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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Longitudinal Recording Media

I) In-Plane & OR (Crystallographic)
II) Grain Isolation & Spacing
III) Glass Media - Inverse Multiplicity

T. Nolan, JAP, 1993
Circumferential Texture Scratches

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

IBM Hard Disk Drive

Lubricant (C-F)
Protective Overcoat (C)
Media Layer (Co-alloy)
Seed Layer (Cr-alloy)

Gap Spacing ~1 μm

Microprocessor Speed (MHz)
Storage Density (MB/in²)

100000
10000
1000
100
10
1

1980 1990 2000

Ni(P)-Plating
Aluminum Substrate

Read/Rite Head
Thin Film Head
XRD of Co-X on Cr-X (Very well-oriented)

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
Out-of-Plane Diffraction of Well-Oriented [11\overline{2}0] Co-Alloy on [002] Cr
[1120] - Co // [002] - Cr
Hetero-epitactical Growth

\[ d_{110} \approx d_{0002} \approx 0.204 \text{nm} \]
\[ d_{110} < d_{1110} \approx 0.220 \text{nm} \]

**TLK** Terrace-Ledge-Kink
Growth Model

Substrate normal
(growth direction)

Source of atoms

Growth direction
of plane

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
[1120] - Co // [002] - Cr

Hetero-epitactical Growth

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.

Substrate normal (growth direction)

Source of atoms

Growth direction of planes

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

Substrate normal (growth direction)

Source of atoms

Growth direction of planes

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.
Well-Oriented [002] Cr Polycrystalline Thin Film

Radial Averaging

110  200

TEM-SADP ~5sec area: 1-10 bits


[002] Cr Thin Film
Grazing Incidence XRD
G. Khanna, Stanford
Synchrotron (1/2hr, $20M)
Well-Oriented [002] Cr Polycrystalline Thin Film
Randomly-Oriented Cr Polycrystalline Thin Film

2a

2b

2c

110

112

112

200

220

310

112Cr

d = 0.1178 nm

200Cr

d = 0.1443 nm

d-spacing
Thin-Film Media
Epitaxial Co
(with Cr, Pt. Ta)
Oriented [1\(\bar{2}0\)] Co-Alloy with 0\% Pt
Oriented [11\(\overline{2}0\)] Co-Alloy with 8\% Pt
[1120] -Co (grown on [002]-Cr)

TEM-planar sample
Selected Area Diffraction

Cr
Co
Ni(P)

"Goodness" of 1120 indicated by absence of 1120

360° full radial average

0002 Co

90° arc parallel to texture

0002 Co

90° arc perpendicular to texture

Selected Angles of Radial Average of Diffraction Intensities

İ101
İ102
İ103

İ101 ring is isotropic so use for internal reference
Co-alloy with Orientation Ratio, Hc is 50% higher along texture scratches

360° full radial average

90° arc 30° arc 10° arc

texture direction

normal to texture along circumferential texture

0.205nm 0002 Co
0001 1100
1101
0.1145nm 1103
1102 2201
Dark Field Imaging

DF-TEM

2a

DF-TEM

2b

100 nm

WBDF-TEM

3a

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)
Chemical Imaging
(due to inelastic scattering)

Structural Imaging
(Dark Field due to diffraction)

J. Wittig, J. Bentley
$\text{Co}_{80}\text{Cr}_{16}\text{Ta}_4$

EF-TEM Co Jump Ratio

EF-TEM Cr Jump Ratio

WBDF-TEM

$100 \text{ nm}$

$g = 0002$

$10-30 \text{ nm}$
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)
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)
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
Cross-sectioned magnetic recording media

EF-TEM reveals intergranular segregation between columnar grains

zero loss

Co jump ratio

Cr jump ratio

CoCrTa  Cr  Ni-P

60 nm

Jim Wittig, Vanderbilt
Jim Bentley, ORNL
Dark Field Imaging & Cross Correlation to Measure Grain Size

BF-TEM

DF-TEM

WBDF-TEM

0.4

central peak truncated

central peak width

α segregation width

fine peak spacing

α grain size

large peak spacing

α texture spaces

Cross Correlation of WBDF-TEM

8 - 25 nm
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 - X Alloy

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

2A: Co - X₁
Process "A"
Hc=2200

2B: Co - X₁
Process "B"
Hc=3100

5-9 nm segregant spacing
60 nm

However, some bicrystals can be connected around segregant

3: HR-TEM

10 nm

Co - X₁ has constant X%
Change process from "A" to "B" causes
larger spacing of segregant,
but more discrete;
Thus "B" has lower media noise
(and higher coercivity),
even with larger grains
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

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

Read/Rite Head

Gap Spacing ~1 μm

Lubricant (C-F)
Protective Overcoat (C)
Media Layer (Co-alloy)
Seed 2 (Cr-alloy)

Seed - NiAl

Alternative Substrates
(Glass / Plastic / Metal)
NiAl on Glass
15 hrs XRD

many weak peaks
no strong orientation

Intensities increase with
film thickness

as NiAl thickens, orientation changes

Add layers to see where orientation came from
Intensities proportional to film thickness

All XRD peaks are weak
no strong orientation

Add layers to see where orientation is going
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

All rings present in thin films; - 3D random

200 ring disappears @ top of thick films; singular orientations lost as grains grow
Planar - TEM of NiAl films; Grain size increases as film thickens

All rings present in thin films; - 3D random

200 ring disappears @ top of thick films; singular orientations lost as grains grow
Energy - Filtered Cr Image
With computer-generated grain boundary profiles

Jim Wittig, Vanderbilt
Jim Bentley, ORNL
Electron Diffraction & Radial Average of Planar - TEM

3b
Cubic B2 structure
top of thick NiAl

4b
Cubic BCC structure
Cr-X on top of thick NiAl

3c
011 112 111 200 022 123

4c
011 112 022 013 123

TEM planar samples
Cr
Thick NiAl Substrate

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
Cross-Section TEM of Media on NiAl

112 Cubic ring is continuous

1100 Co has arc normal to surface

(0002)Co

(110)Cr
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

Source of atoms

Substrate normal (growth direction)

Growth direction of planes

10 nm

(0002)_{Co}

(110)_{Cr}
NiAl B2 Cubic Structure

Stereographic Projection of random cubic direction

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

Any general orientation has (011) planes within 20° from surface normal
Cubic - to - Hexagonal Multiplicity
Reduces Orientations

Stereographic Projection of hexagonal structure

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
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
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)
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