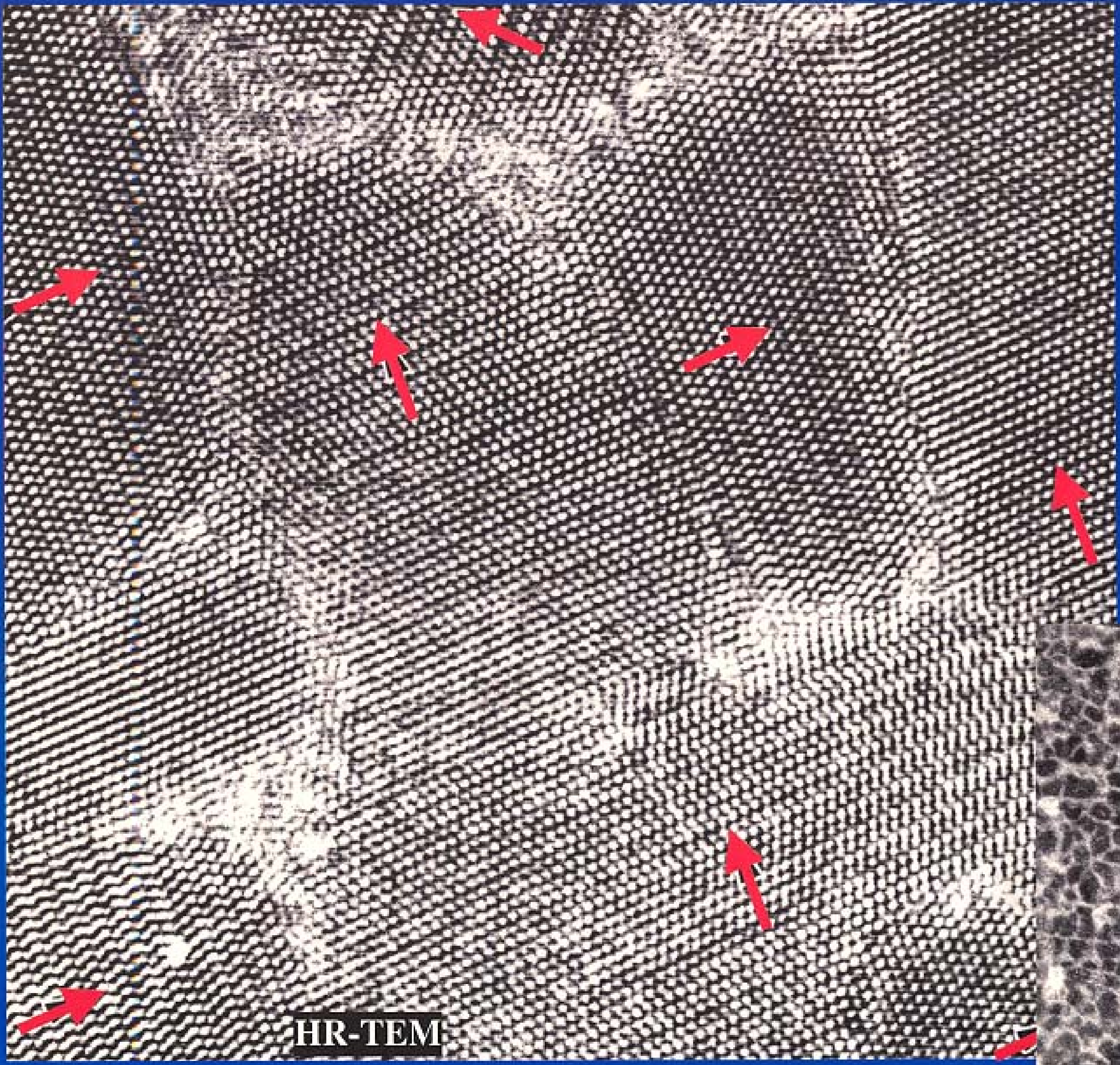


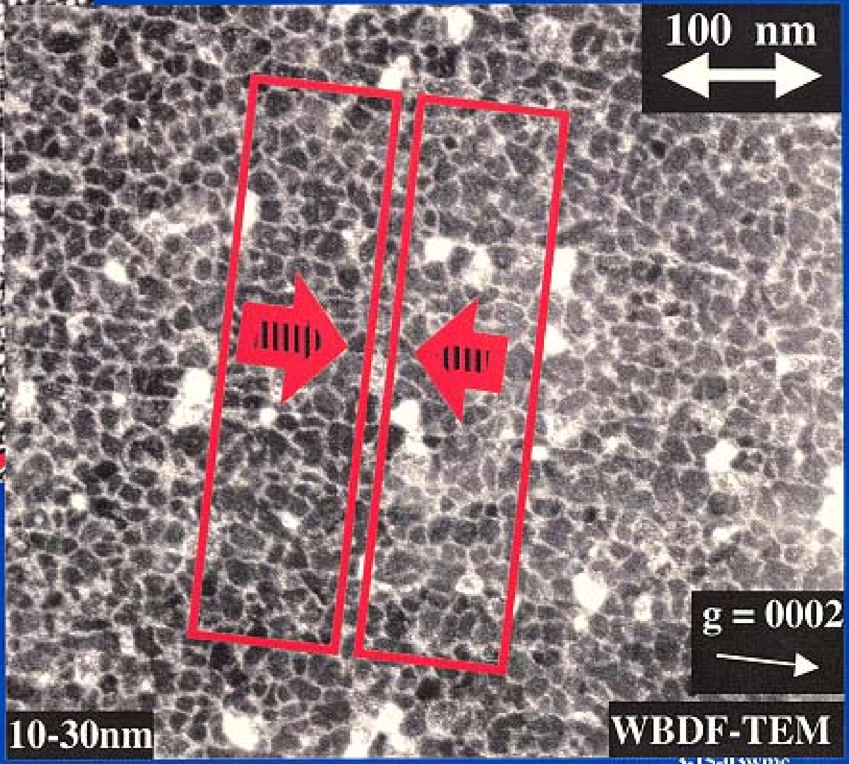
3-15-03wmc

To increase storage density
Need smaller grains
For a sharper transition between bits

But small grains have low Hc,
Are thermally less magnetically stable,
And also become superparamagnetic

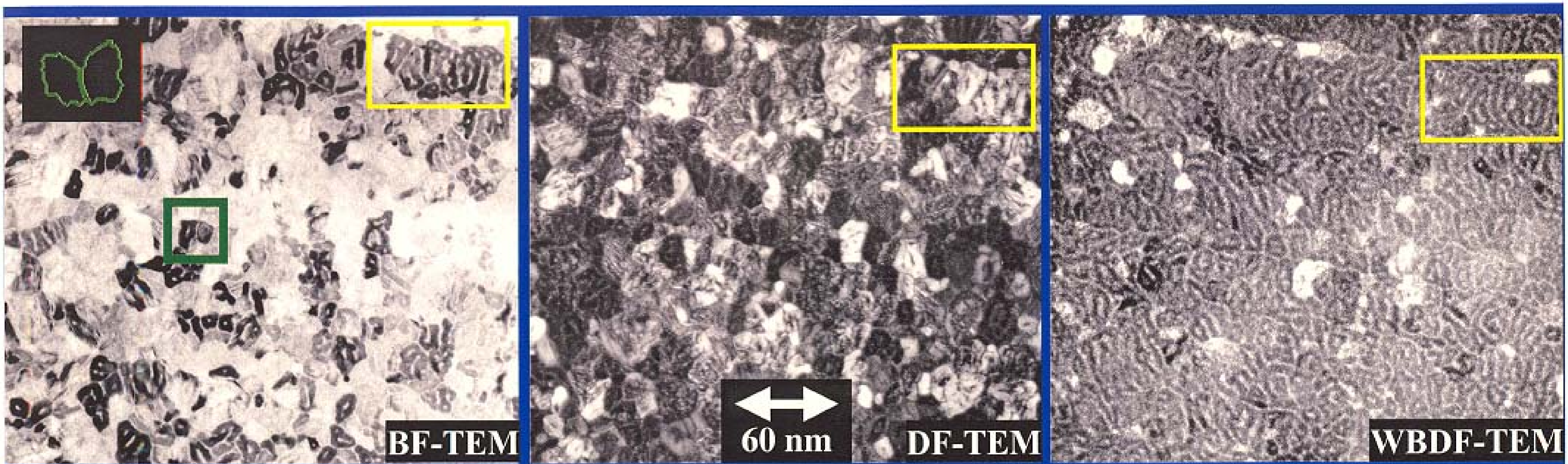
To make small grains requires thin films
(Bad: Lower signal, Harder to process)
(Good: Reduce head/media spacing)



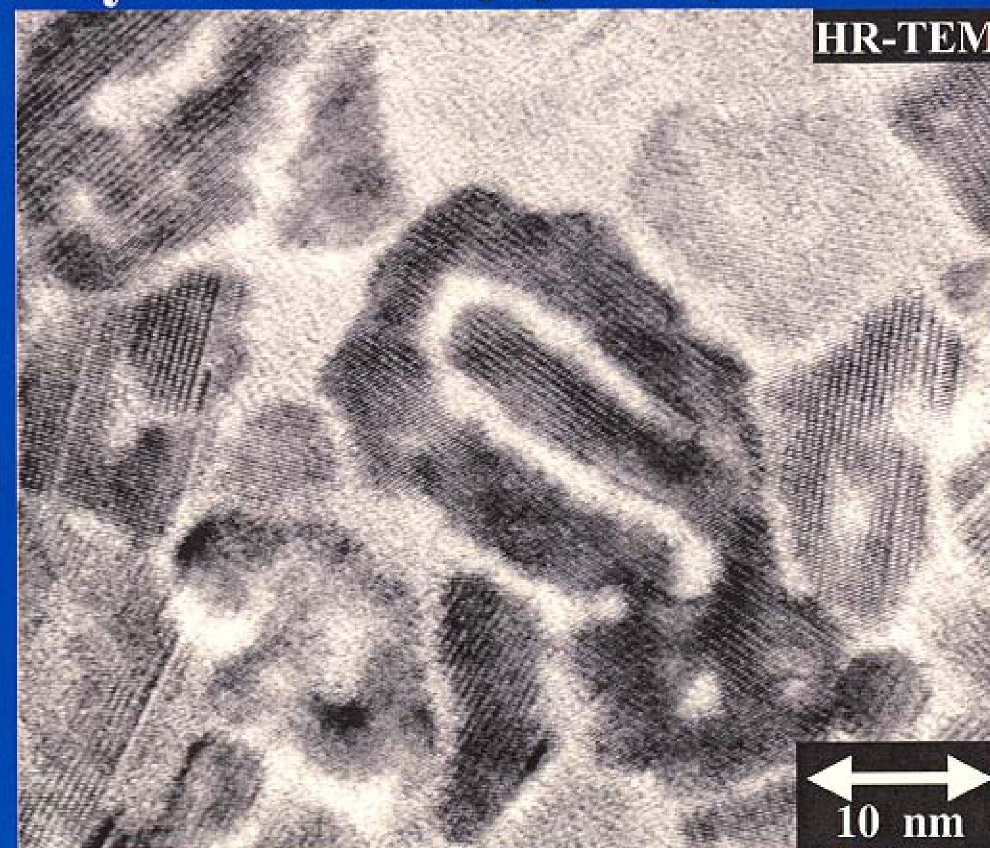


Recording signal limited by transition noise,
Need to improve separation between grains

Design microstructure to optimize separation between grains

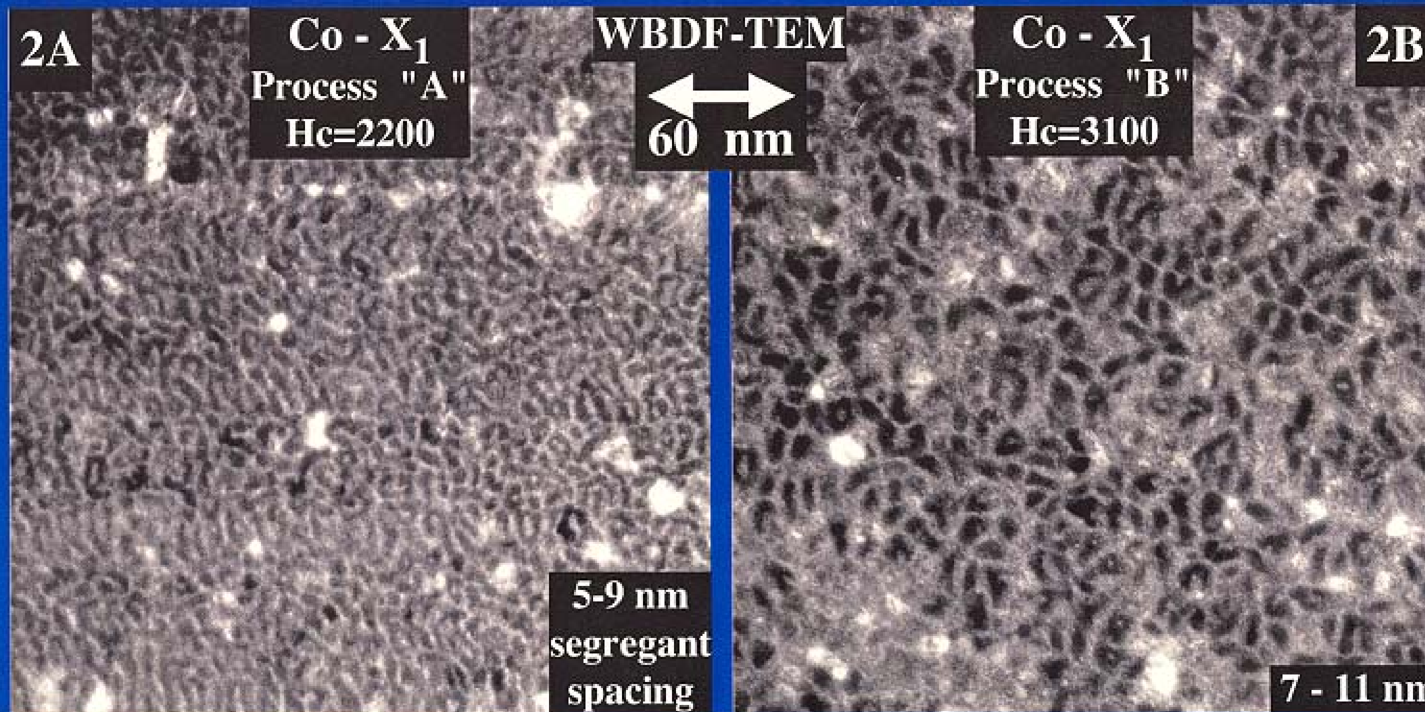


Co - alloy Media (w/ segregated 2nd phase)

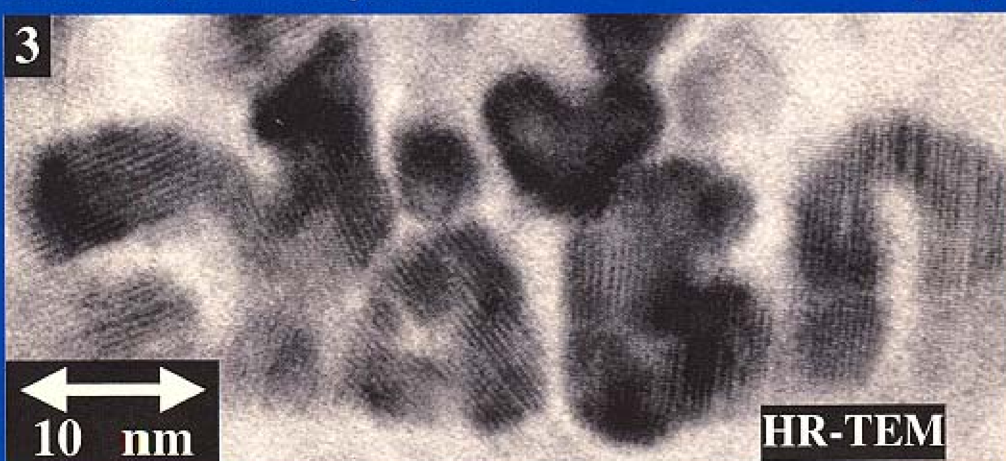


Co - X Alloy

High X% means more segregant & smaller spacing
(to decrease media noise & increase storage density)



However, some bicrystals can be connected around segregant



Co - X_1 has constant X%

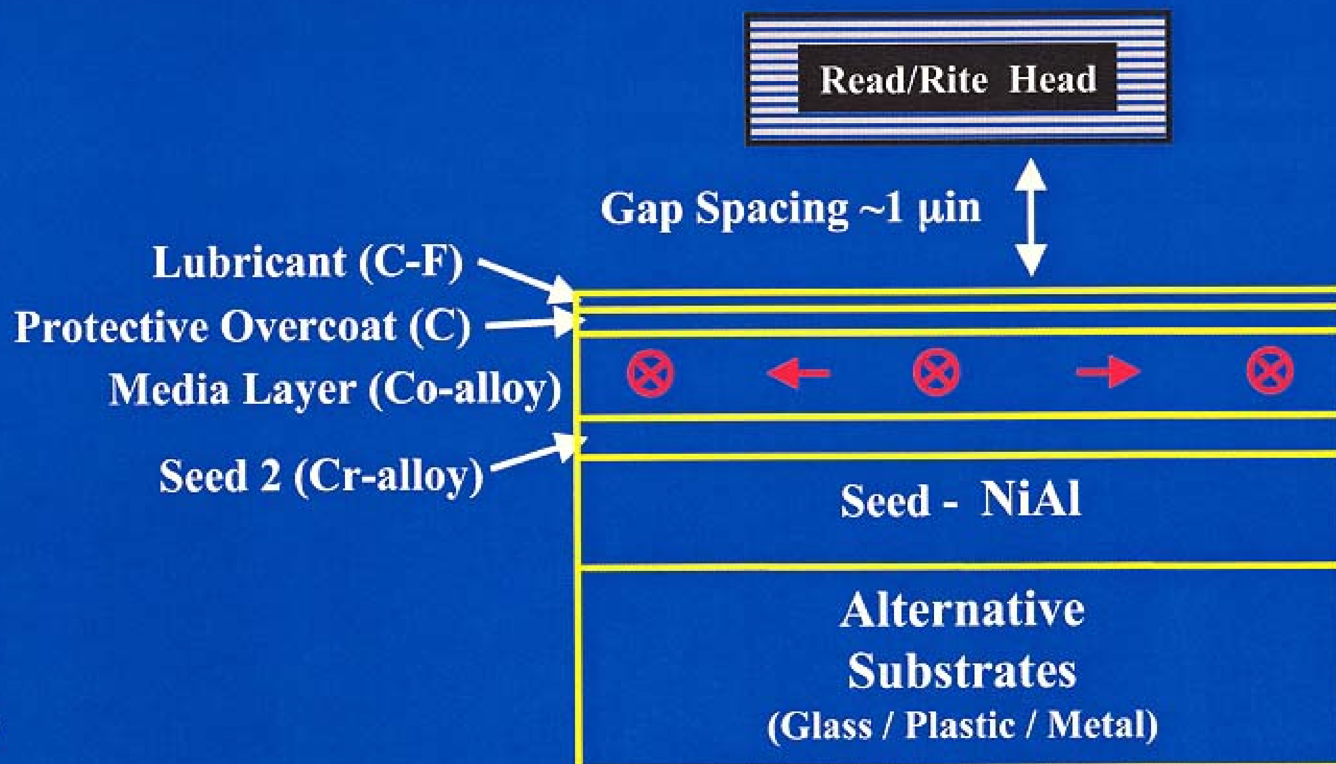
Change process from "A" to "B" causes larger spacing of segregant, *but* more discrete;

Thus "B" has lower media noise (and <u>higher</u> <u>coercivity</u>), even with larger grains

3-15-03wmc

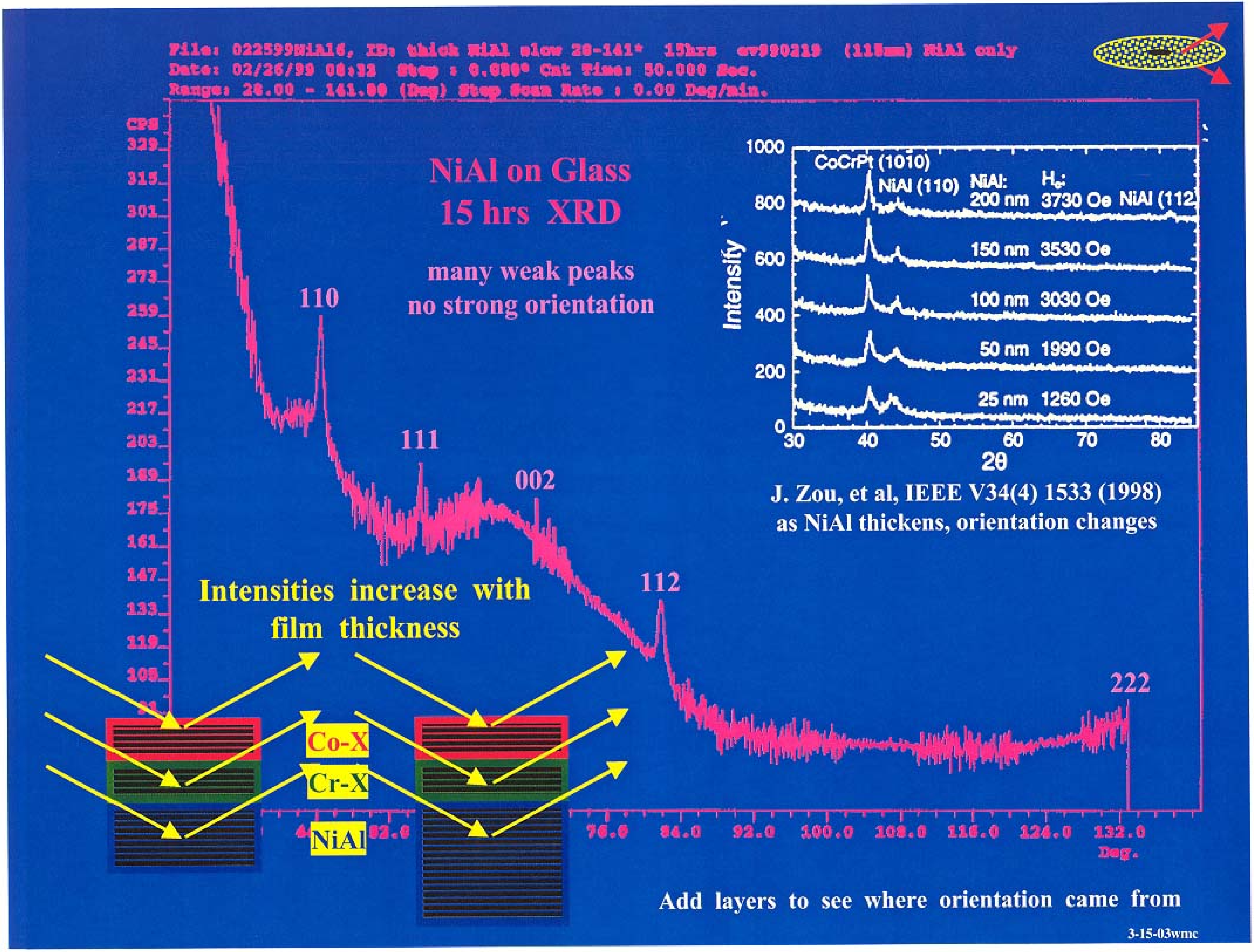
Impact Resistance in Portable Drives & Higher Speed Require Harder (& Thinner) Substrates

Longitudinal Winchester Media

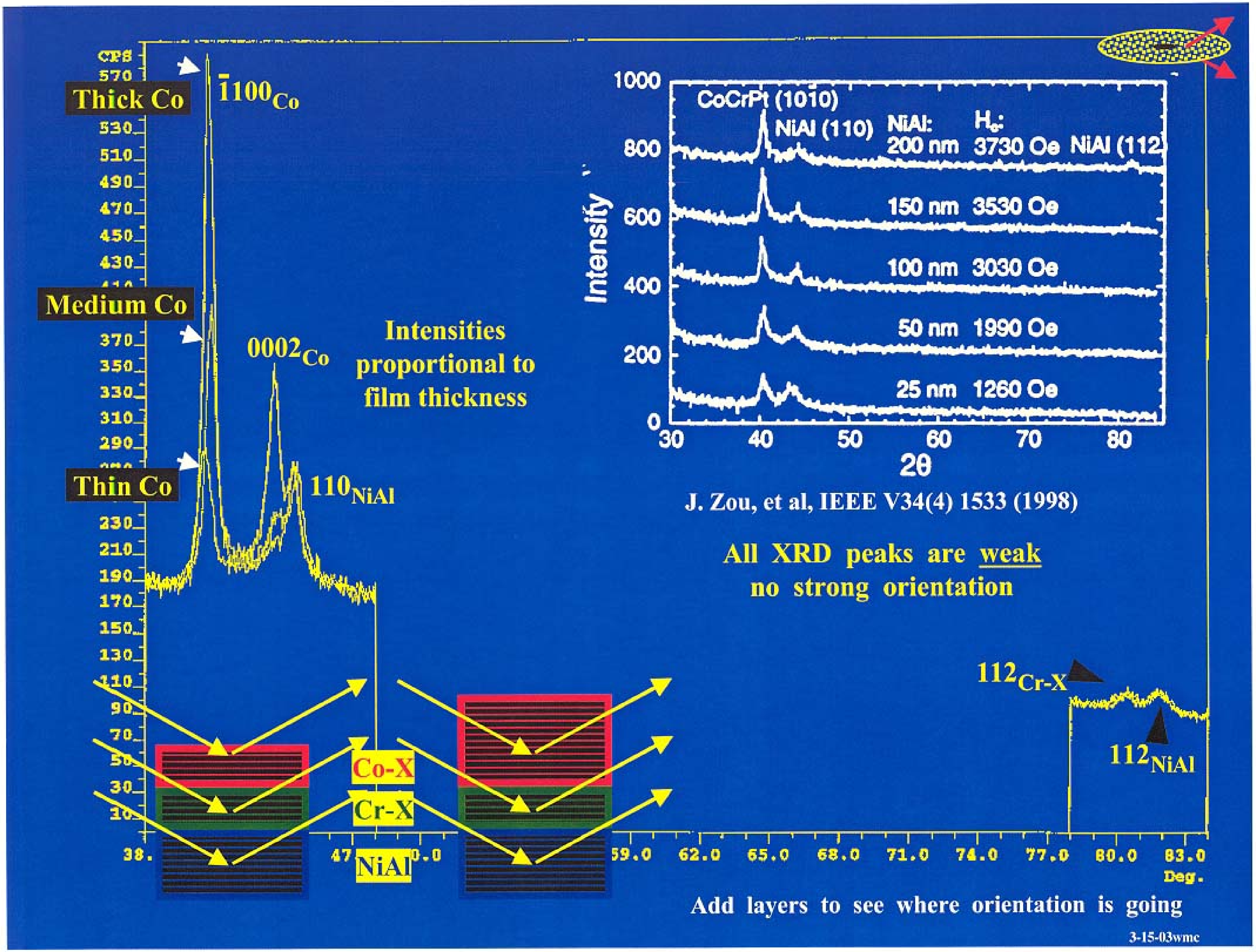


Why NiAl Seed Layer?

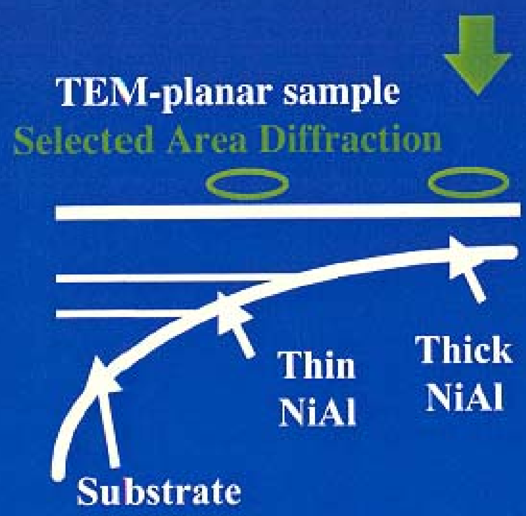
a) cover "any" substrate
b) smaller grains:
lower media noise,
higher storage density
c) same d-spacing as Cr

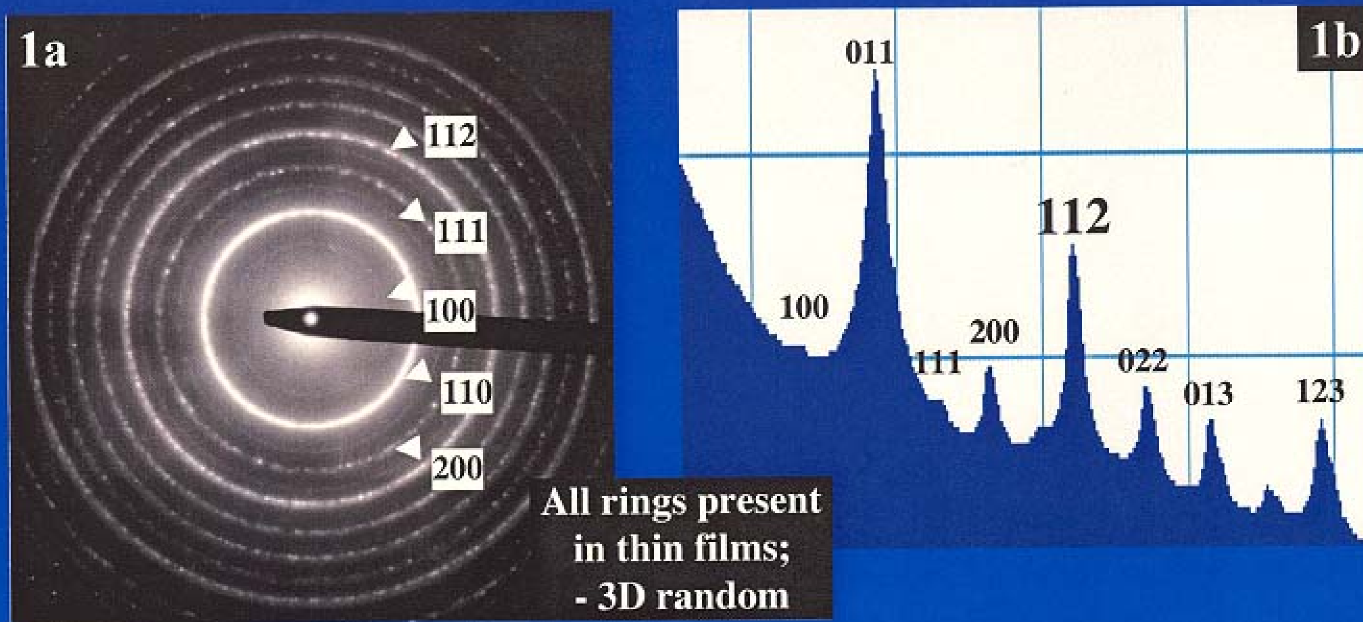


NiAl on Glass
15 hrs XRD
many weak peaks
no strong orientation
110
111
002
112
222
Intensities increase with film thickness
Co-X
Cr-X
NiAl
CPS
Deg.
CoCrPt (1010)
NiAl (110)
NiAl:
Hc:
200 nm 3730 Oe
NiAl (112
150 nm 3530 Oe
100 nm 3030 Oe
50 nm 1990 Oe
25 nm 1260 Oe
Intensity
2θ
J. Zou, et al, IEEE V34(4) 1533 (1998)
as NiAl thickens, orientation changes
Add layers to see where orientation came from
3-15-03wmc

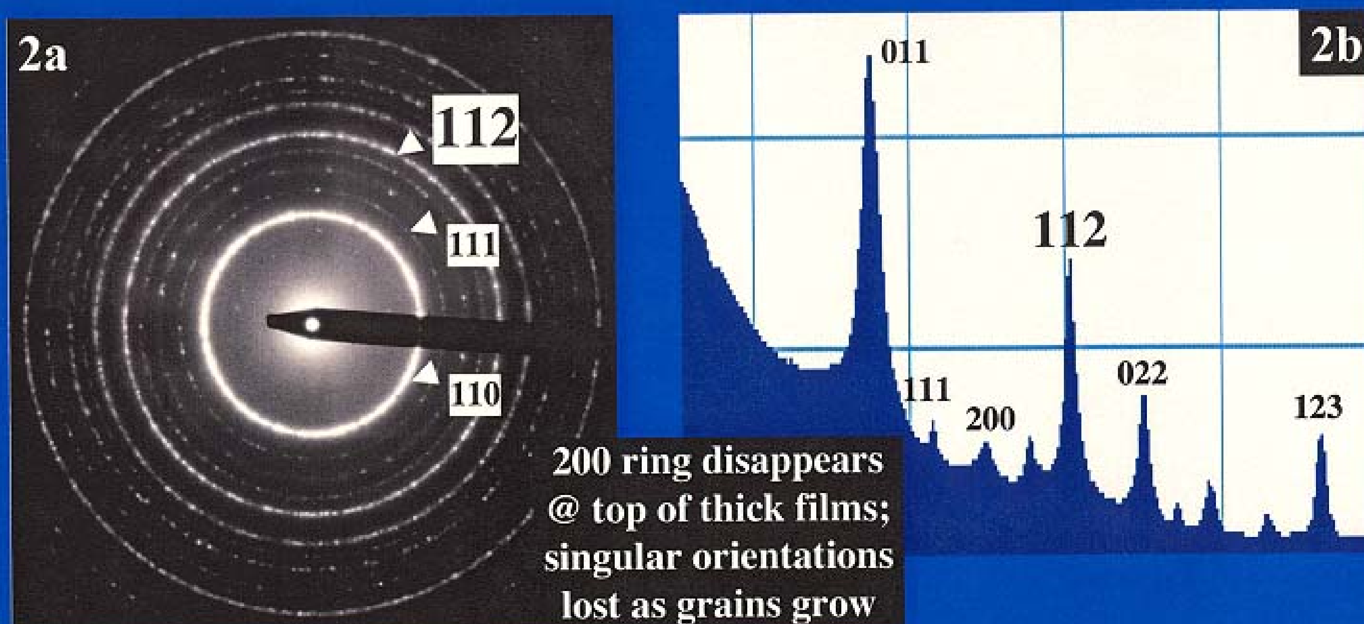

CPS
570
530
510
490
470
450
430
370
350
330
310
290
230
210
190
170
150
130
110
90
70
50
30
10
Thick Co
$\bar{1}100_{Co}$
Medium Co
Thin Co
0002_{Co}
110_{NiAl}
Intensities proportional to film thickness
Co-X
Cr-X
NiAl
38.
59.0
62.0
65.0
68.0
71.0
74.0
77.0
80.0
83.0
Deg.
CoCrPt (10$\bar{1}$0)
NiAl (110)
NiAl:
H_c:
200 nm 3730 Oe
NiAl (112
150 nm 3530 Oe
100 nm 3030 Oe
50 nm 1990 Oe
25 nm 1260 Oe
Intensity
1000
800
600
400
200
0
30
40
50
60
70
80
2θ
J. Zou, et al, IEEE V34(4) 1533 (1998)
All XRD peaks are weak
no strong orientation
112_{Cr-X}
112_{NiAl}
Add layers to see where orientation is going
3-15-03wmc

Electron Diffraction & Radial Average of NiAl Films





All rings present in thin films; - 3D random



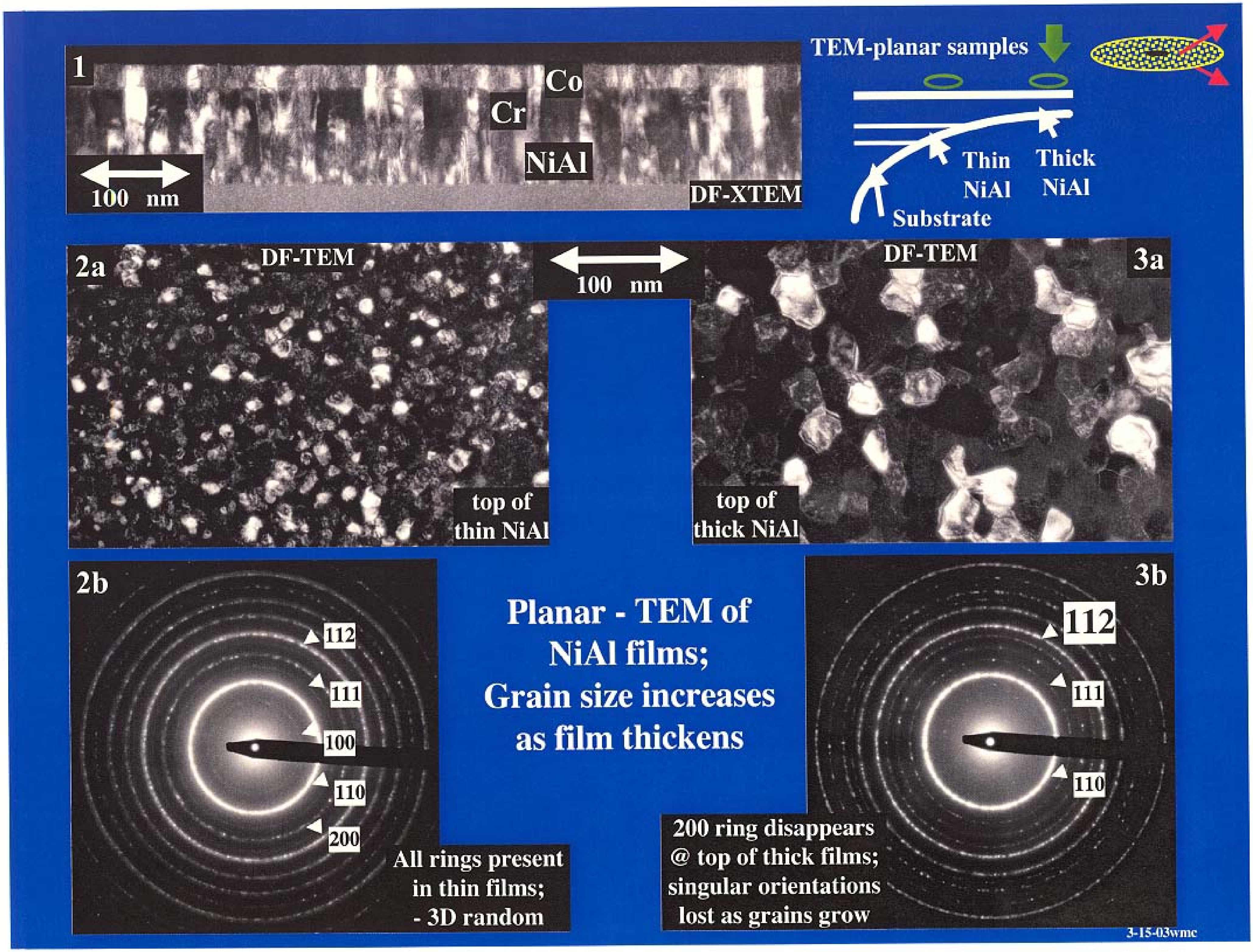
200 ring disappears @ top of thick films; singular orientations lost as grains grow

1) ED - RA has more signal than XRD

2) ED can selectively sample the topmost of a film

3) ED is like GIX: samples in-plane spacings which control epitaxy

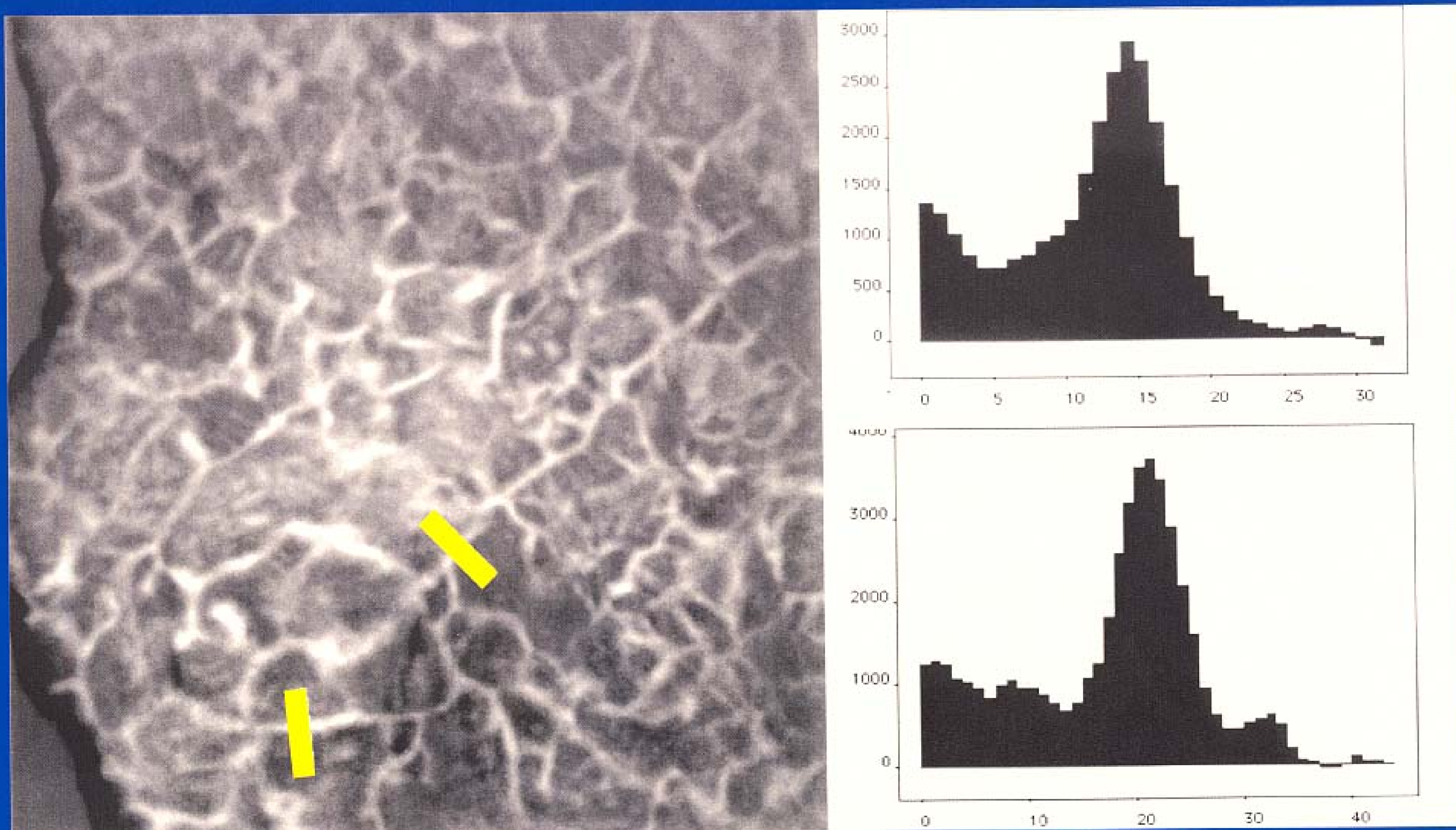
3-15-03wmc



1
Co
Cr
NiAl
100 nm
DF-XTEM
TEM-planar samples
Thin NiAl
Thick NiAl
Substrate
2a
DF-TEM
100 nm
DF-TEM
3a
top of thin NiAl
top of thick NiAl
2b
112
111
100
110
200
All rings present in thin films; - 3D random
Planar - TEM of NiAl films; Grain size increases as film thickens
3b
112
111
110
200 ring disappears @ top of thick films; singular orientations lost as grains grow
3-15-03wmc

Energy - Filtered Cr Image

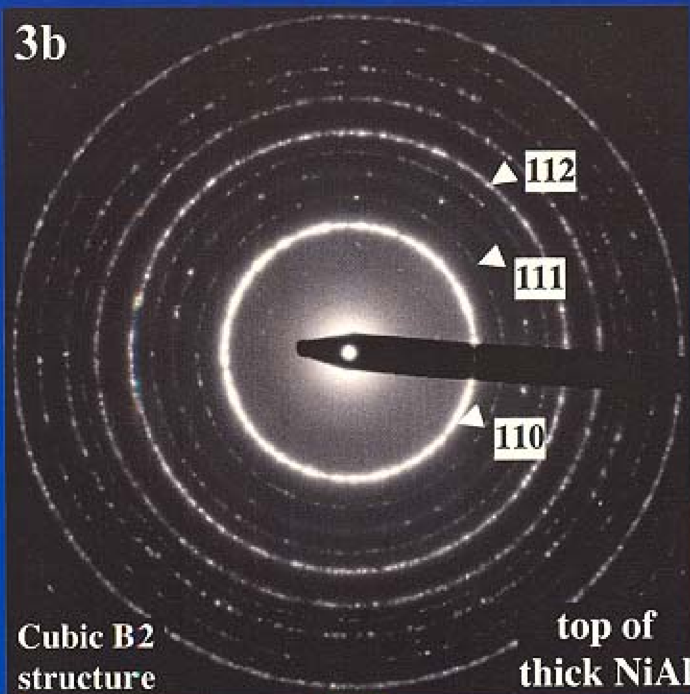
With computer-generated grain boundary profiles

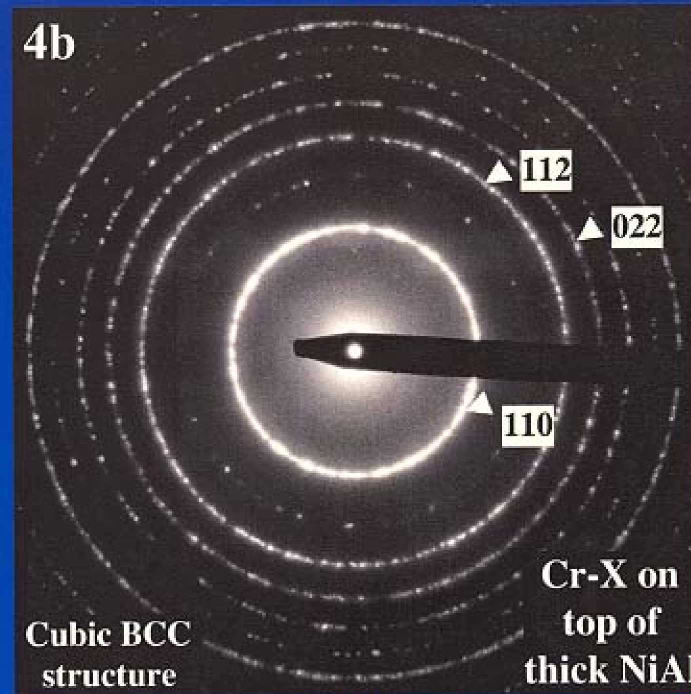


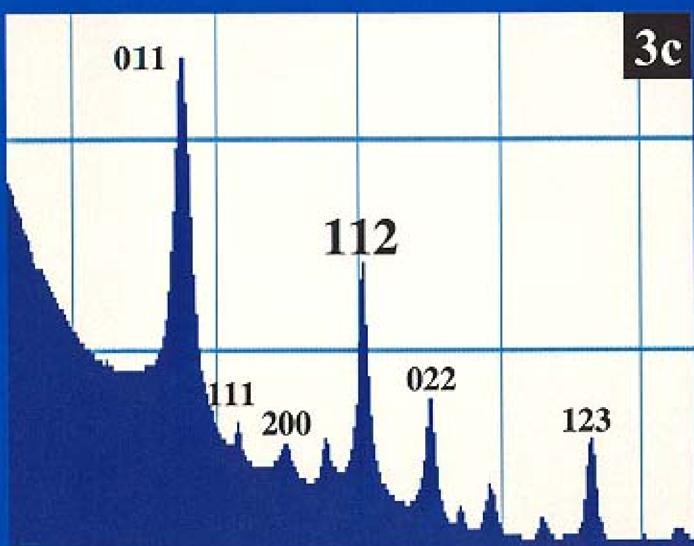
Jim Wittig, Vanderbilt
Jim Bentley, ORNL

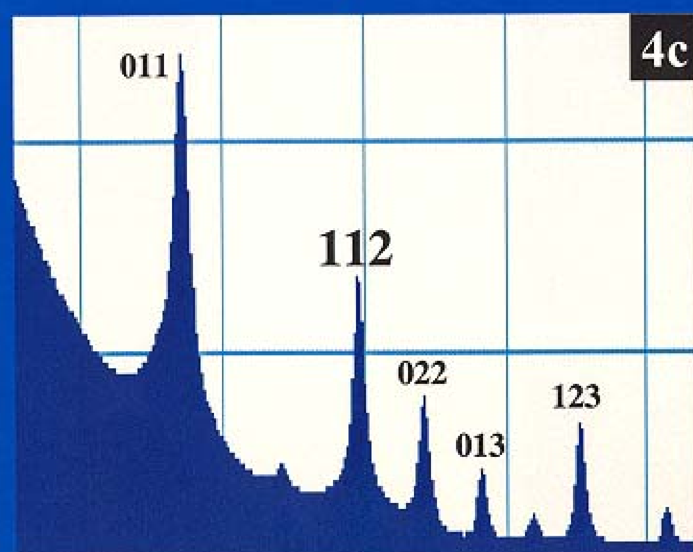
3-15-03wmc

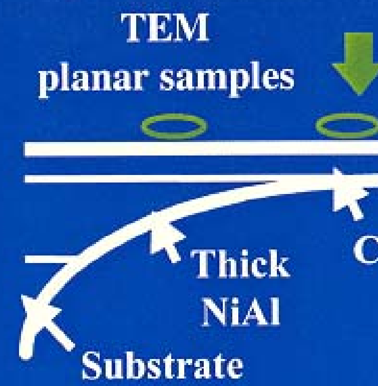
Electron Diffraction & Radial Average of Planar - TEM











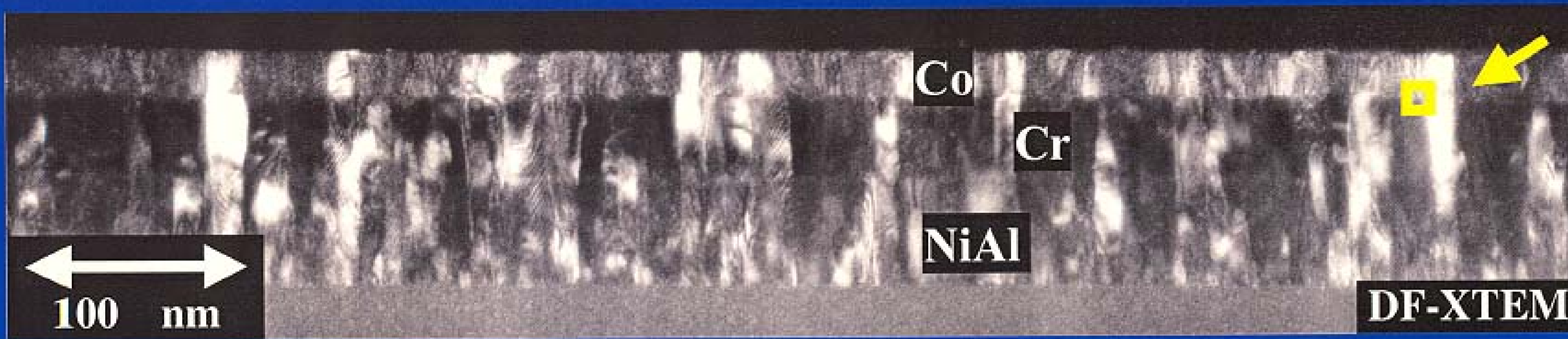
2nd seed layer
Structure change from
B2 to BCC Cubic

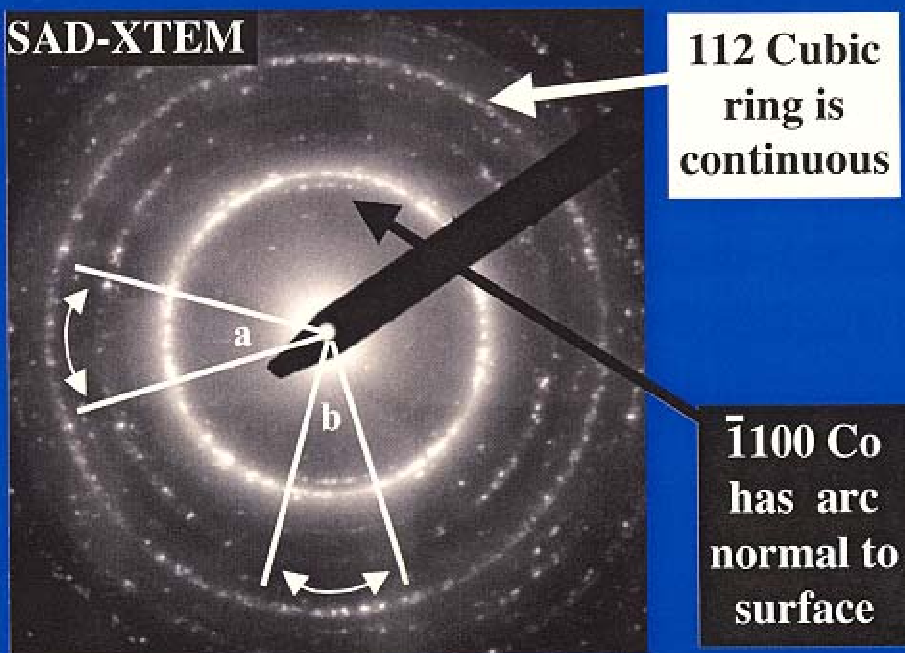
Still, grains with
{112} planes
Perpendicular to surface
can not have
<112> orientation

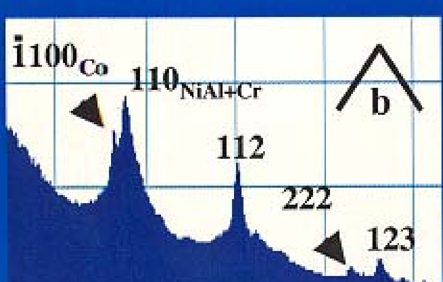
Grains continue
Growing with thickness

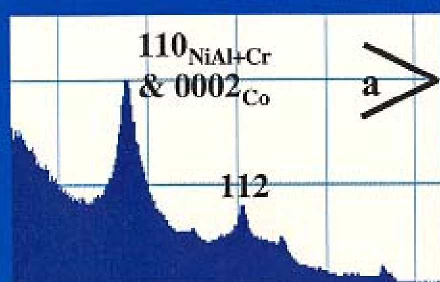
3-15-03wmc

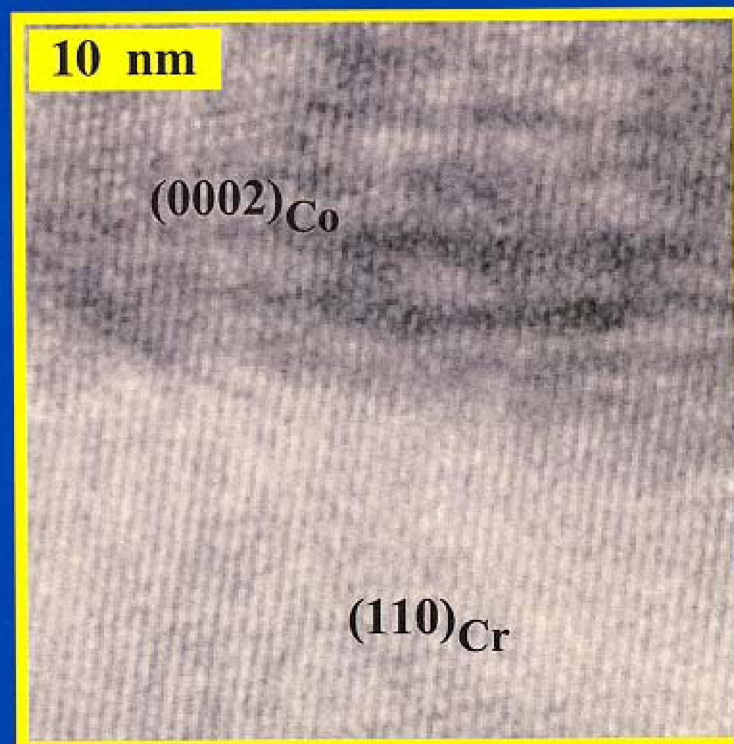
Cross - Section TEM of Media on NiAl

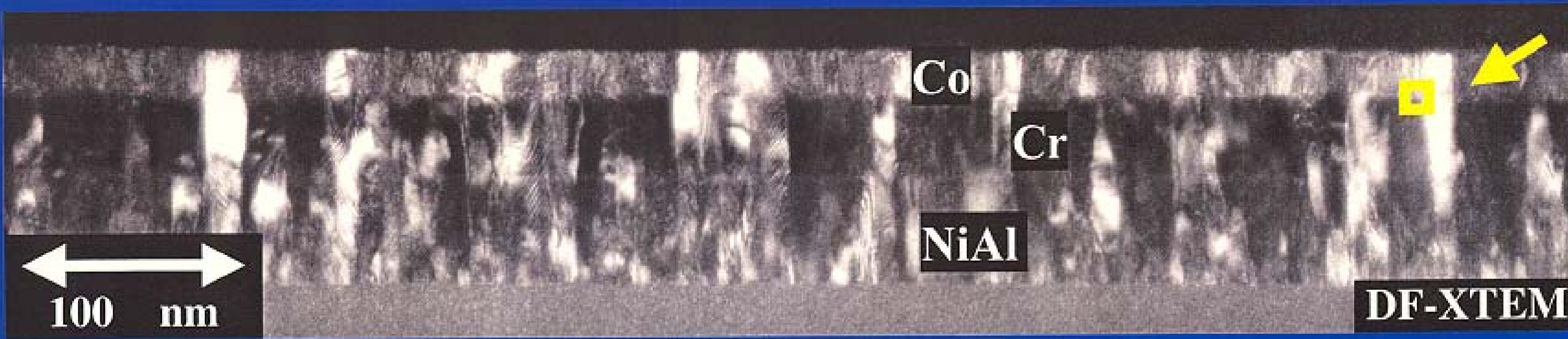






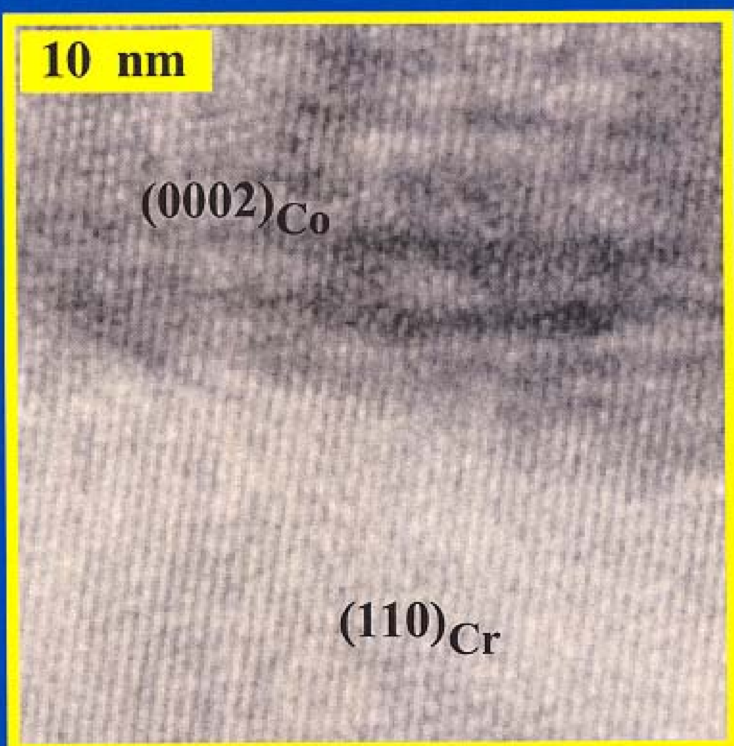


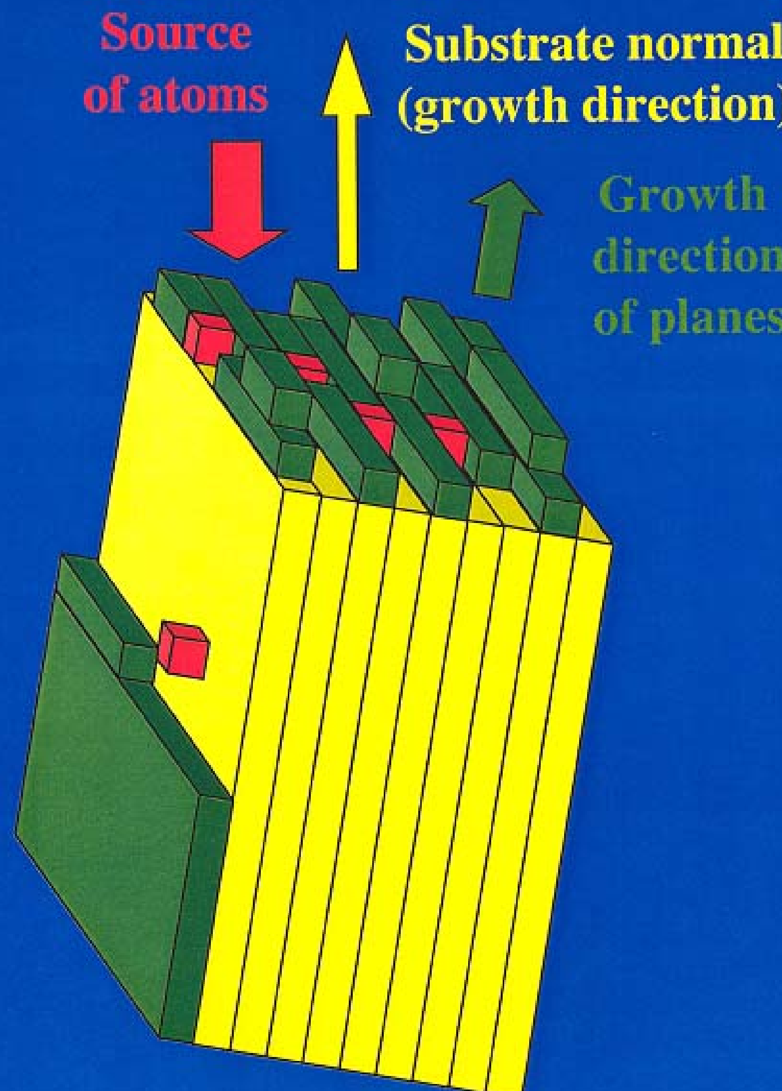




Cross - Section TEM of Media on NiAl

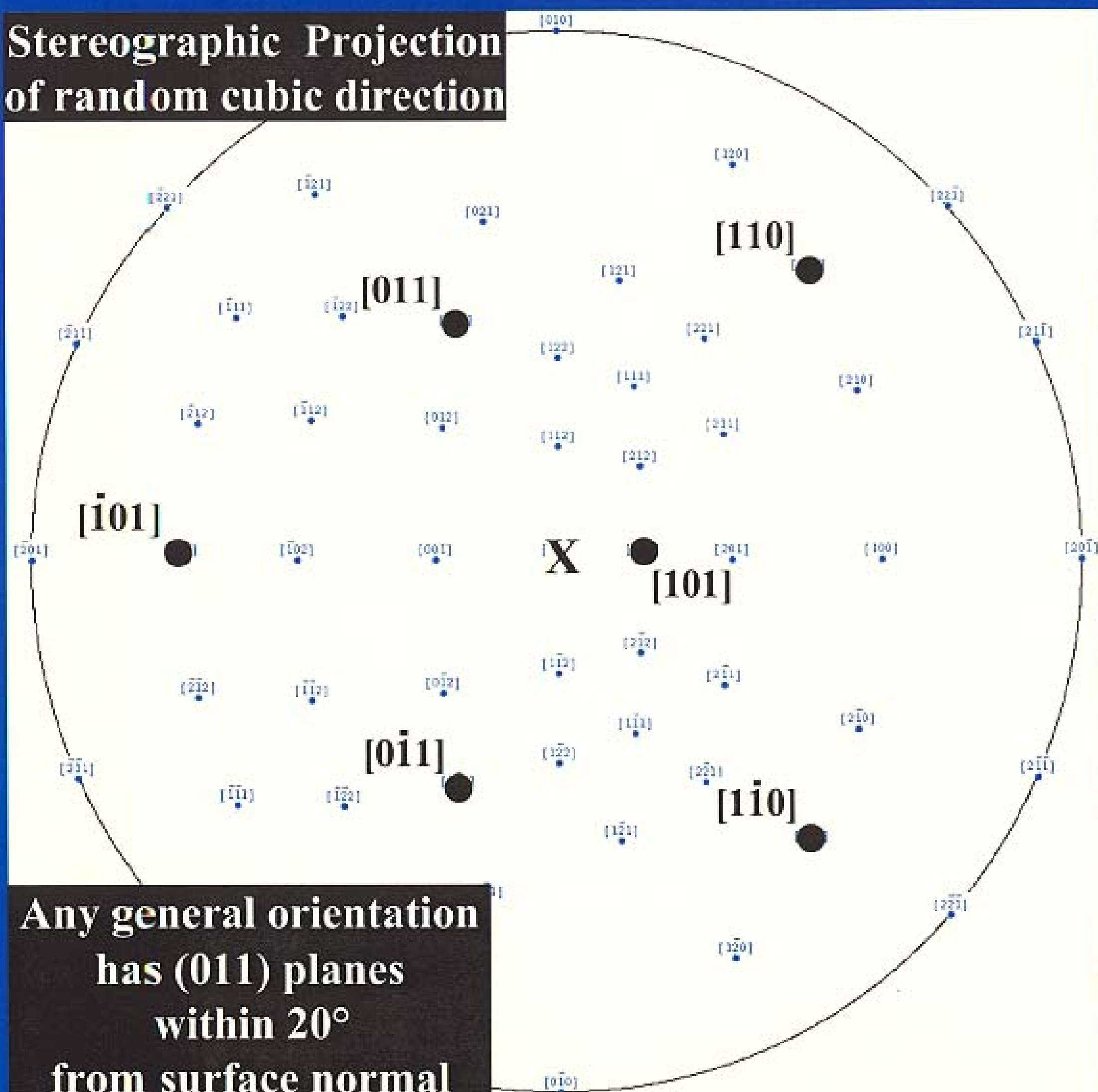
One set of closest-packed planes in seed layer(s)
Becomes close-packed (0002) planes in Co layer





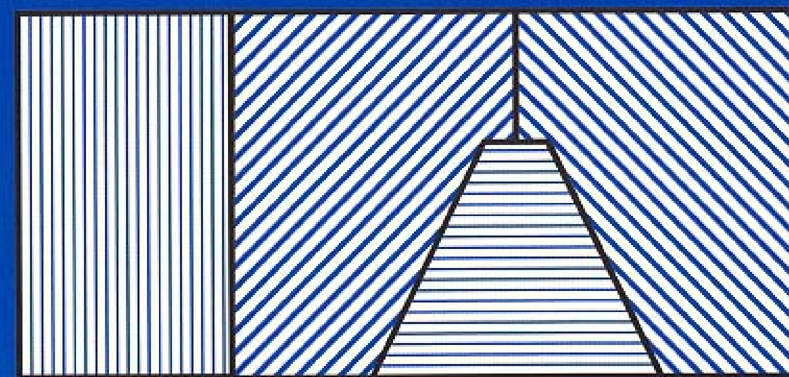
NiAl B2 Cubic Structure

Stereographic Projection of random cubic direction



Any general orientation has (011) planes within 20° from surface normal

Closest-Packed Planes (110)



1) Low energy orientations grow slowly (and die) <100> dies 1st, then <110>

2) Grains grow bigger as film thickens

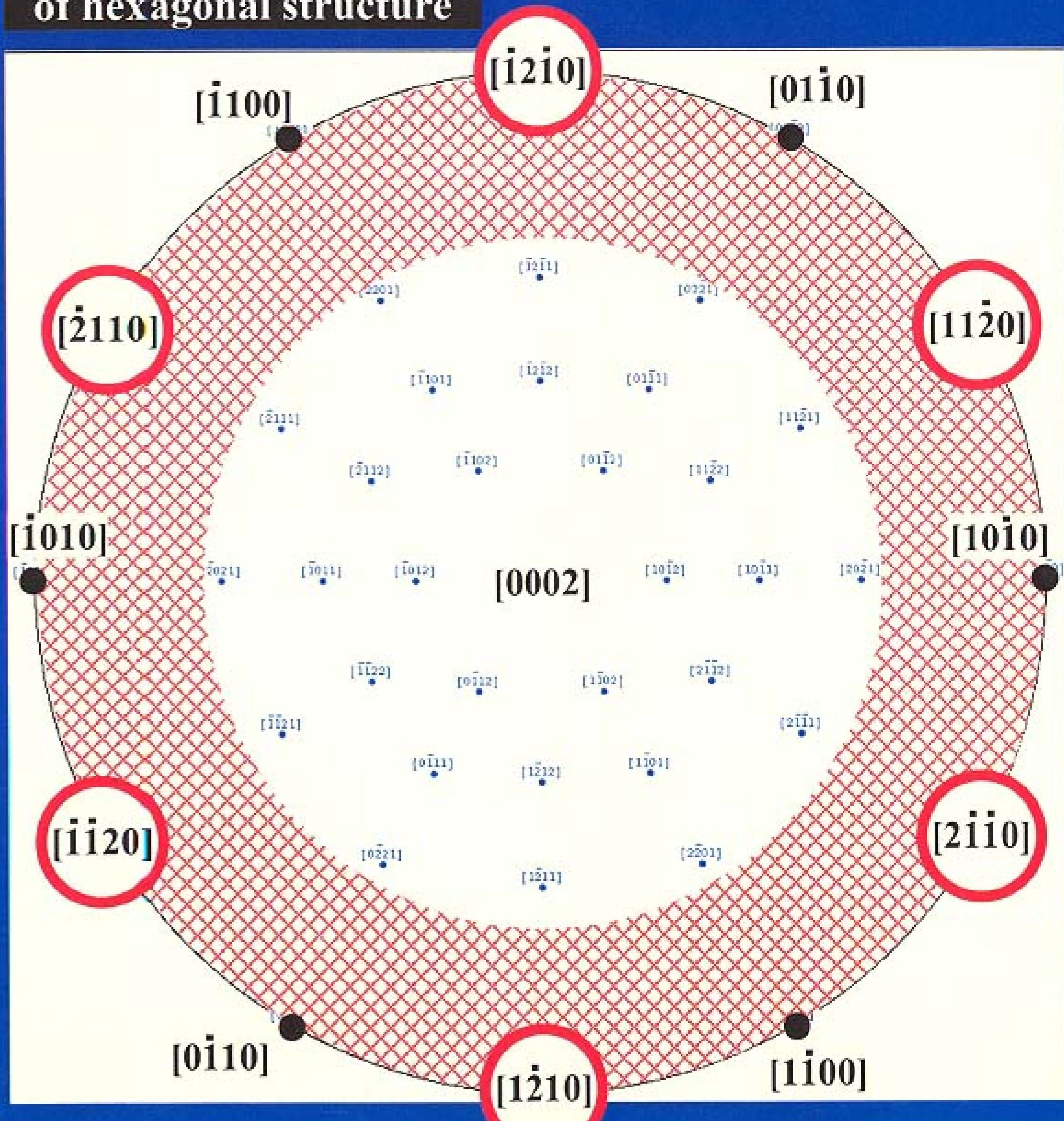
3) Most orientations remain; Film nearly 3D isotropic

3-15-03wmc

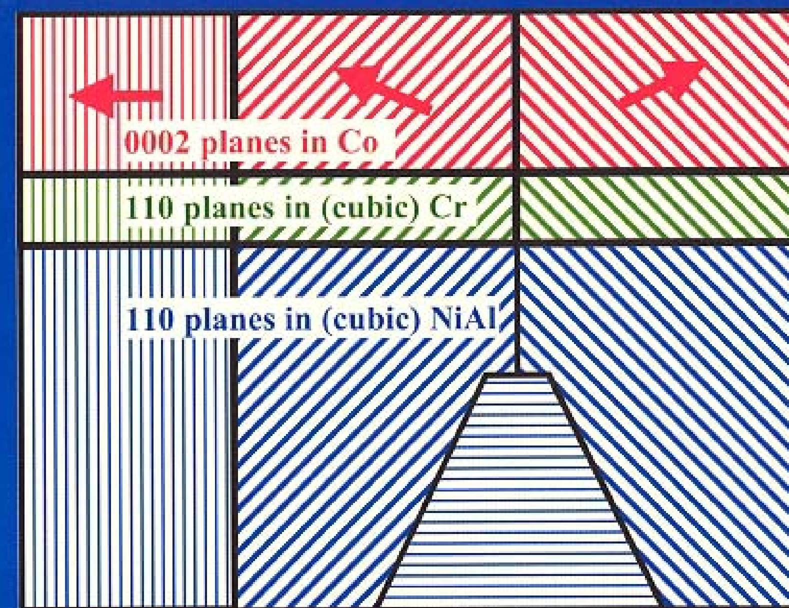
Cubic - to - Hexagonal Multiplicity

Reduces Orientations

Stereographic Projection of hexagonal structure



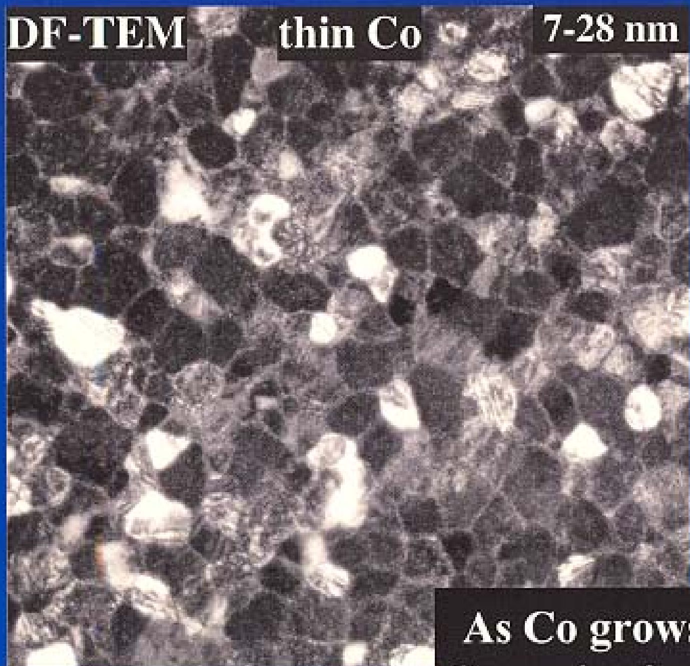
Closest-Packed Planes

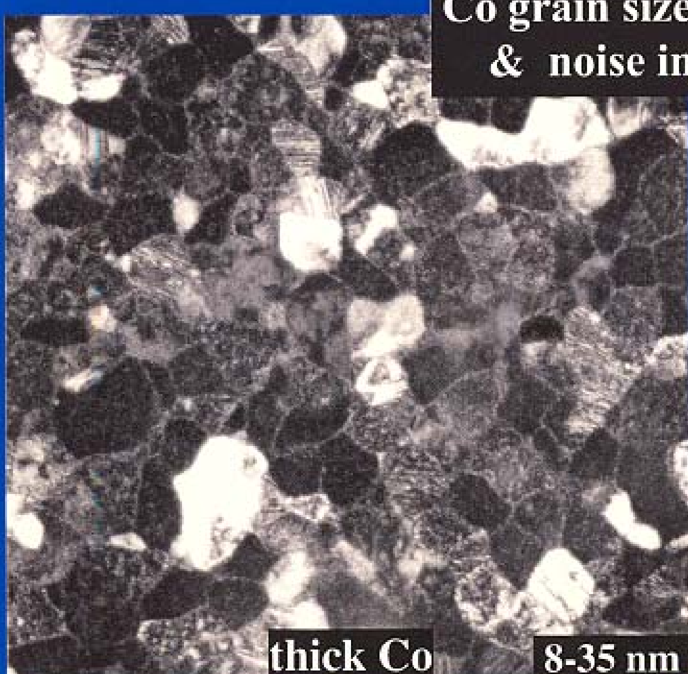


1) Cr (110) planes grow out of NiAl (110) planes that intersect surface
2) Co (0002) planes grow out of Cr (110)
3) Cubic-to-Hexagonal Multiplicity means all (0002) planes are +/-20° from in-plane
4) Magnetic dipoles +/- 20° from being in-plane (between 2D and 3D isotropic) limited squareness
5) Co thinner than its grain size pulls dipoles in-plane by shape anisotropy

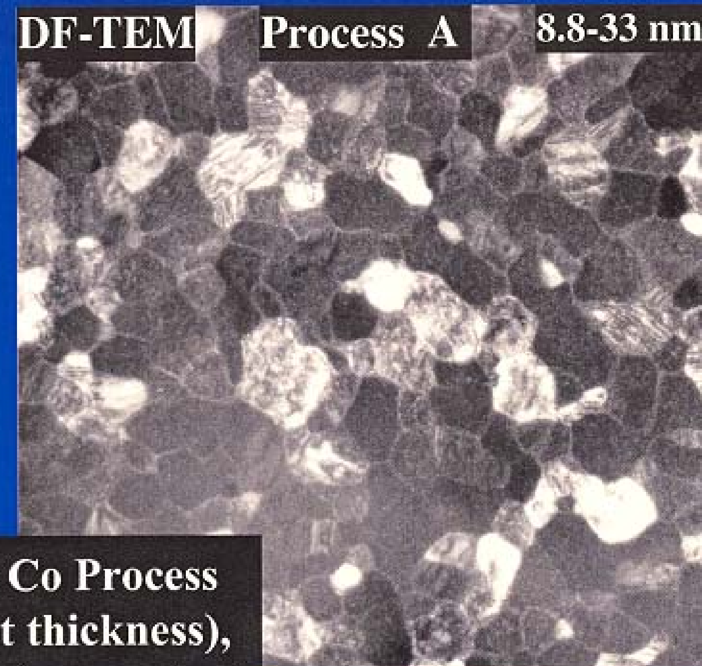
3-15-03wmc

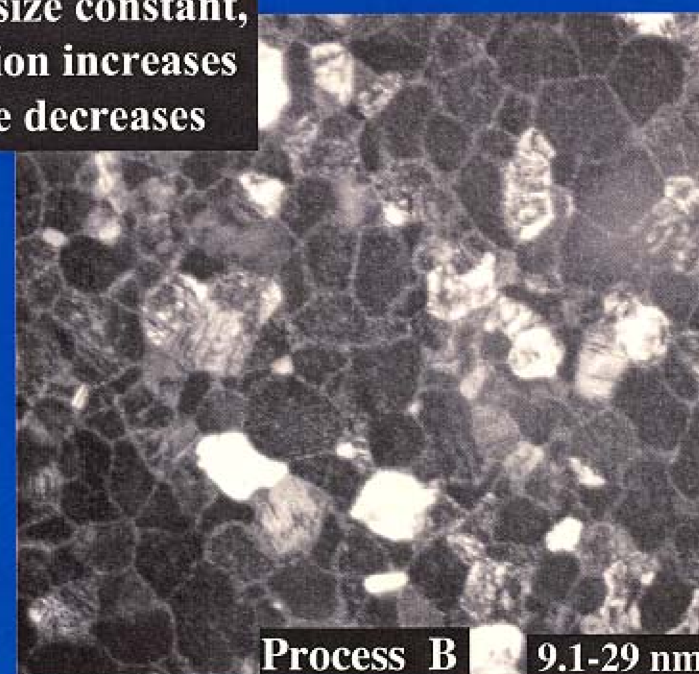
Co-X
on
constant Cr-X
on
constant NiAl





As Co grows thicker,
Co grain size increases
& noise increases





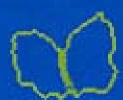
Change Co Process
(constant thickness),
Co grain size constant,
Segregation increases
& noise decreases

↔ 50 nm

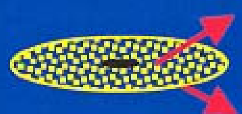
3-15-03wmc

summary

Develop Computer Analysis of Radial Averaging Electron Ring Diffraction
Used to Monitor Crystallographic Orientation Ratio
For 1-&-1/2-D Longitudinal Media



Weak Beam Dark Field Imaging Monitors Grain Size,
and (more importantly) Degree of Grain Separation



Cubic - to - Hexagonal Inverse Multiplicity Provides
2-&-1/4-D Isotropic Media on (nearly) Random NiAl Seed Layers;
Enabling Limited Squareness
(Epitaxy Controlled by Planes Extending Normal to Interface)

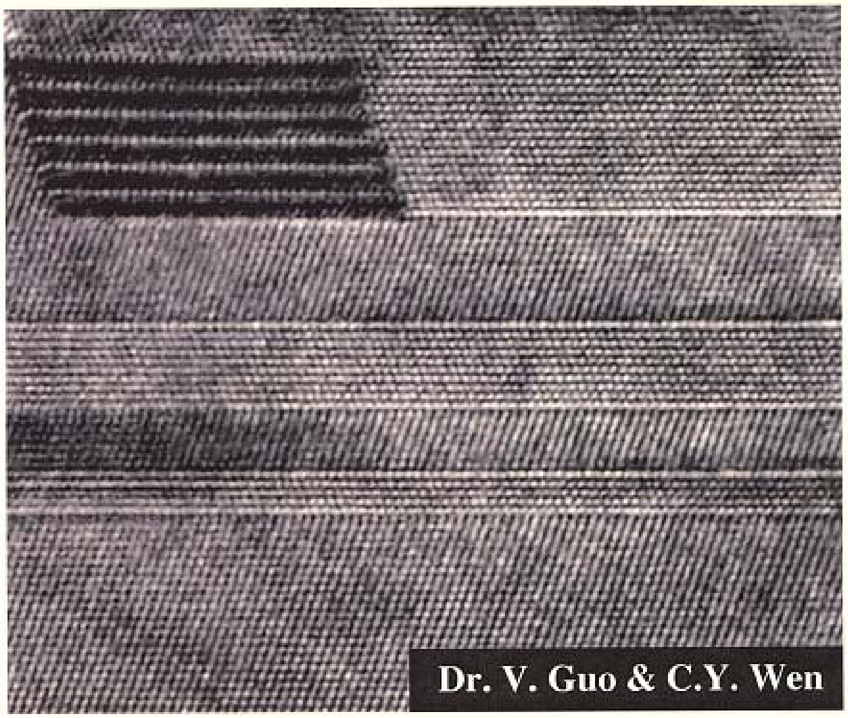
Cross-Sectioned Si Wire depicting twins

Common TEM view of fiber is longitudinally (lying down), which would be a view normal to this cross section:

A view from the top down is a <111> direction normal to the twin planes and would not see the defects

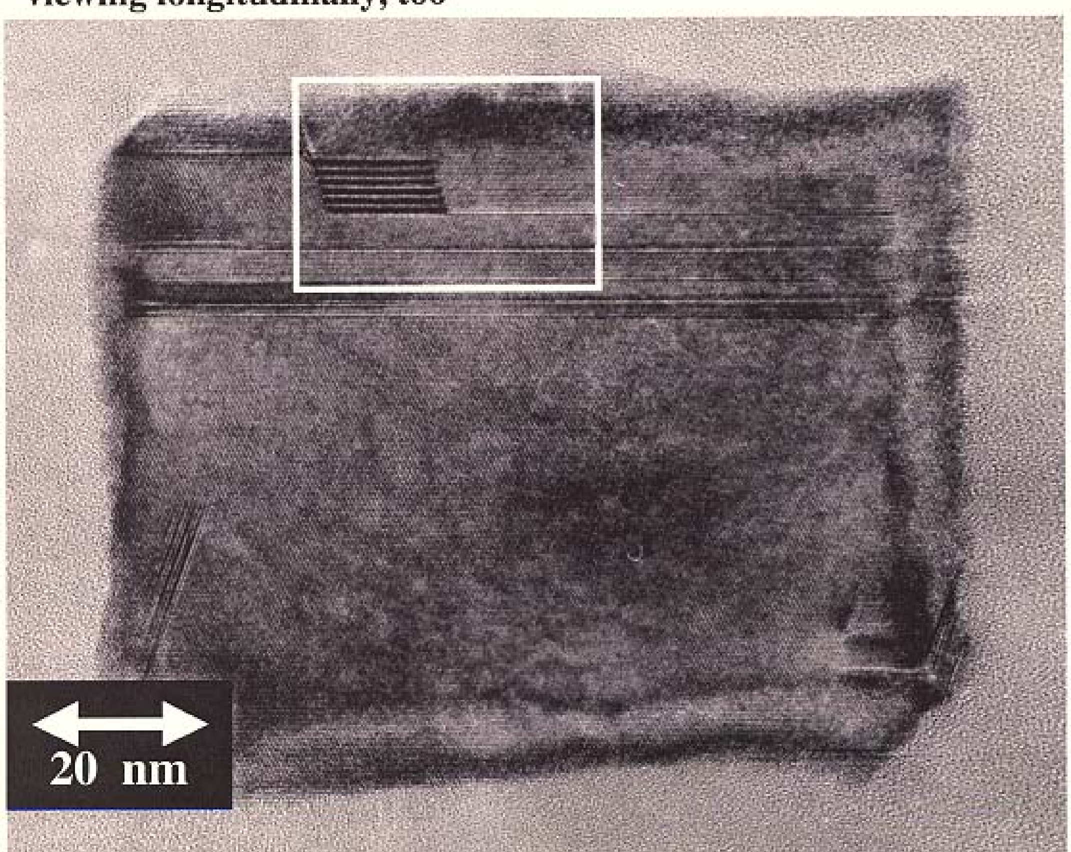
A view from side is a <112> which does not appear different for the two twin variants

A view from angles (e.g. 2 o'clock) would view the twin planes as faults in Bright/Dark Field imaging, and by double diffraction, but not directly imaged in HR-TEM.



Cross-Sectioned Si Wire

1) Wire is not round
2) Edges are not atomically flat this view is supported by viewing longitudinally, too



3-15-03wmc