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Understanding the Structure of Nanograined Polycrystalline Materials by *in-situ* and Precession Electron Microscopy

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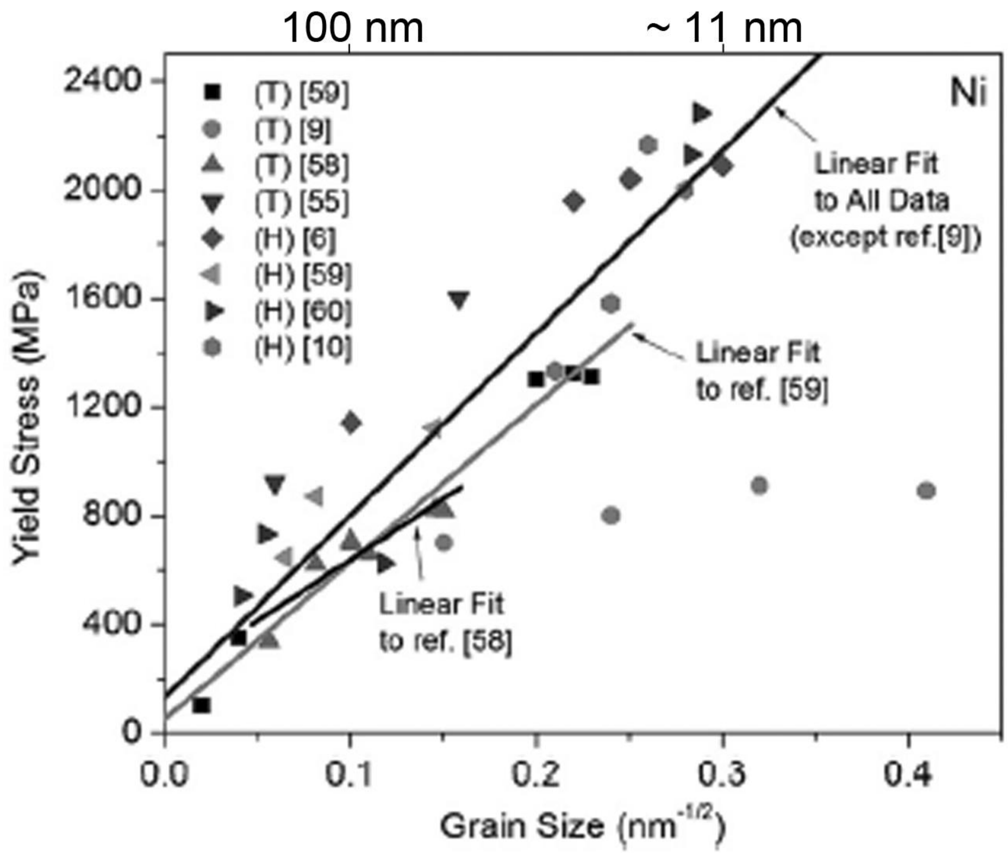
WHAT STARTS HERE CHANGES THE WORLD

Cockrell School of Engineering

The interest in nanograined metals

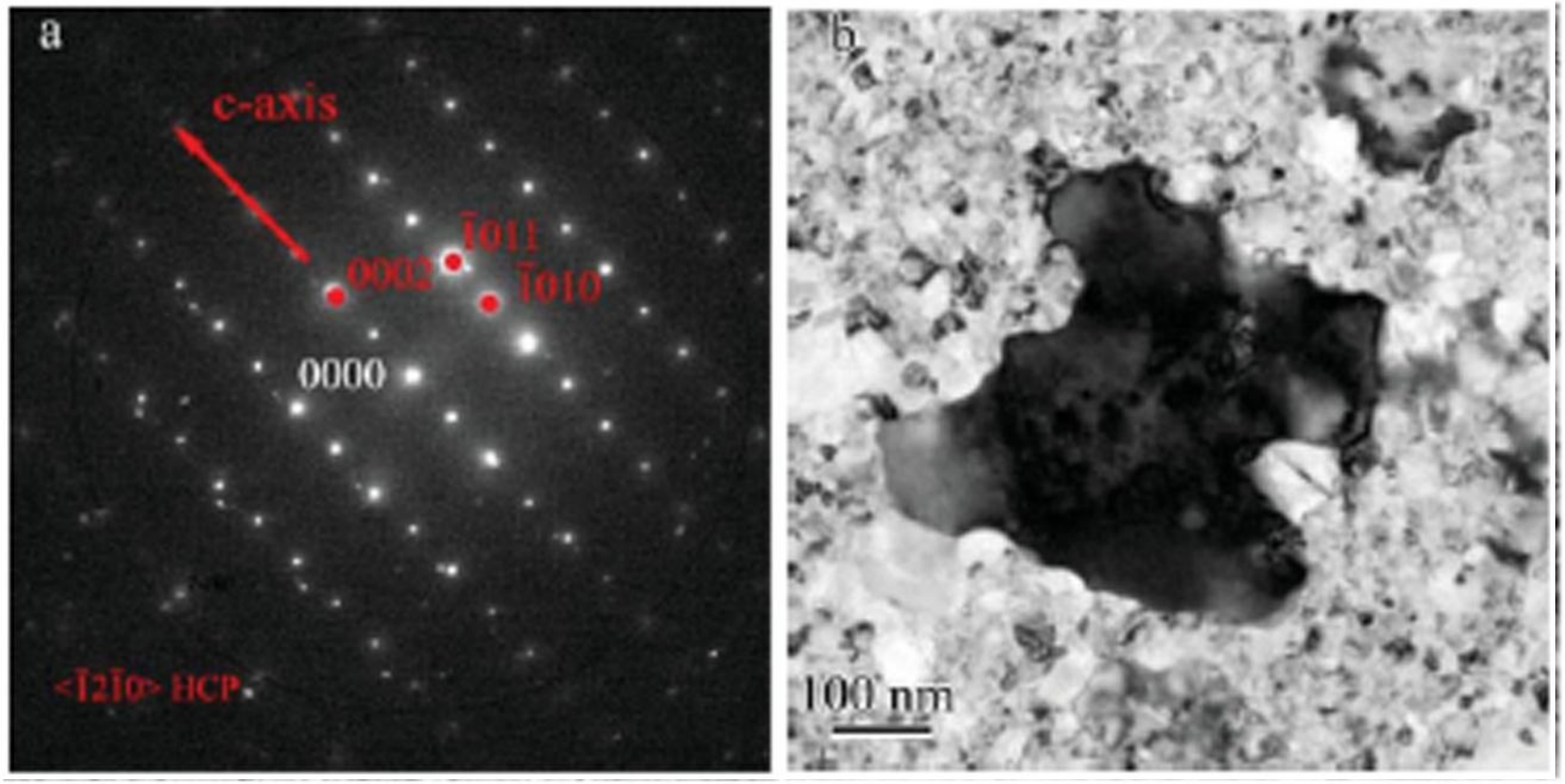


- Nanocrystalline metals are attractive because of their high strength.
- Basis for high strength is captured by the Hall – Petch relationship: $\sigma = \sigma_o + kd^{-1/2}$

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



Nanobeam 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

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

ii. What is its the local texture?

ii. How does it evolve upon annealing?

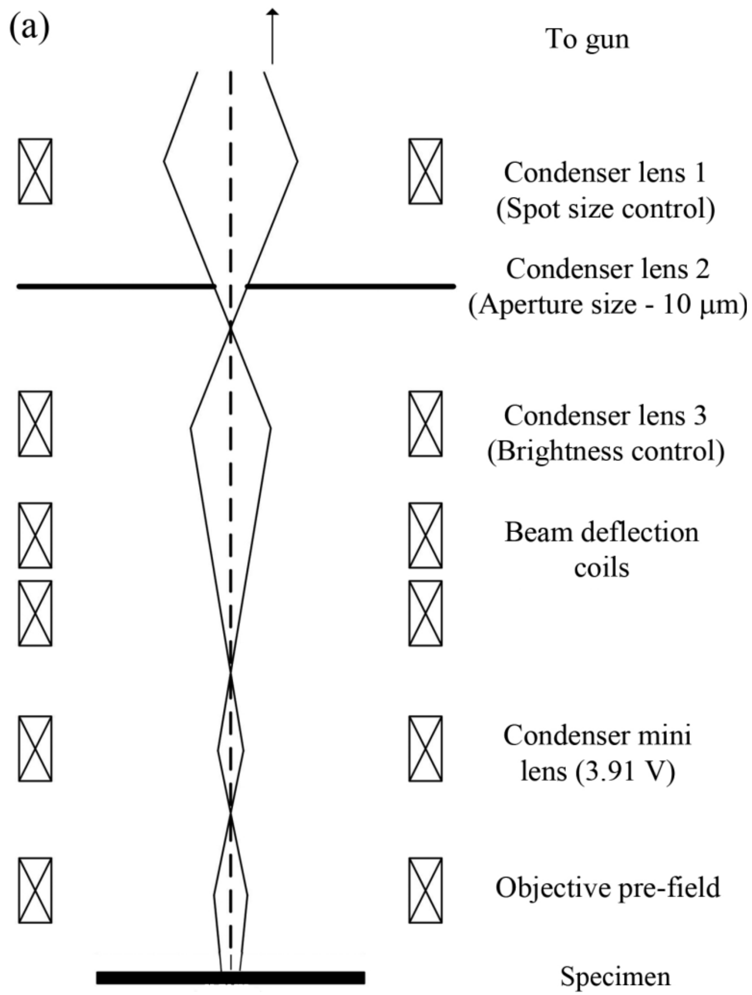
Both phase and texture in nanograined materials are not fully understood

Available electron diffraction techniques

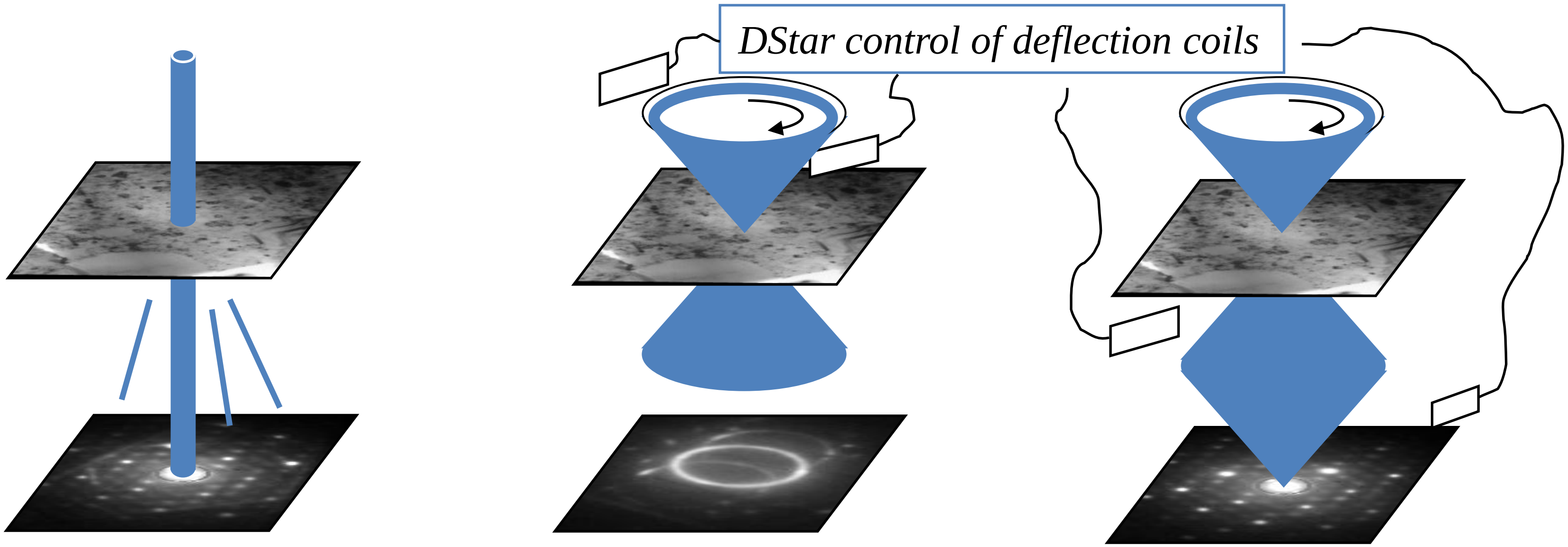
Technique	Advantages	Disadvantages
EBSD (SEM)	- Statistically significant - Accurate	- Difficult to accurately index nanoscale grains (< 30 nm) - Sample dependent
Kikuchi maps (TEM/STEM)	- Analysis of individual nanoscale particles	- Time consuming - Pole-piece gap constraint - Difficult to automate
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

Difficult to characterize nanocrystalline metals

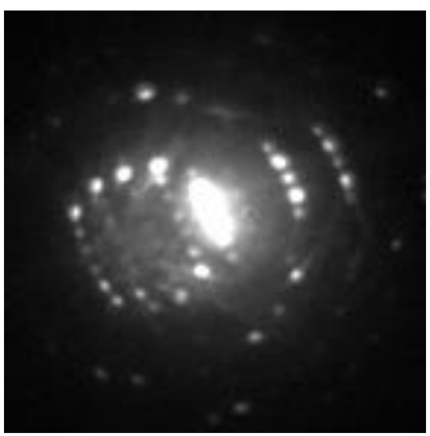
Creating a near parallel nanoscale probe

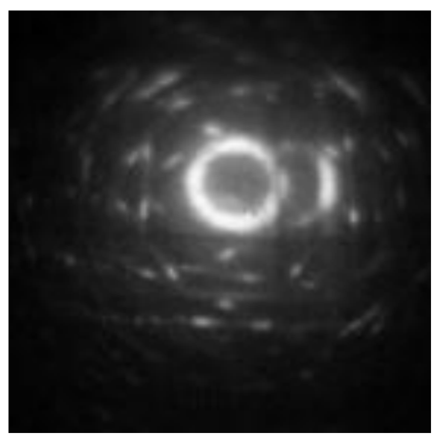


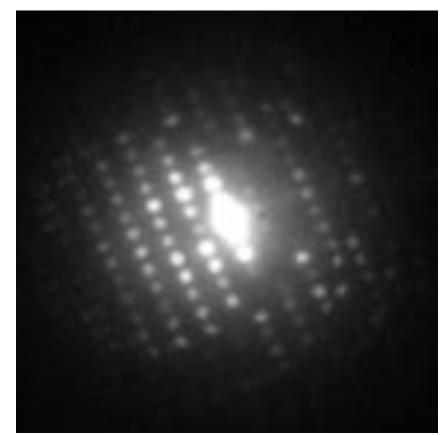
Precession microscopy



Standard setting | ½ Precession | Full precession







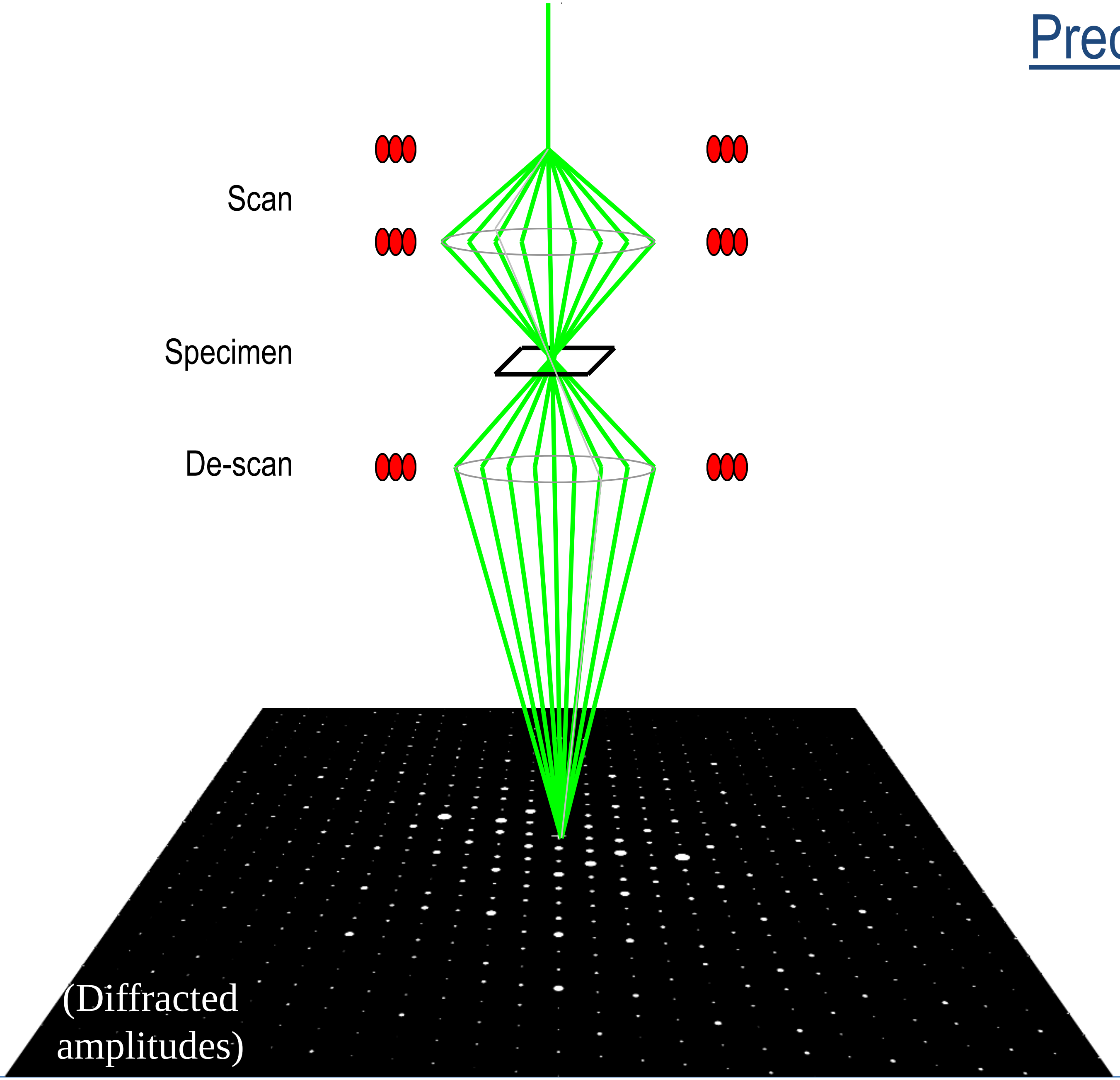
NanoMEGAS
Advanced Tools for electron diffraction

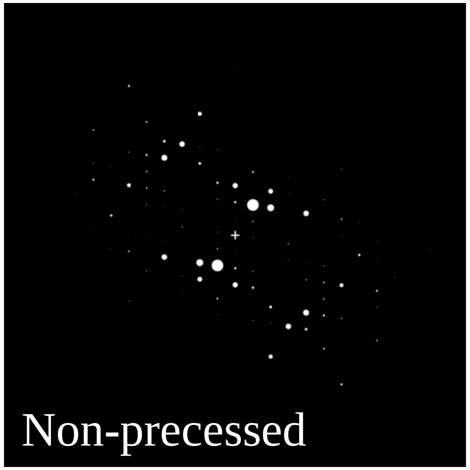
AppFive

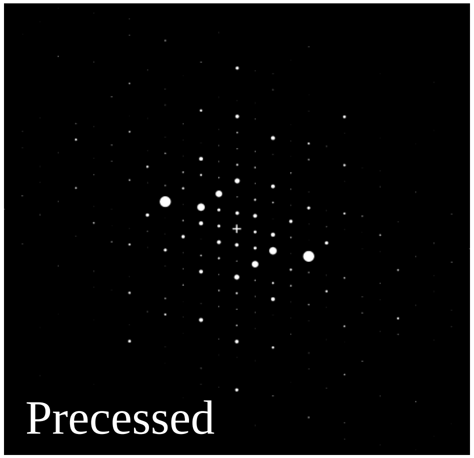
R. Vincent, P.A. Midgley, *Ultramicroscopy*, **53** (1994) 271

Slide Courtesy: E.F. Rauch

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!

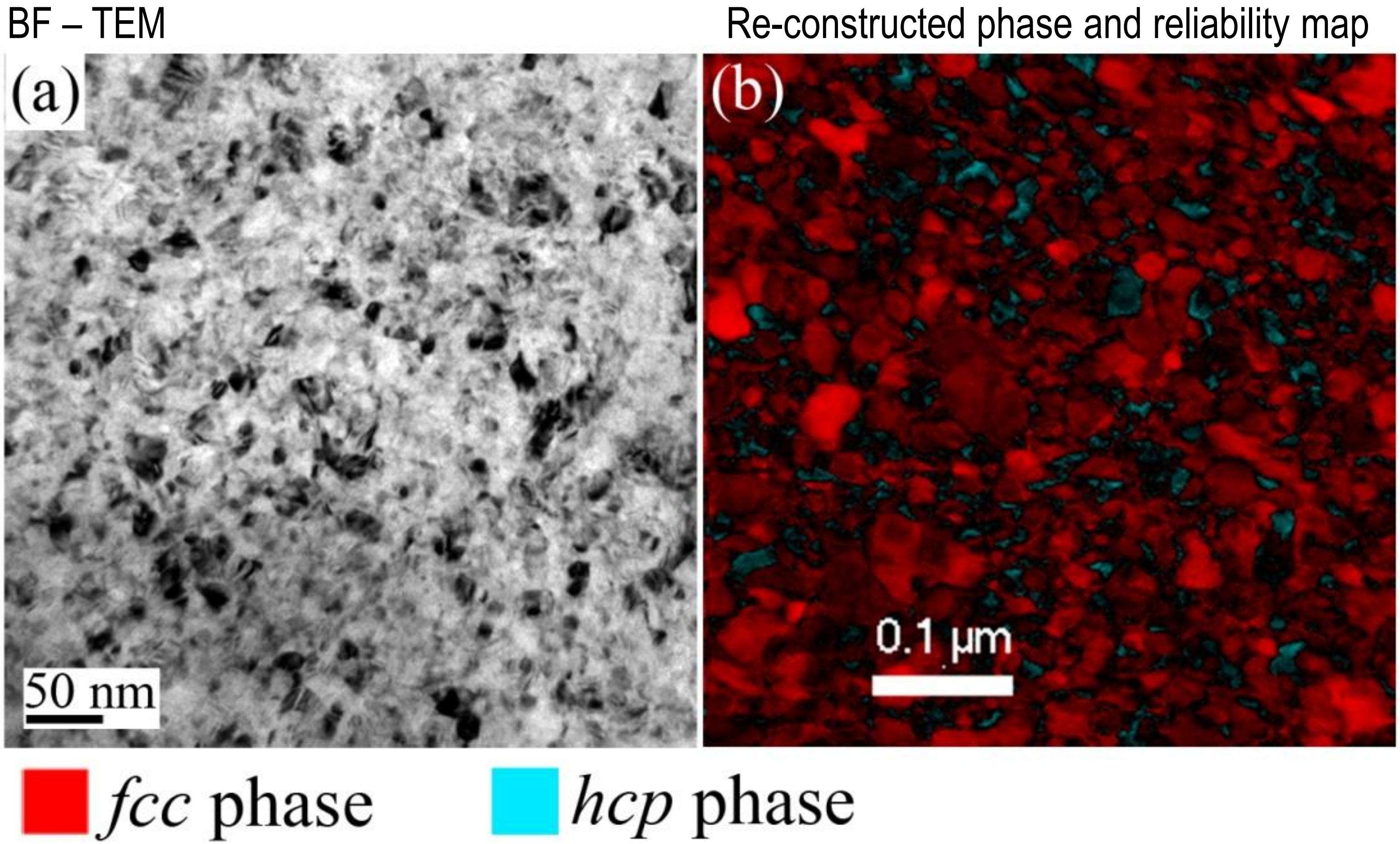
Sample preparation:

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

Microstructure evaluation:

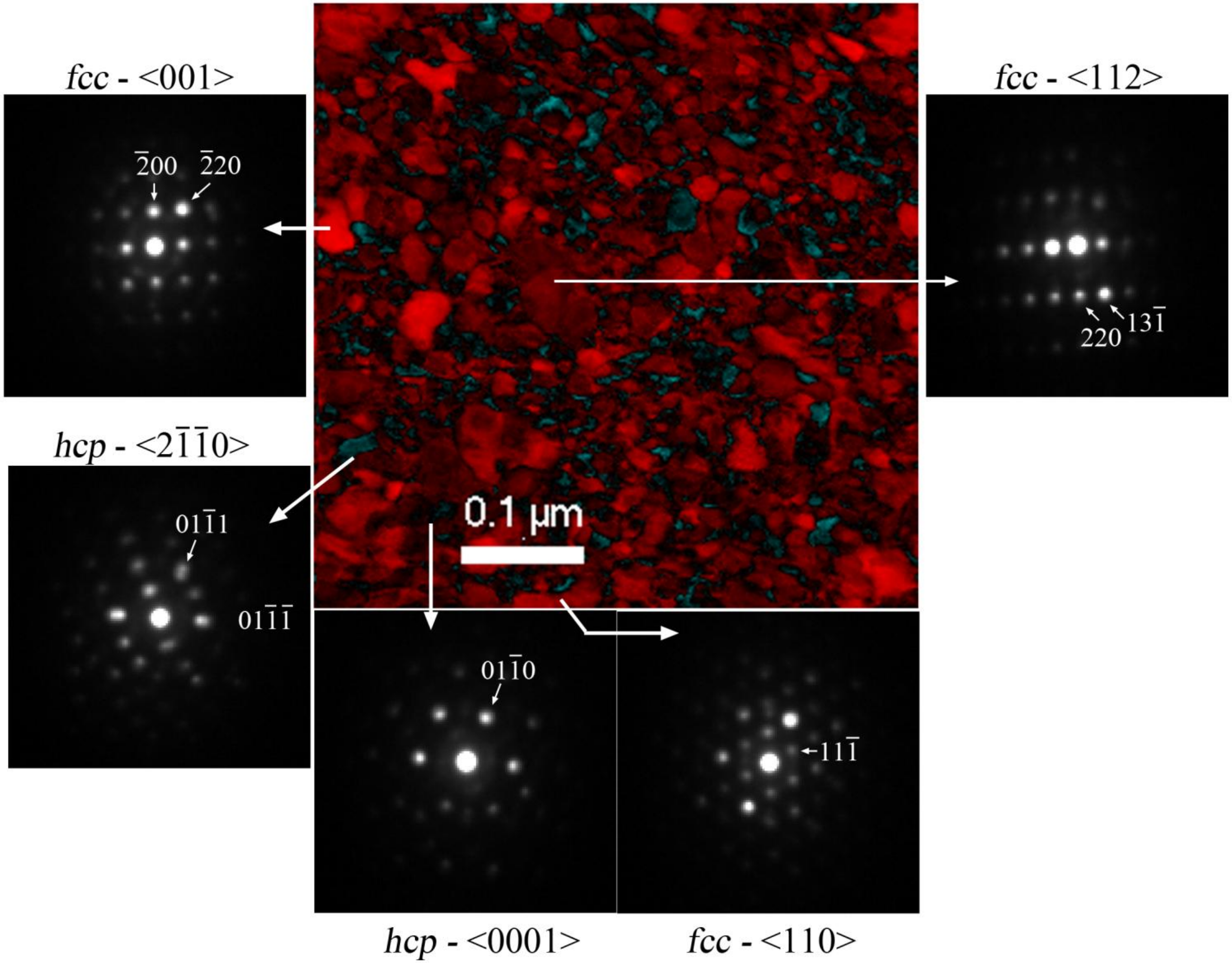
- Diffraction data acquisition by precession microscopy
- Phase and texture analysis

hcp grain size and phase fraction

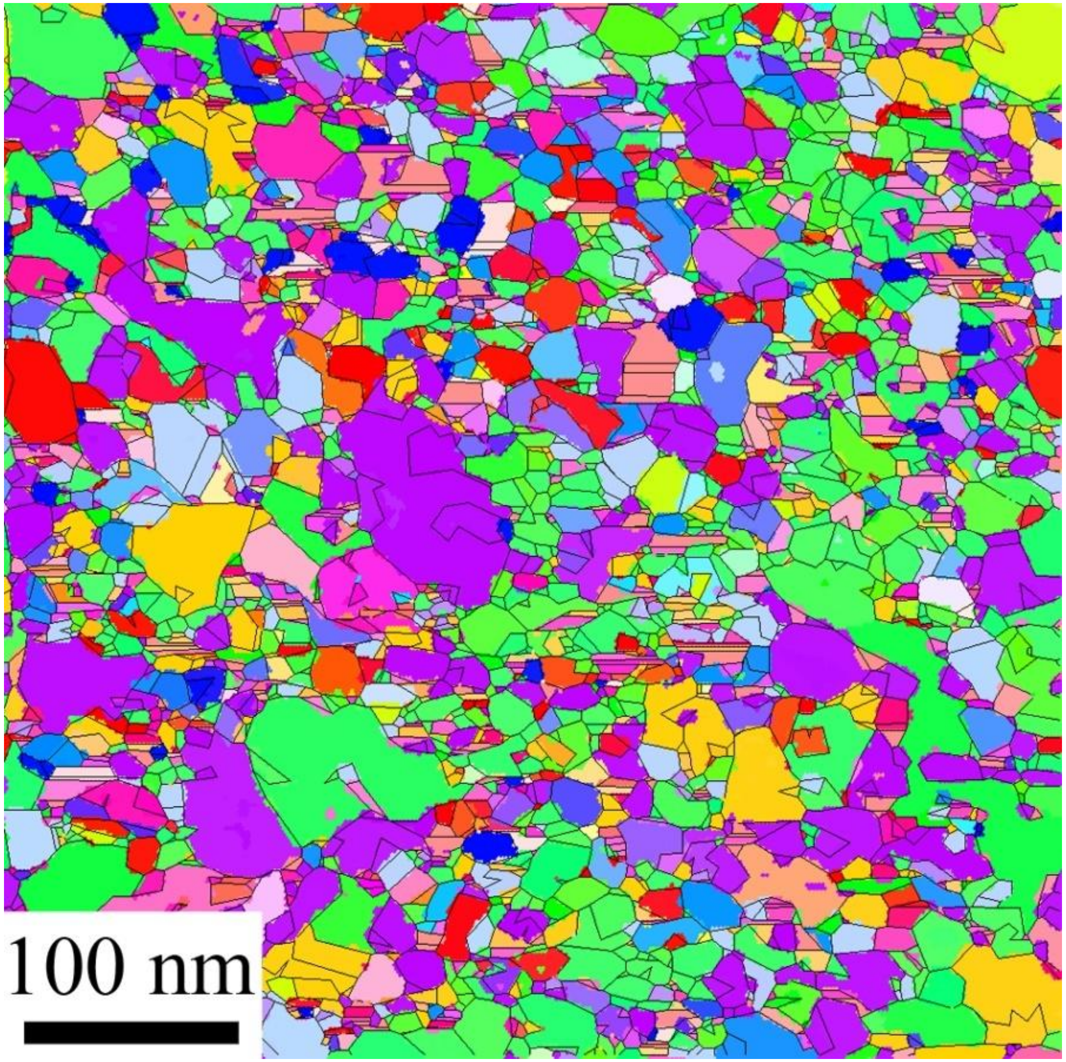


- 831 *hcp* phase grains (in 0.75 μm^2)
- Average *hcp* grain size : 10.3 nm
- Average *hcp* phase percentage: 9.0%

Phase analysis of 50 nm PLD - Ni film

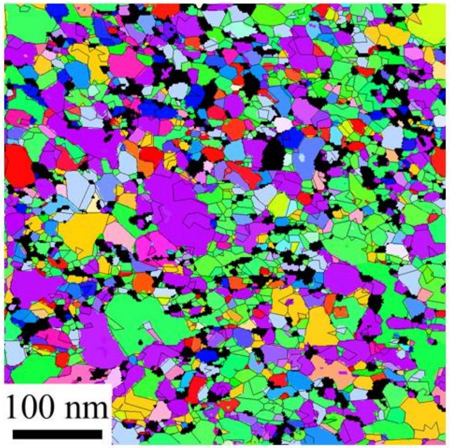


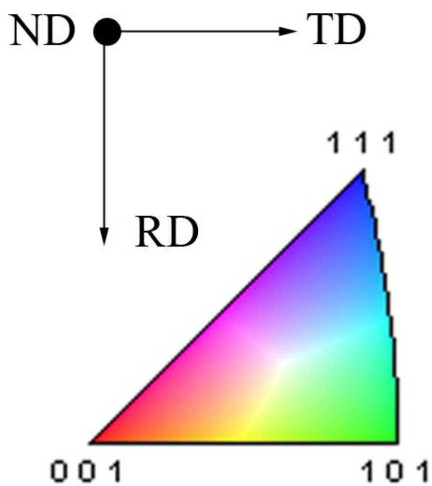
Texture of 50 nm PLD – Ni film

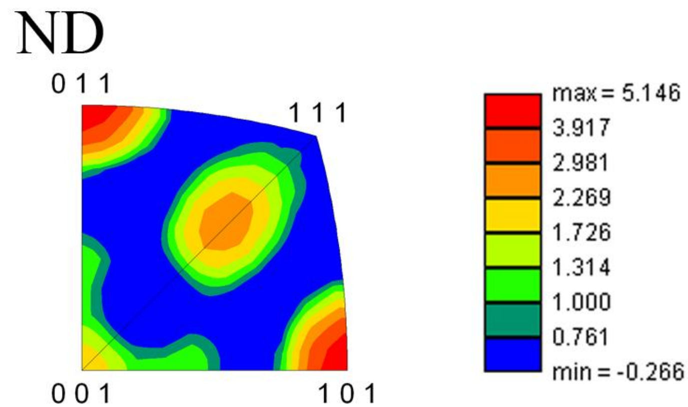


Texture of 50 nm PLD – Ni film

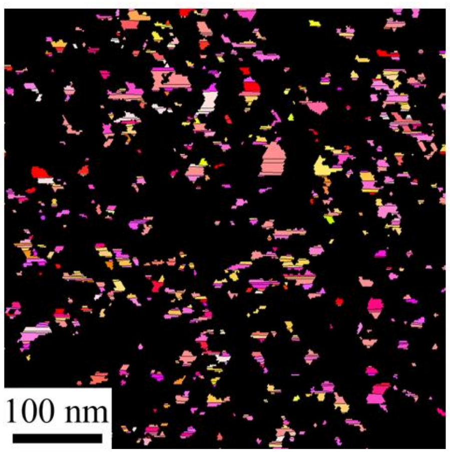
fcc nickel phase

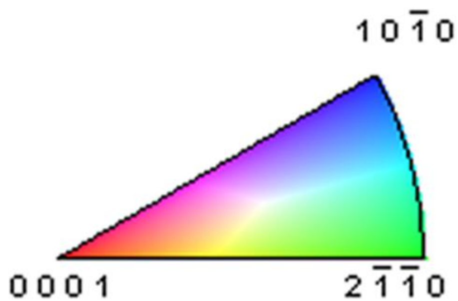


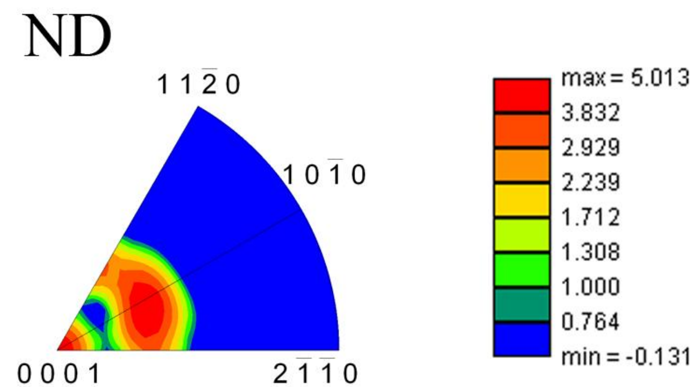




hcp nickel phase

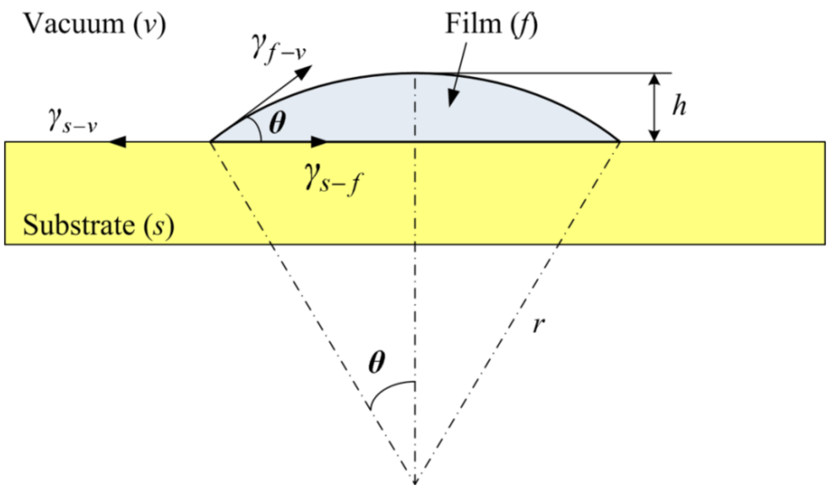






i. Why is *hcp* phase nickel present?

ii. Why the observed texture?



Assumption: Volmer-Weber type polycrystalline film growth

- **intrinsic stresses**
  - recrystallization
  - impurities
  - grain boundaries
- **substrate/film lattice mismatch**

PLD – Ni film formation

$$\Delta G_{film} = V_{film}\Delta G_v + A_{film}.\gamma_{f-v} + A_{interface}.(\gamma_{s-f} - \gamma_{s-v}) + \sigma_{gb}A_{interface}h + G_s + G_E$$

$$\Delta G_v = -\left(\frac{RT}{V_{crystal}}\right).\ln(\xi)$$

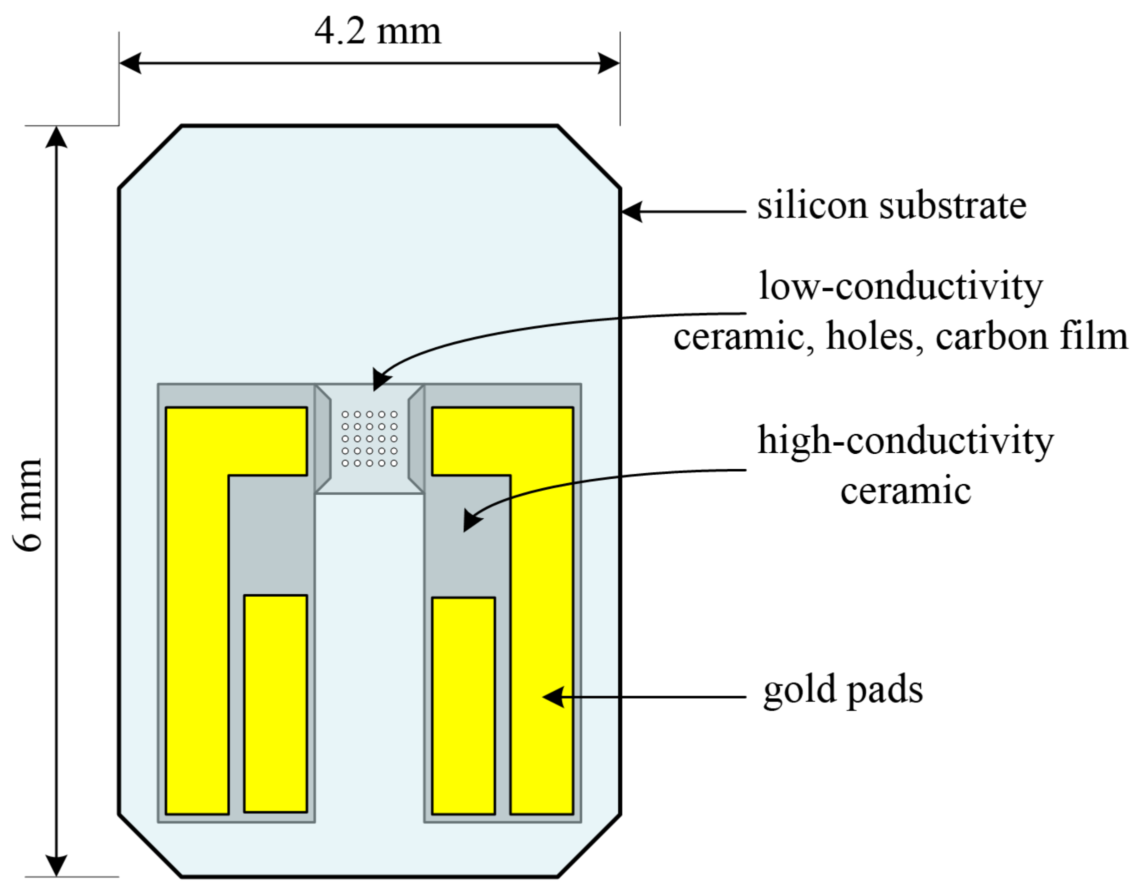
$$\sigma_{gb} = \frac{E}{(1-v)}\frac{\delta}{\bar{d}}$$

$$G_s = \frac{1}{2}\left[M_x\left(\varepsilon_x - \frac{b}{S}\right)^2 + M_y\left(\varepsilon_y - \frac{b}{S}\right)^2 + G_{xy}\gamma^2\right]A_{interface}.h$$

$$G_E = \frac{A_{interface}G_{xy}b}{4\pi(1-v)}\frac{2}{S}\log\left(\frac{\beta h}{b}\right)$$

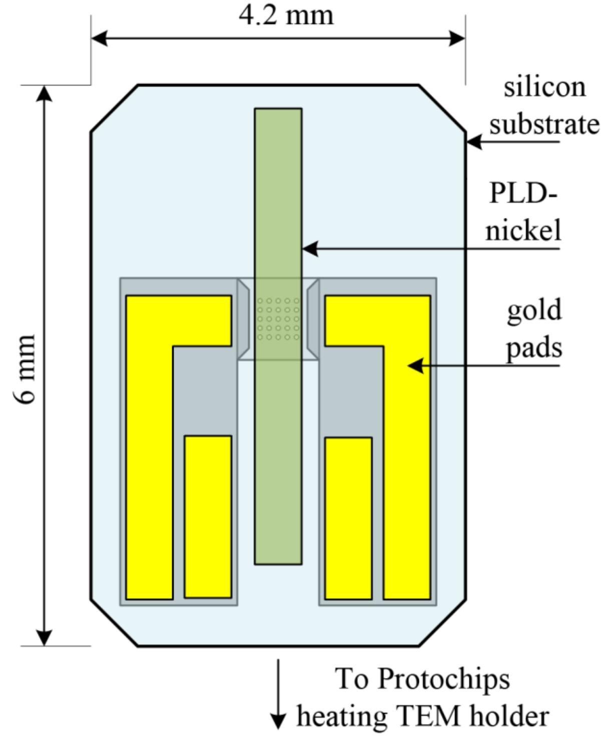
Phase and texture evolution

- in-situ annealing in Aduro™ Protochips devices
- Acquisition of diffraction data by precession microscopy



Sample preparation:

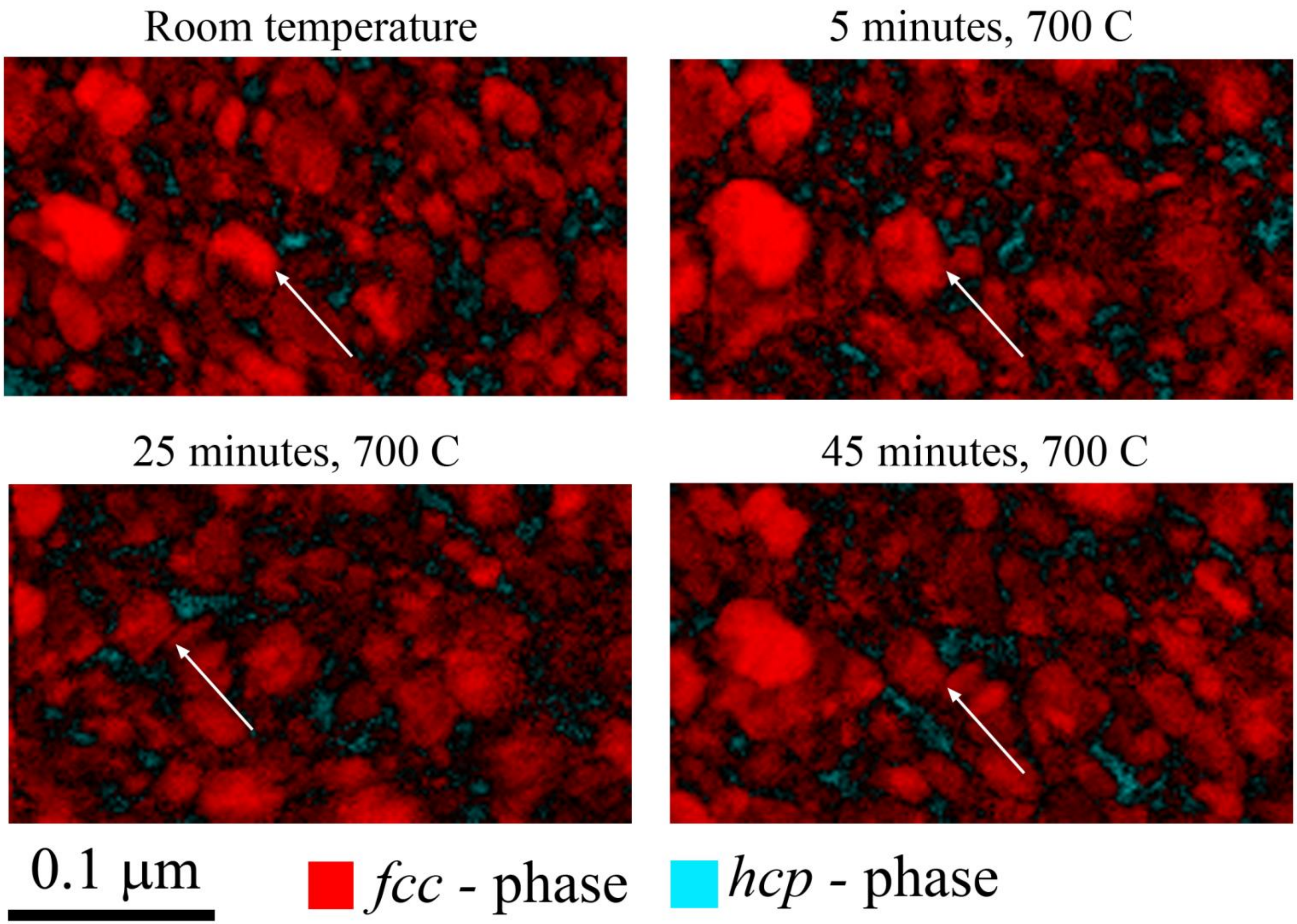
- Pulsed Laser Deposition of 25 nm nickel film on Aduro^{TM} Protochips devices



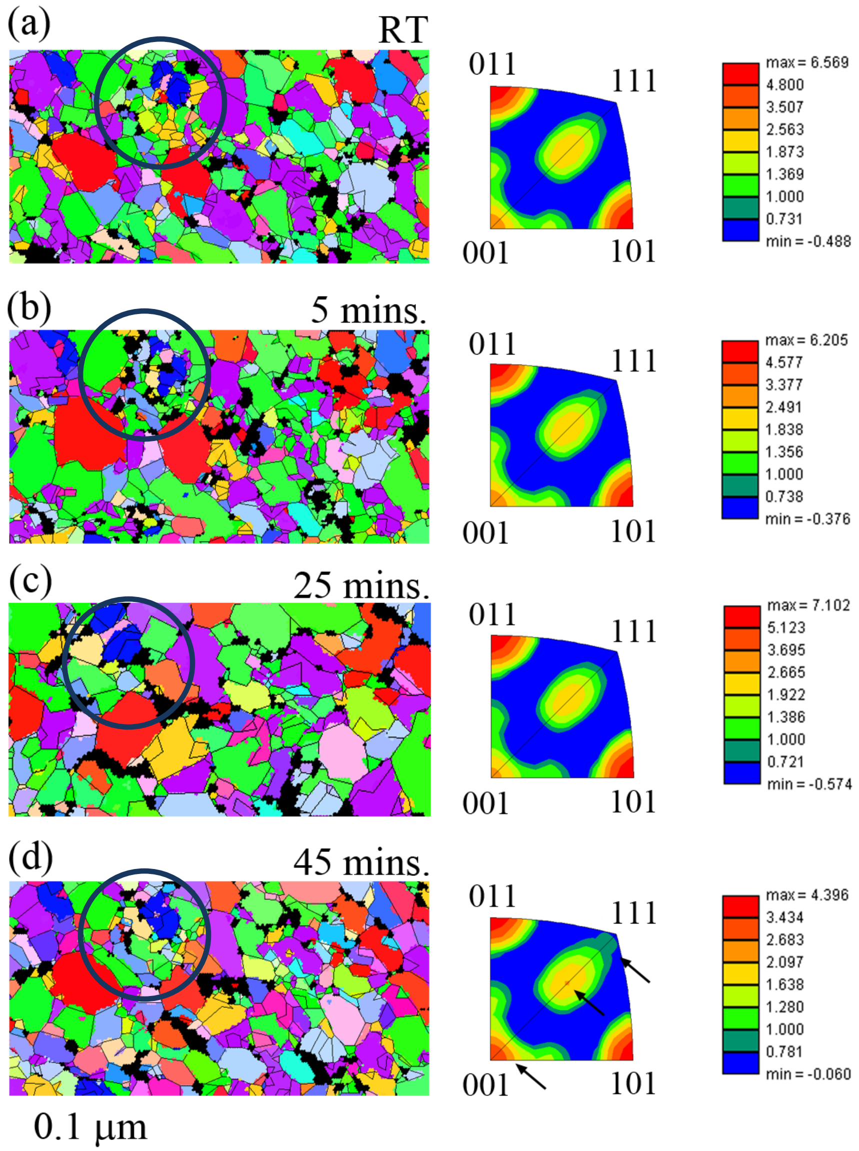
In-situ heating and Precession microscopy:

- Isothermal heating at 700 °C
- Diffraction data acquisition at room temperature, 5, 25 and 45 minutes of annealing

in-situ heating and Precession microscopy



Texture evolution – *fcc* phase

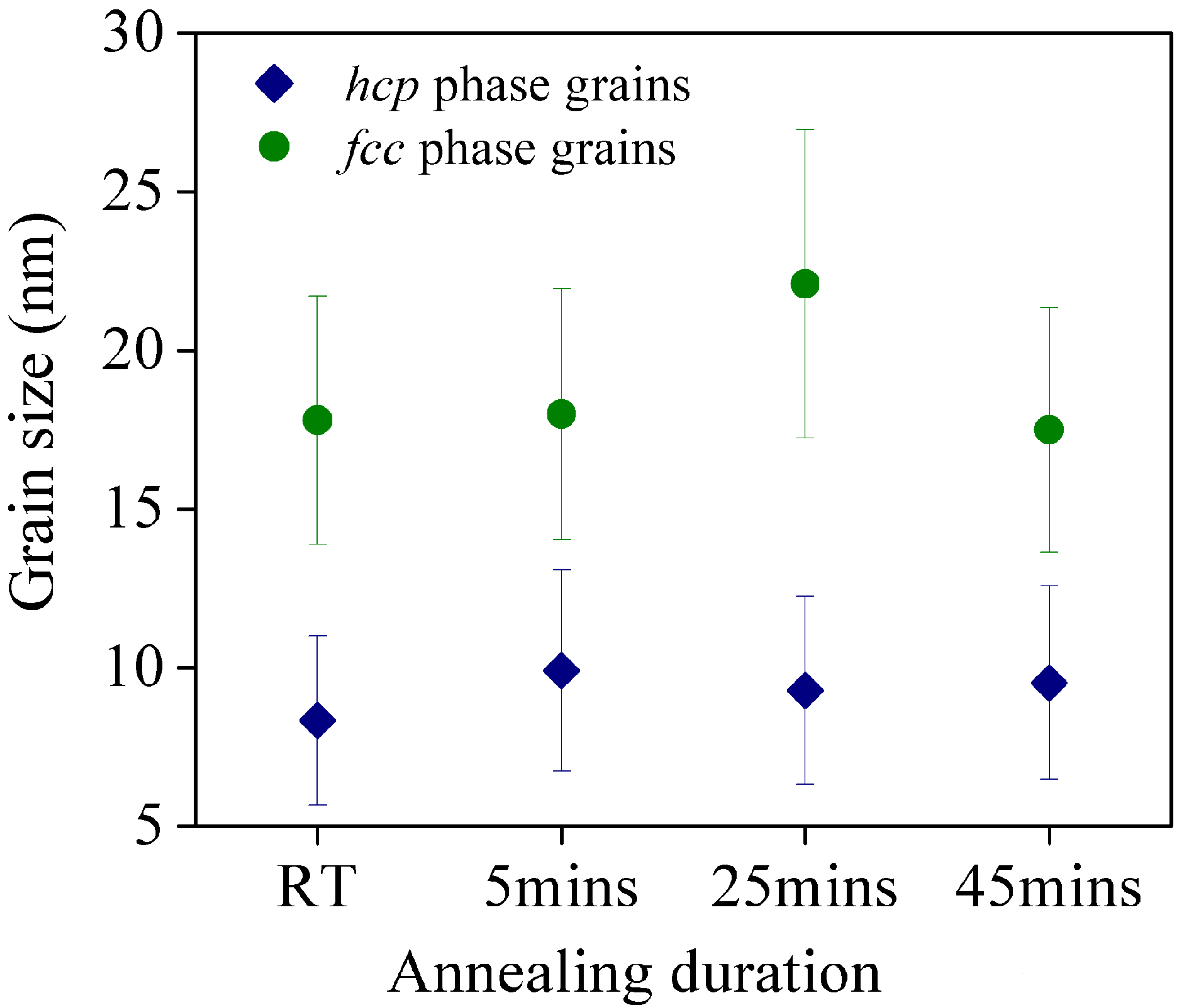


<011> // ND

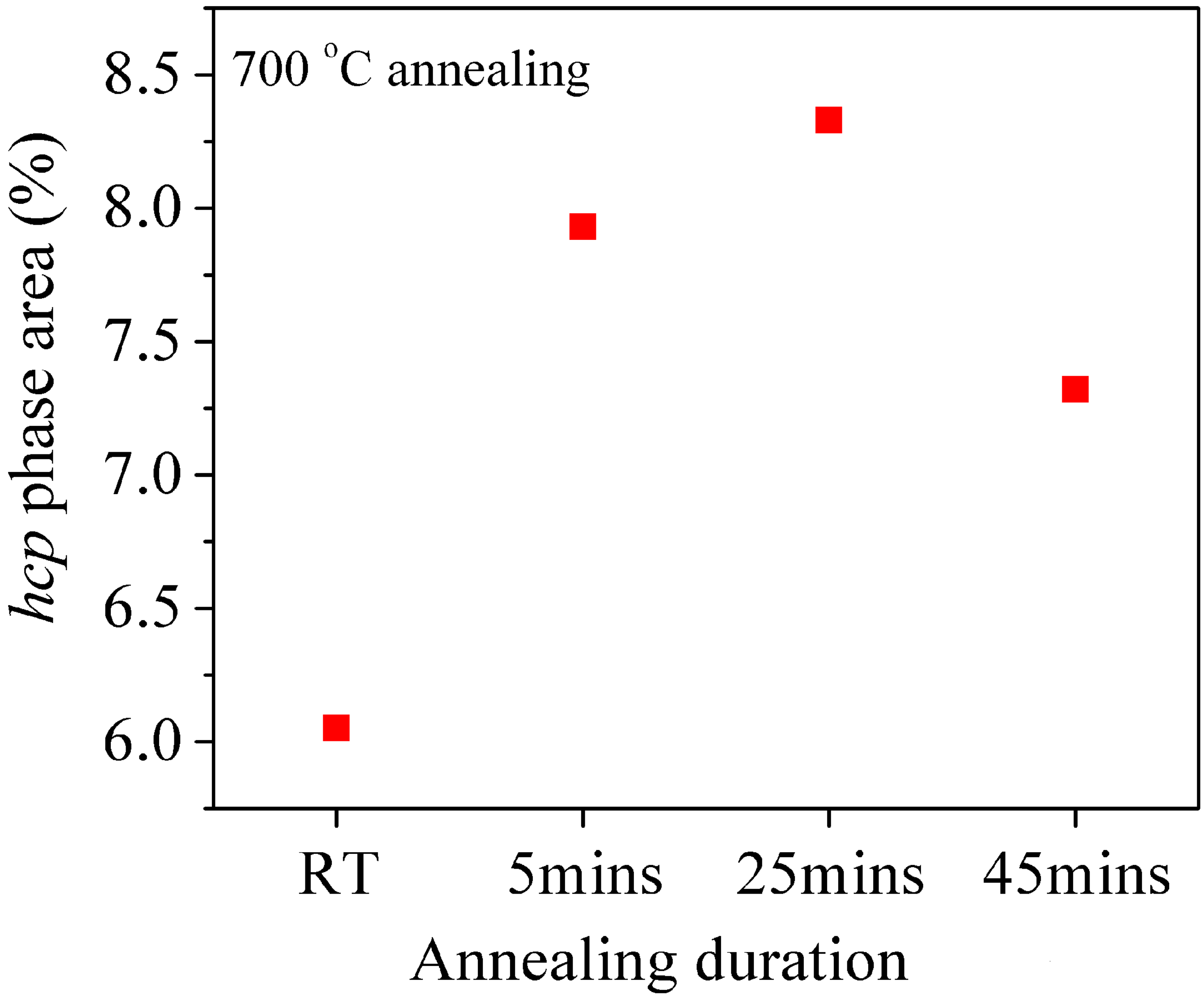
Progression of annealing

<001> // ND, <112> // ND

Grain size evolution



hcp phase evolution



Conclusions

i. Nanoscale *hcp* nickel phase in as-deposited 50 nm film

ii. Approximately 10 nm in size, and with close to <0001> // ND texture

iii. Nanoscale *fcc* nickel phase is approximately 20 nm size, <011> // ND texture

iv. *fcc* nickel phase texture evolves from <011> // ND to <001> // ND and <112> // ND texture on isothermal annealing

Possible directions of future research

i. Analysis of the grain boundary types
ii. Influence of film thickness
iii. Texture and phase evolution in free-standing nickel films
iv. Texture evolution in constrained and free-standing metal films
v. Microstructure evolution in ion-irradiated materials