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Understanding Texture Evolution in Nanocrystalline Nickel Films

Shreyas Rajasekhara, Kameswaran J. Ganesh, Paulo J. Ferreira
Materials Science & Engineering Program
The University of Texas at Austin, Austin, Texas - 78712

Khalid Hattar, James A. Knapp
Physical, Chemical and Nano sciences Center
Sandia National Laboratories, Albuquerque, New Mexico – 87185

Sandia National Laboratories

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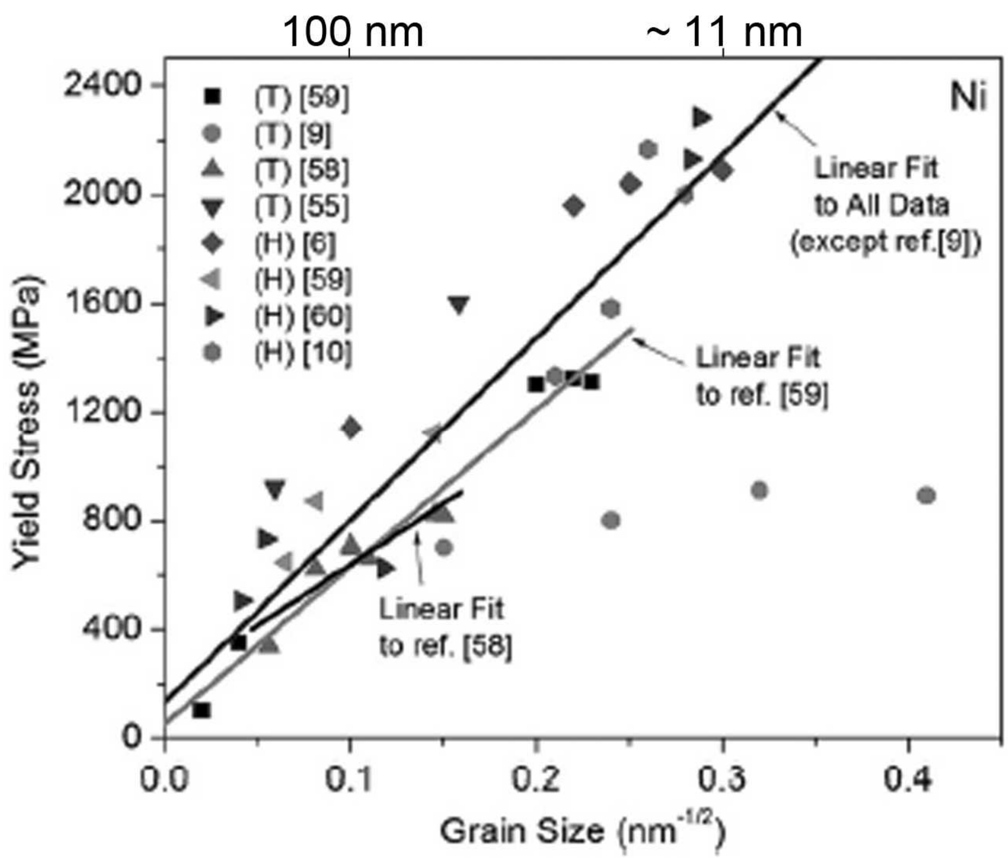
WHAT STARTS HERE CHANGES THE WORLD
Cockrell School of Engineering

The interest in nanograined metals

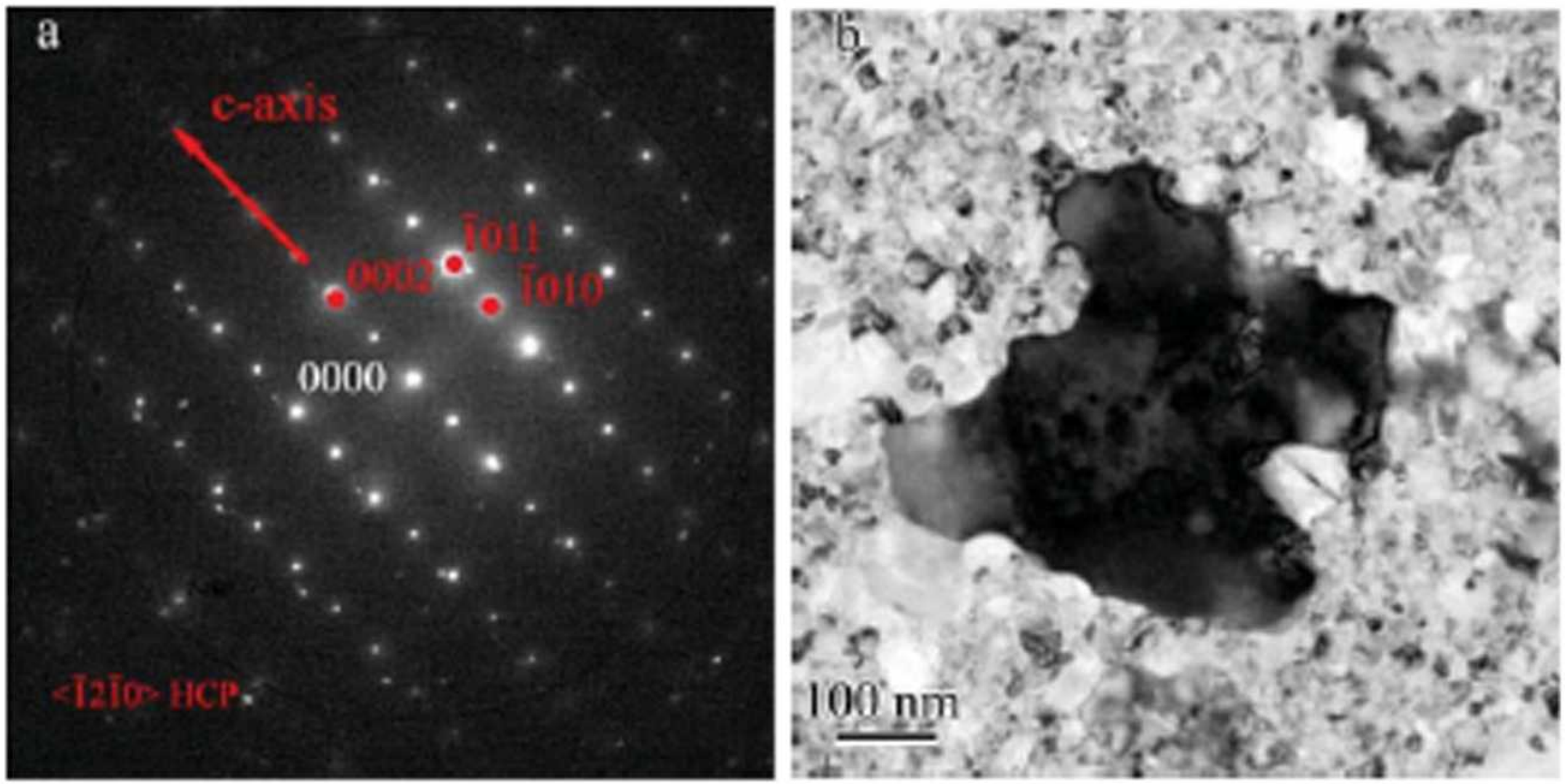


- Nanocrystalline metals are attractive because of their high strength.
- Basis for high strength is captured by the Hall – Petch relationship: $\sigma = \sigma_o + kd^{-1/2}$

C. E. Carlton, P. J. Ferreira, Acta Materialia **55** (2007), 3749

S. Rajasekhara, L. P. Karjalainen, A. Kyrolainen, P. J. Ferreira, Met. Mater. Trans. **37A** (2007), 1202

Thermal stability of nanoscale grains

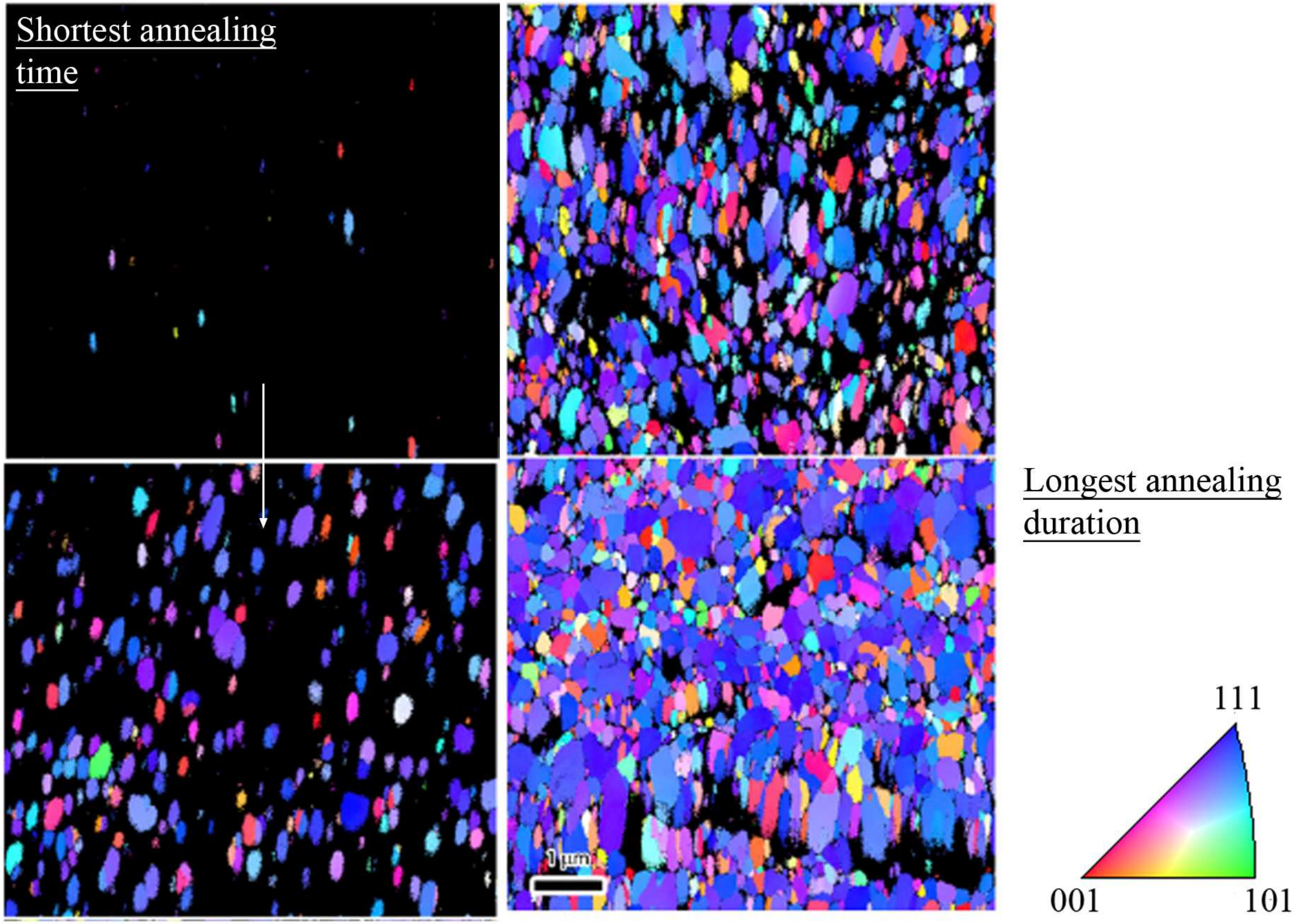


Select area diffraction of an abnormal grain

K. Hattar, 'Thermal and mechanical stability of *fcc* nanograined metals', Ph. D. dissertation (2009), The University of Illinois, Urbana-Champaign

L. N. Brewer, D. M. Follstaedt, K. Hattar, J. A. Knapp, M. A. Rodriguez, I. M. Robertson, Adv. Mater. **22** (2010), 1161

Thermal stability of nanoscale grains



J. Kacher, I. M. Robertson, M. Howell, J. A. Knapp, K. Hattar, Mater. Sci. Engg. A **528** (2011), 1628

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Motivation for this work

i. What is the local texture of as-deposited nanocrystalline films?

ii. How does it change with thickness?

iii. Is the *hcp* phase nickel present in as-deposited nano-crystalline nickel films?

Available electron diffraction techniques

Technique	Advantages	Disadvantages
EBSD (SEM)	- Statistically significant - Accurate	- Difficult to accurately index nanoscale grains (< 30 nm) - Sample dependent
Kikuchi maps (TEM/STEM)	- Analysis of individual nanoscale particles	- Time consuming - Pole-piece gap constraint - Difficult to automate
Nanobeam diffraction (NBD)	- Faster than Kikuchi maps - Analysis of nanoscale grains	- Slower than EBSD - Difficult to automate
Convergent beam diffraction (CBED)	- Spatial resolution - Symmetry information	- Local heating - Complex patterns

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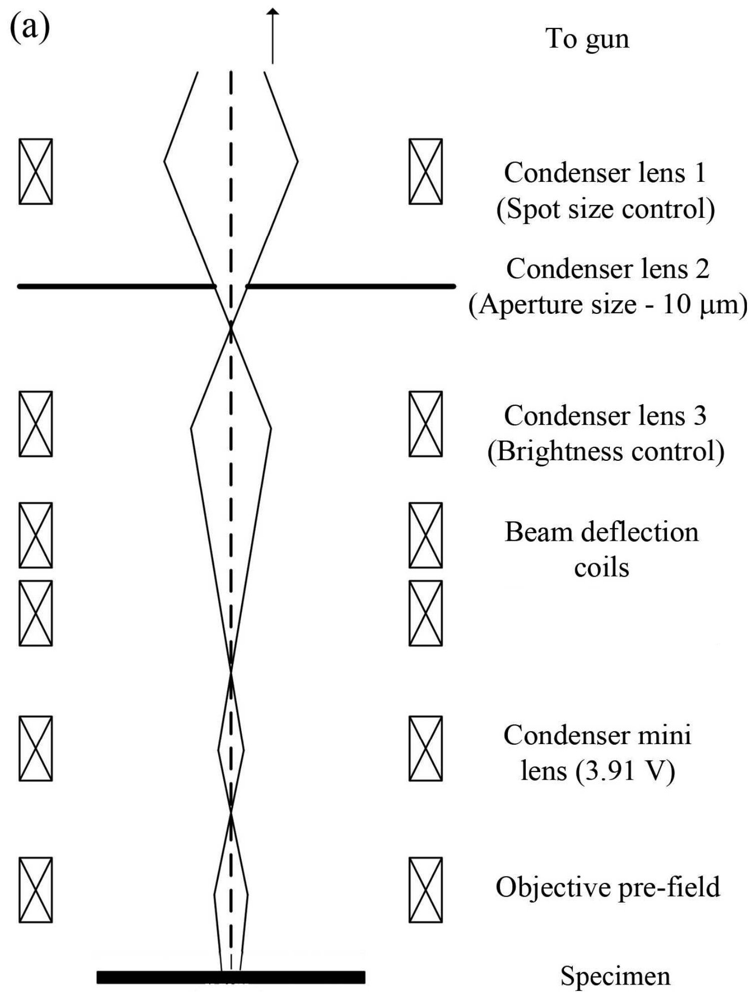
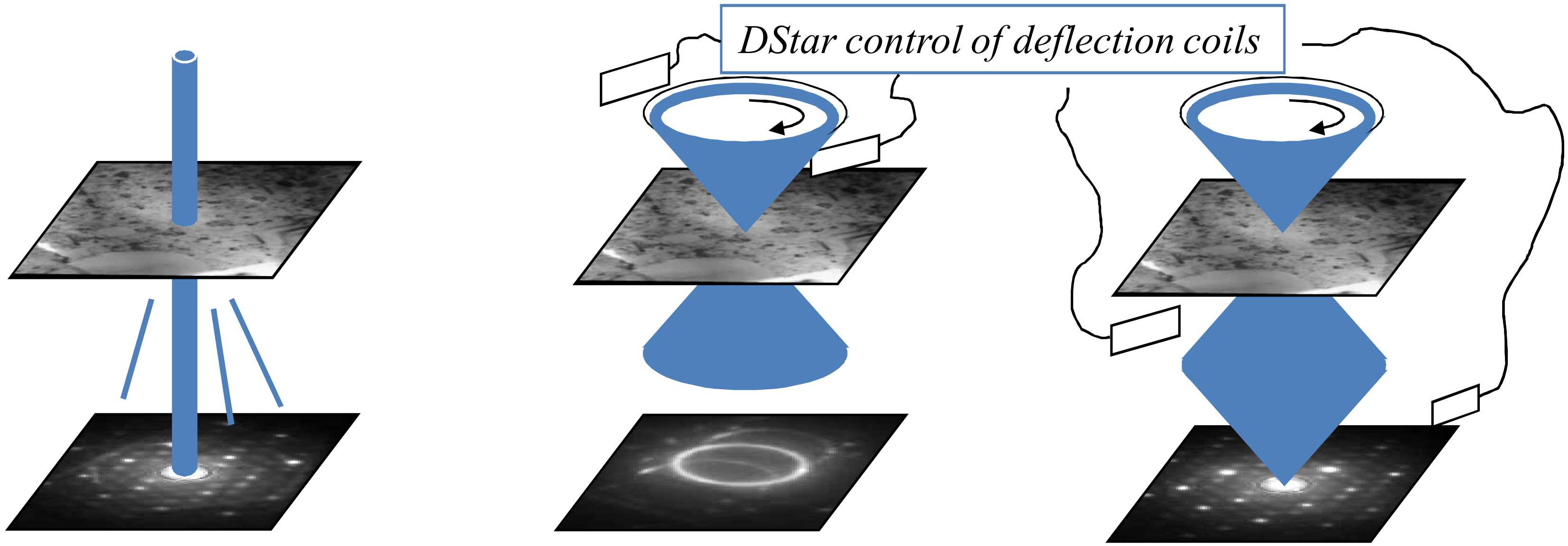
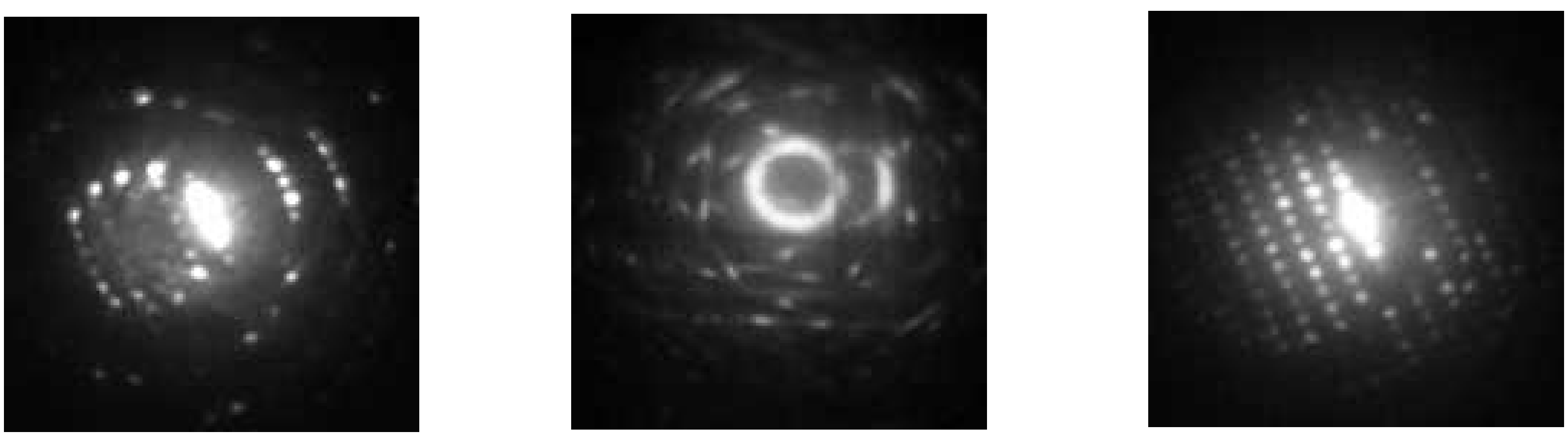
Creating a near parallel nanoscale probe



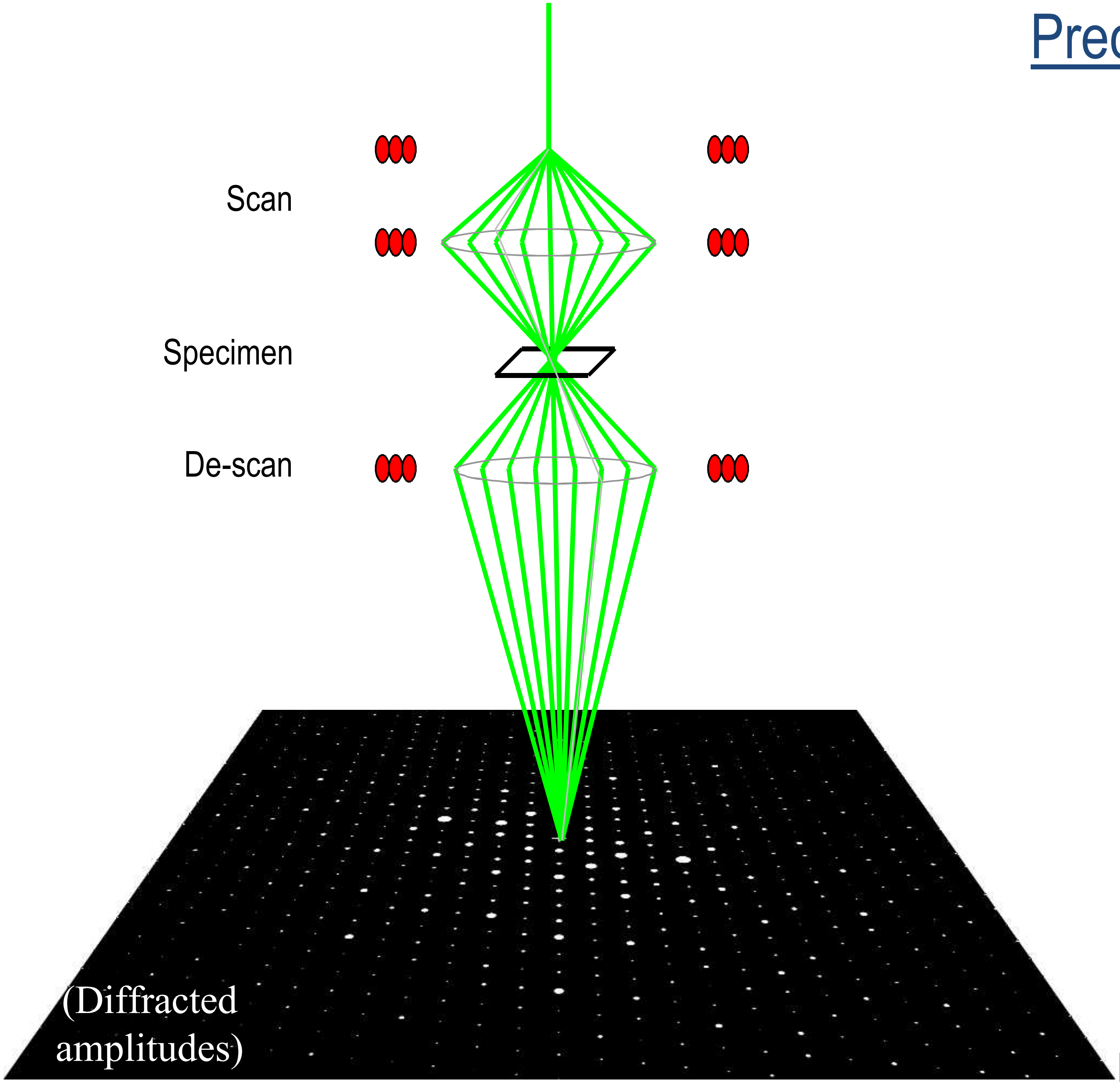
Precession microscopy

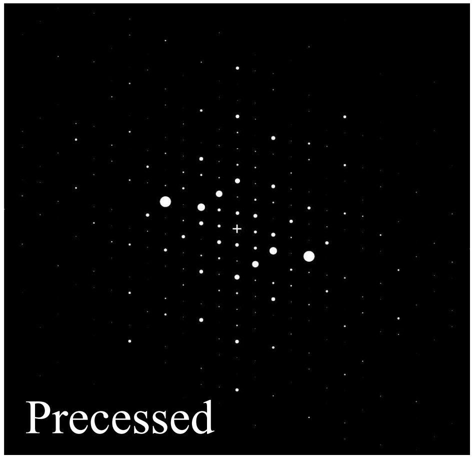


Standard setting | ½ Precession | Full precession



Precession microscopy





Chris Own, PhD Dissertation, 2004

Slide courtesy ; K. J. Ganesh

Advantages:

i. < 10 nm spatial resolution

ii. Near kinematical electron diffraction

iii. Symmetry ambiguities are resolved

iv. Fast and automated acquisition (at least 200 grains in 15 minutes)

v. Template generation and matching

Apply precession microscopy to characterize nanocrystalline nickel!

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Experimental

Sample preparation:

- Pulsed Laser Deposition of 50 and 100 nm nickel films on NaCl <001> substrates
- Athene® thin bar grid glued to the film
- NaCl <001> substrate was dissolved away in de-ionized water

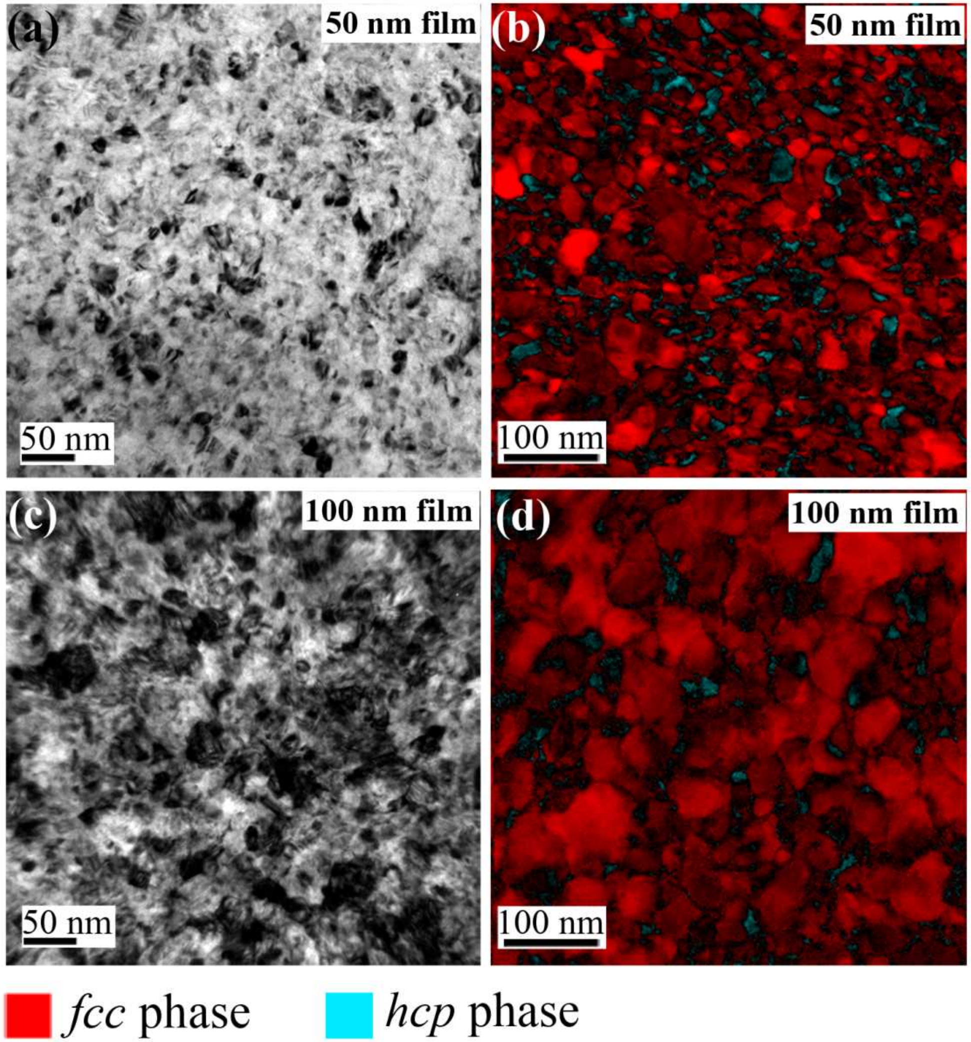
Microstructure evaluation:

- Diffraction data acquisition by precession microscopy
- Phase and texture analysis

hcp grain size and phase fraction

BF – TEM

Re-constructed phase and reliability map



- 1124 *hcp* phase grains (in 1.5 μm²)
- Average *hcp* grain size : 8.1 ± 0.3 nm
- Average *hcp* phase percentage: 6.0%

- Average *fcc* phase grain size: 22.0 ± 1.2 nm (395 grains)

- 952 *hcp* phase grains (in 1.5 μm²)
- Average *hcp* grain size : 9.5 ± 0.4 nm
- Average *hcp* phase percentage: 6.0%

- Average *fcc* phase grain size: 39.1 ± 1.6 nm (375 grains)

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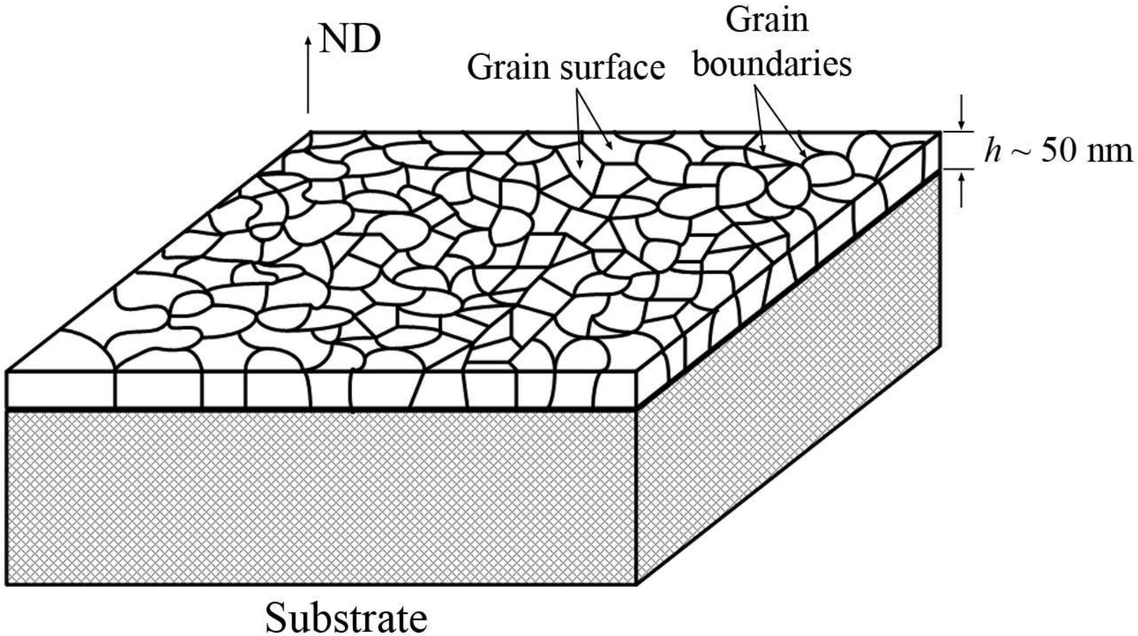
What is the reason for *fcc*-nickel grain size increase a function of film thickness?

Equilibrium grain size: 50 nm film



Equilibrium grain size (d): Balance between total grain boundary energy and surface energy

$$d = h\left(\frac{\langle \gamma_{gb} \rangle}{\gamma_s}\right)\frac{4}{\sqrt{3}}$$

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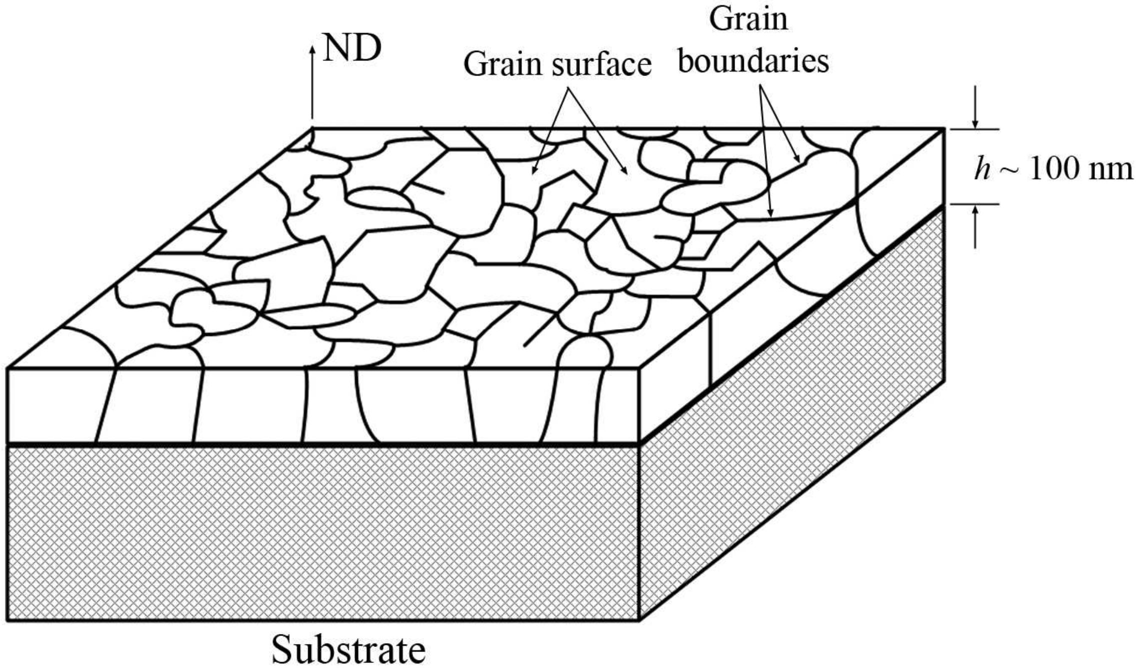
Equilibrium grain size: 100 nm film



Equilibrium grain size (*d*): Balance between total grain boundary energy and surface energy

$$d = h\left(\frac{\langle \gamma_{gb} \rangle}{\gamma_s}\right)\frac{4}{\sqrt{3}}$$

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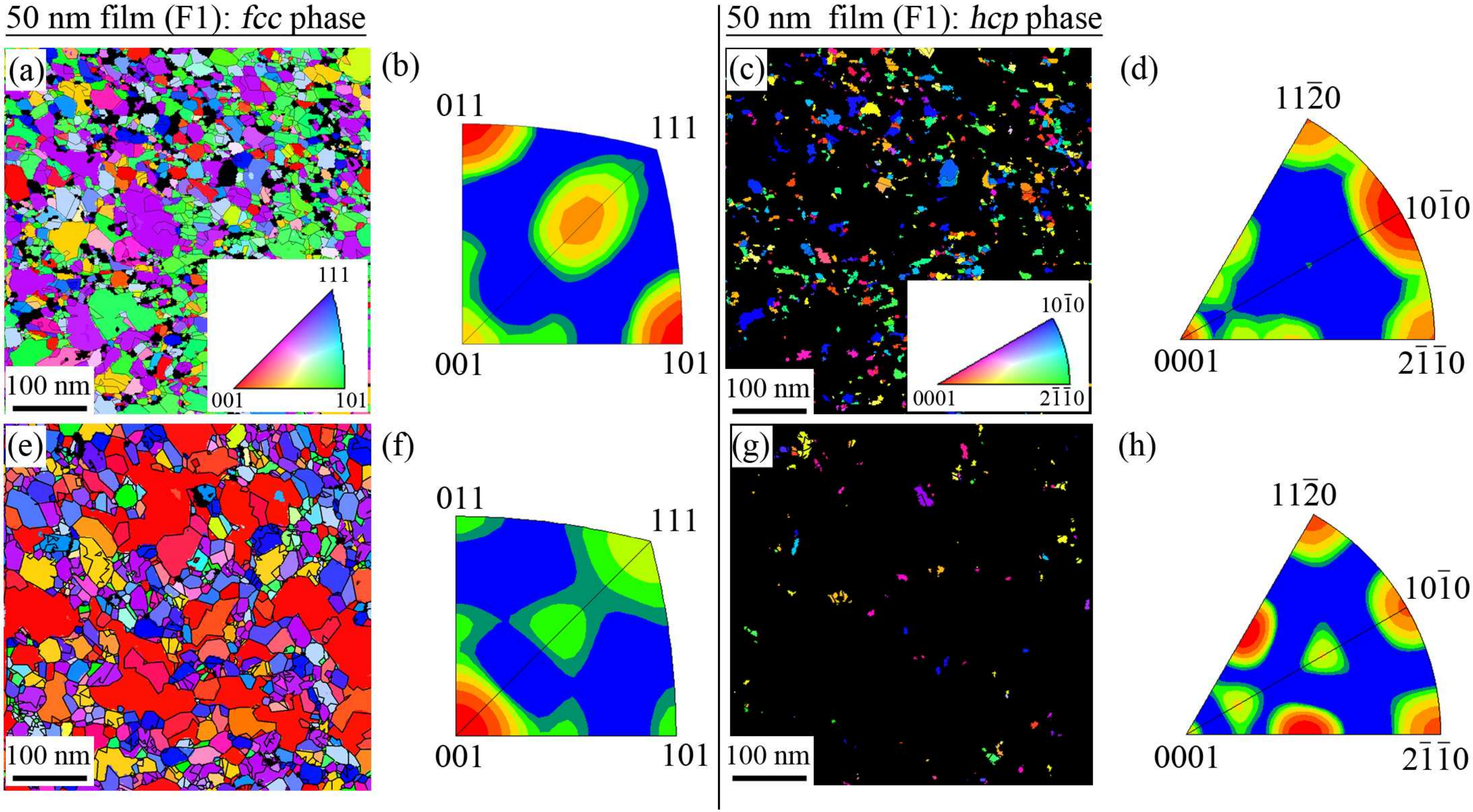
Texture of 50 PLD – Ni films



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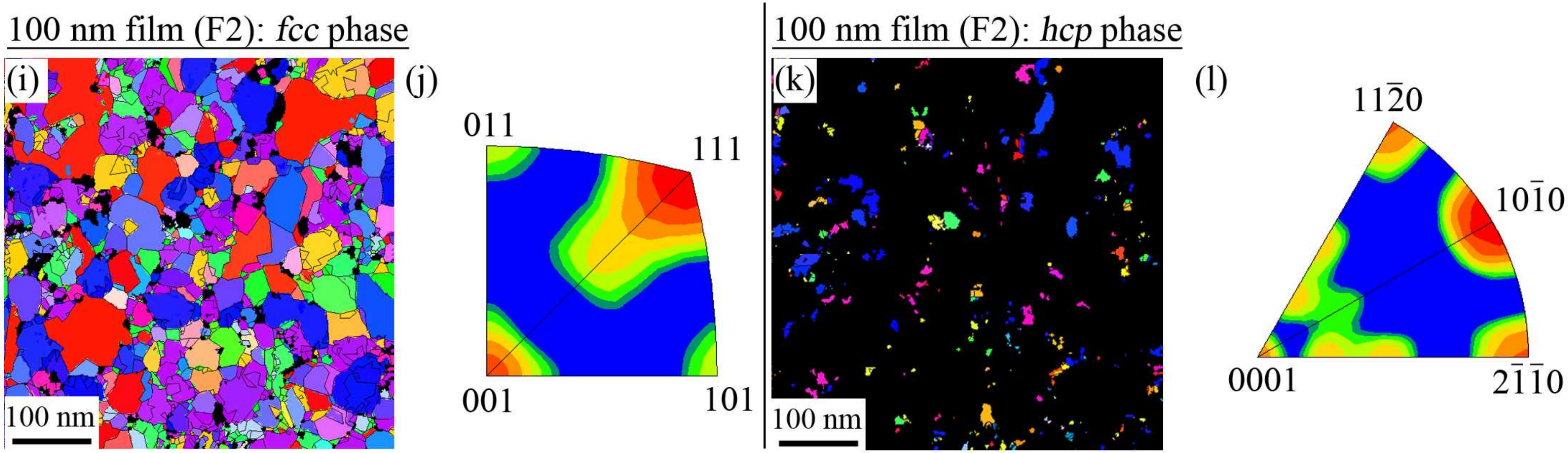
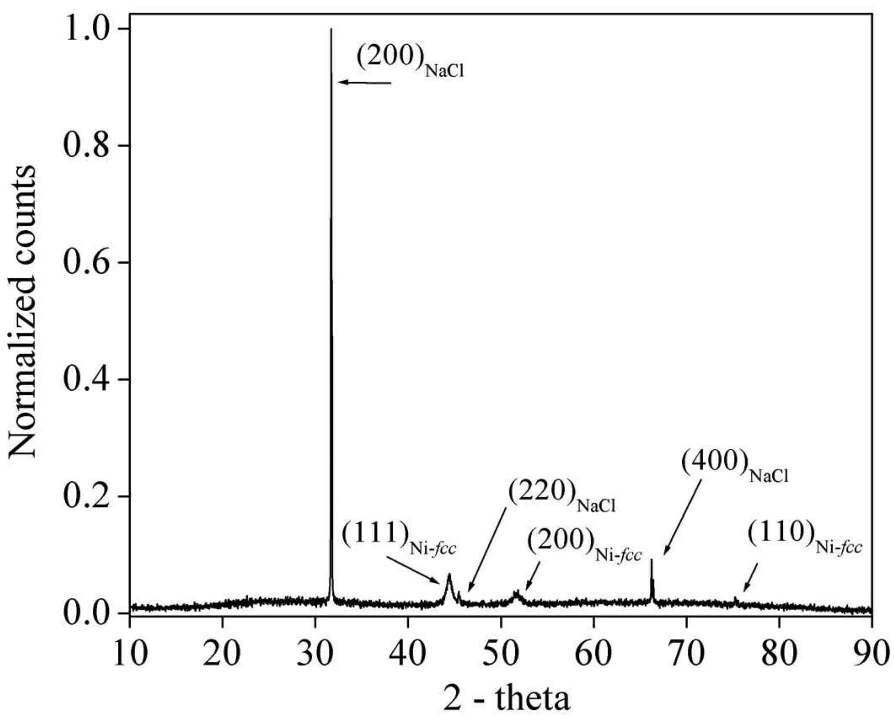
Texture of 100 PLD – Ni films

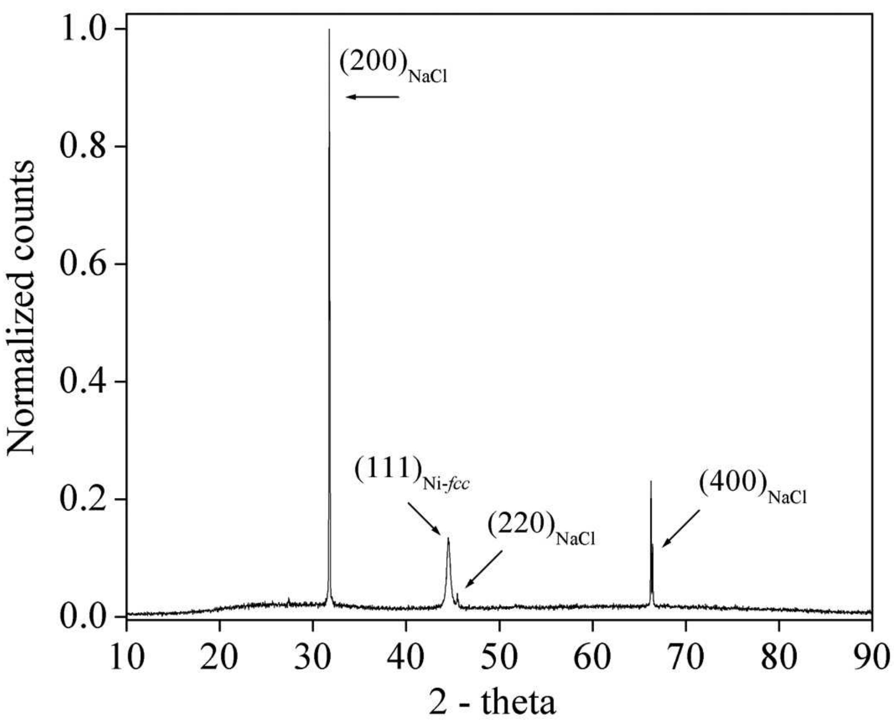


X-Ray Diffraction of 50 and 100 nm PLD – Ni films

50 nm thick film



100 nm thick film



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What is the reason for texture change a function of film thickness?

<001> // ND and <011> // ND fiber texture in 50 nm film:

- Influence of <001> // ND NaCl substrate
- Substantial strains in the film due to lattice mismatch (a_{NaCl} ~ 5.66 Å, $a_{Ni\text{-}fcc}$ ~ 3.52 Å)
- Strain energy density minimization results in the formation of <011> // ND
- Catenoidal grain shapes result in grain boundary grooving

<111> // ND fiber texture in 100 nm film:

- Substrate and grain boundary grooving effects diminish
- Achieve equilibrium texture

C. V. Thompson, J. Floro, J. Appl. Phys. **67** (1990), 4099
J. E. Sanchez Jr., E. Arzt, Scripta Metall. et. Mater. **27** (1992), 285
C. V. Thompson, R. Carel, Mater. Sci. Engg. B **32** (1995) 211
W. W. Mullins, Acta Metall. **6** (1958) 417

Conclusions

i. Nanoscale *hcp* nickel phase in as-deposited 50 and 100 nm films

ii. Approximately 10 nm in size, and with close to $<10\bar{1}0>$ // ND texture

iii. Nanoscale *fcc* nickel phase is approximately 21 nm size, <001> and <011> // ND texture

iv. *fcc* nickel phase texture evolves from <001> and <011> // ND to <111> // ND and 37 nm size with increasing film thickness

v. Surface and grain boundary energy minimization plays an important role in grain size increase and texture change.