

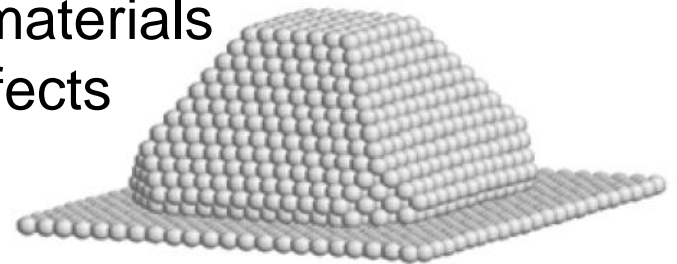


NTNU

Norwegian University of
Science and Technology

TMT4320 Nanomaterials **August 29th, 2016**

- Unique properties of Nanomaterials
- Physical properties: size effects
- TNN. Chapter 2.
- NN. Chapter 8.



Last week

- Introduction to Nanomaterials
 - Definition of Nanomaterials
 - Introduction to properties, applications, challenges and concerns
 - Problem A
- Unique properties of Nanomaterials
 - Basic definitions
 - Classification: 0D, 1D, 2D, 3D

Next lecture

- Defects
 - Examples
 - Problem B
- Physical properties: size effects
 - Lattice constant
 - Melting point

Microstructure and defects

Defects in Crystalline materials

How are defects classified?

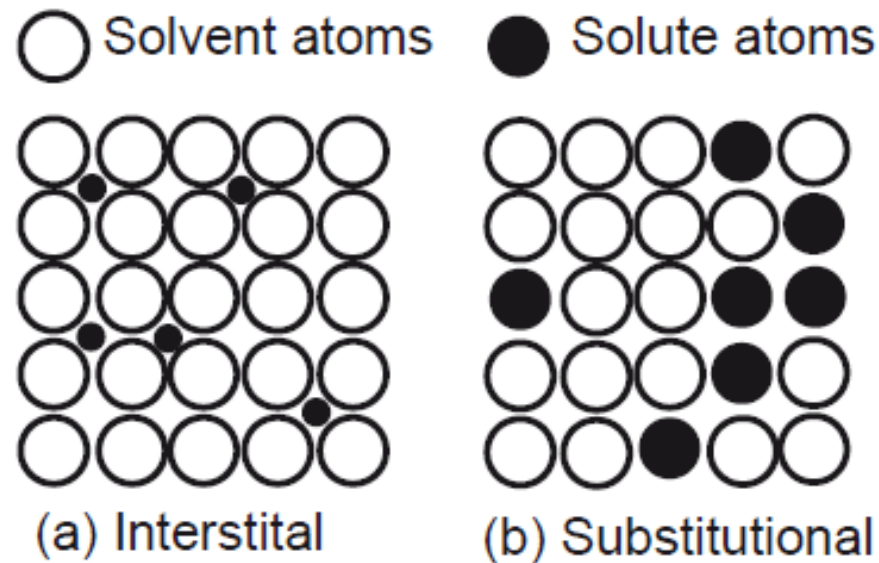
- Point defects (0D)
 - Metal and alloys → Vacancies, substitutional and interstitial
 - Ionic solids → Schottky (anion-cation vacancy pairs) and Frenkel (vacancy-interstitial pairs) defects
- Line defects (1D)
 - Dislocations: missing plane of atoms
- Surface defects (2D)
 - Grain boundaries, twins, stacking faults and free surfaces.
- Volume defects (3D)
 - Voids and microcracks.

Microstructure and defects

Defects in Crystalline materials

How are defects classified?

- Point defects (0D)
 - Metal and alloys → Vacancies, substitutional and interstitial



Common point defects

Microstructure and defects

Point defects (0D)

Metal and alloys → Vacancies, substitutional and interstitial

7475 Al alloys

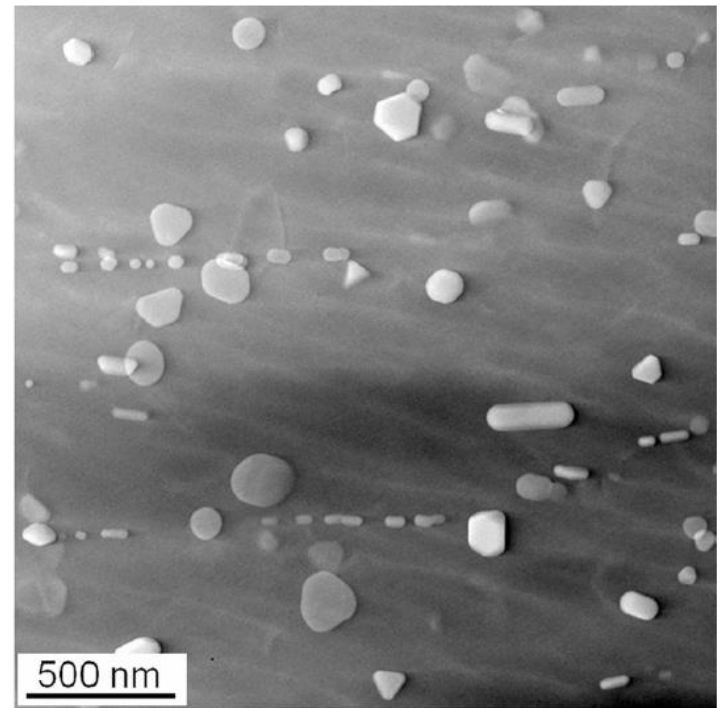


Fig. 1 HAADF-STEM micrograph of $\text{Al}_{18}\text{Cr}_2\text{Mg}_3$ particles in the 7475 Al alloy sample after solid solution treatment

Microstructure and defects

Point defects (0D)

Metal and alloys → Vacancies, substitutional and interstitial

7475 Al alloys

Particles ranges
from 50 to 200 nm

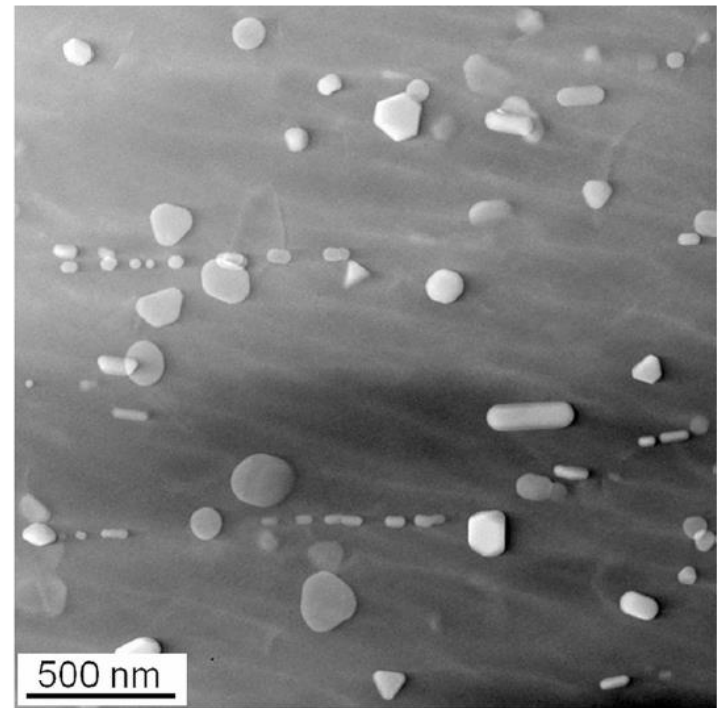


Fig. 1 HAADF-STEM micrograph of $\text{Al}_{18}\text{Cr}_2\text{Mg}_3$ particles in the 7475 Al alloy sample after solid solution treatment

Microstructure and defects

Point defects (0D)

Metal and alloys → Vacancies, substitutional and interstitial

7475 Al alloys

Particles ranges
from 50 to 200 nm

Study about the
stability of the alloy:
crystal-to-amorphous
(C-A) transition
A-C transition

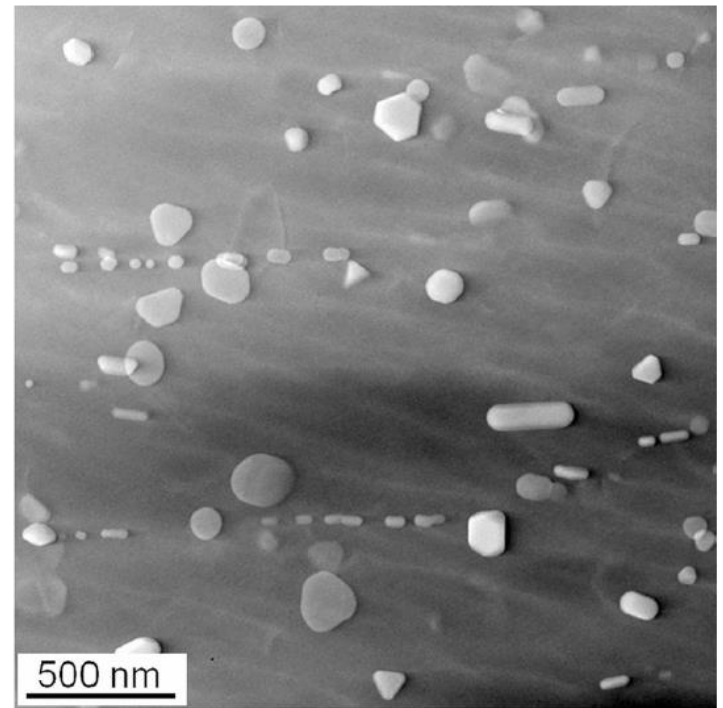


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Microstructure and defects

Point defects (0D)

Metal and alloys → Vacancies, substitutional and interstitial

7475 Al alloys

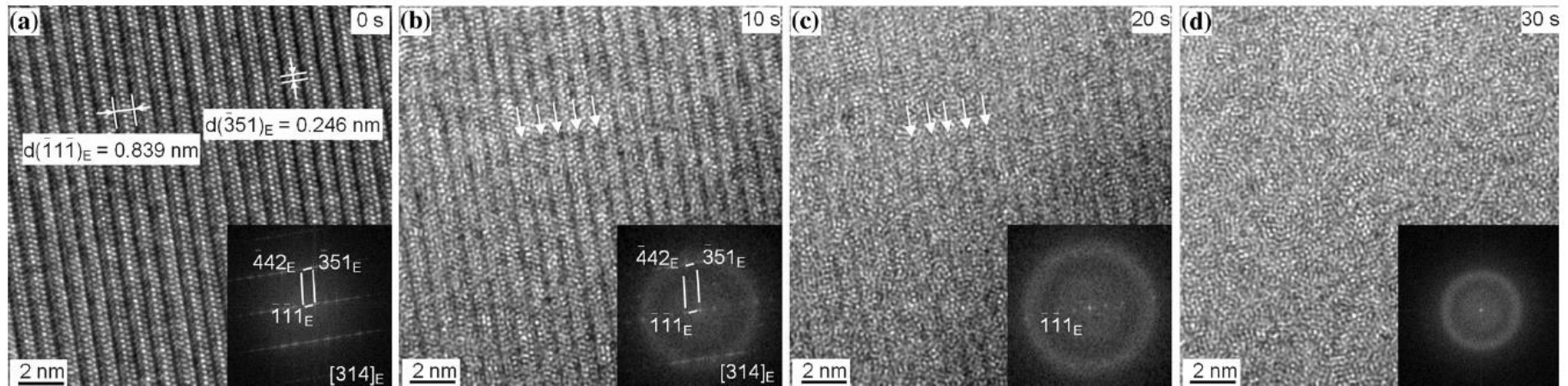


Fig. 2 HRTEM micrographs sequence showing C–A transition in the thick $\text{Al}_{18}\text{Cr}_2\text{Mg}_3$ particle by 300 keV electron irradiation (insets are the corresponding FFT spectra): **a** Before irradiation; **b** After 10 s irradiation; **c** After 20 s irradiation; **d** After 30 s irradiation

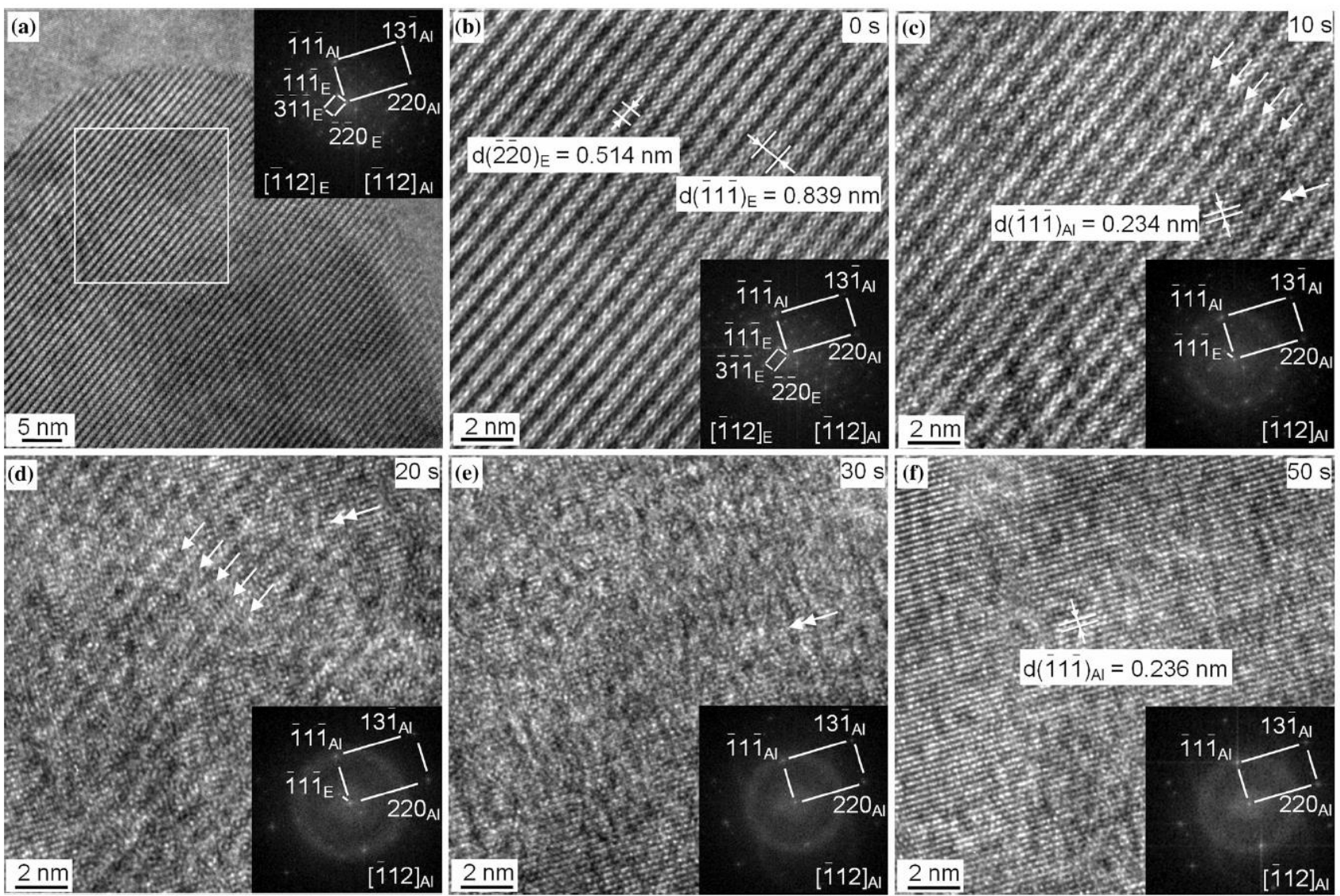


Fig. 3 HRTEM micrographs sequence showing dissolution in the thin $\text{Al}_{18}\text{Cr}_2\text{Mg}_3$ particle by 300 keV electrons irradiation (insets are the corresponding FFT spectra): **a** before irradiation at low magnification; **b** magnifications of the white rectangular region in Fig. 3a before irradiation; **c** the same region of Fig. 3b after 10 s irradiation; **d** the same region of Fig. 3b after 20 s irradiation; **e** the same region of Fig. 3b after 30 s irradiation; **f** the same region of Fig. 3b after 50 s irradiation

Microstructure and defects

Point defects (0D)

Metal and alloys → Vacancies, substitutional and interstitial

7475 Al alloys

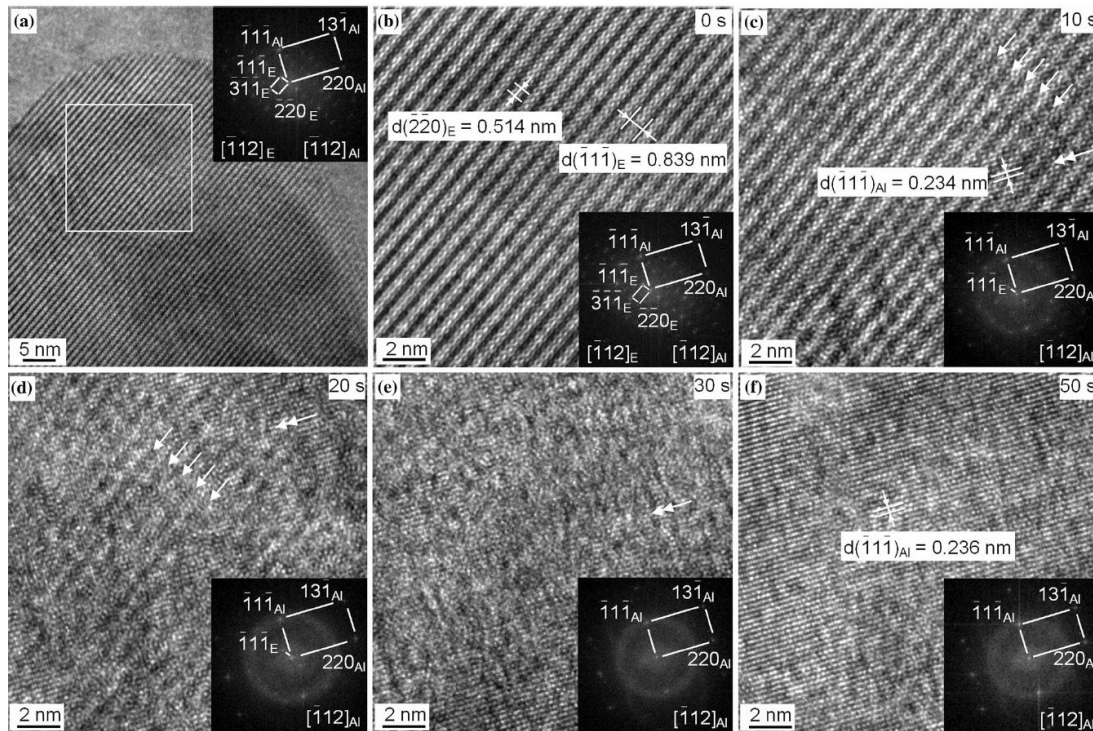


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Amorphization is caused by a combination of chemical disordering and an increase in point defect concentration

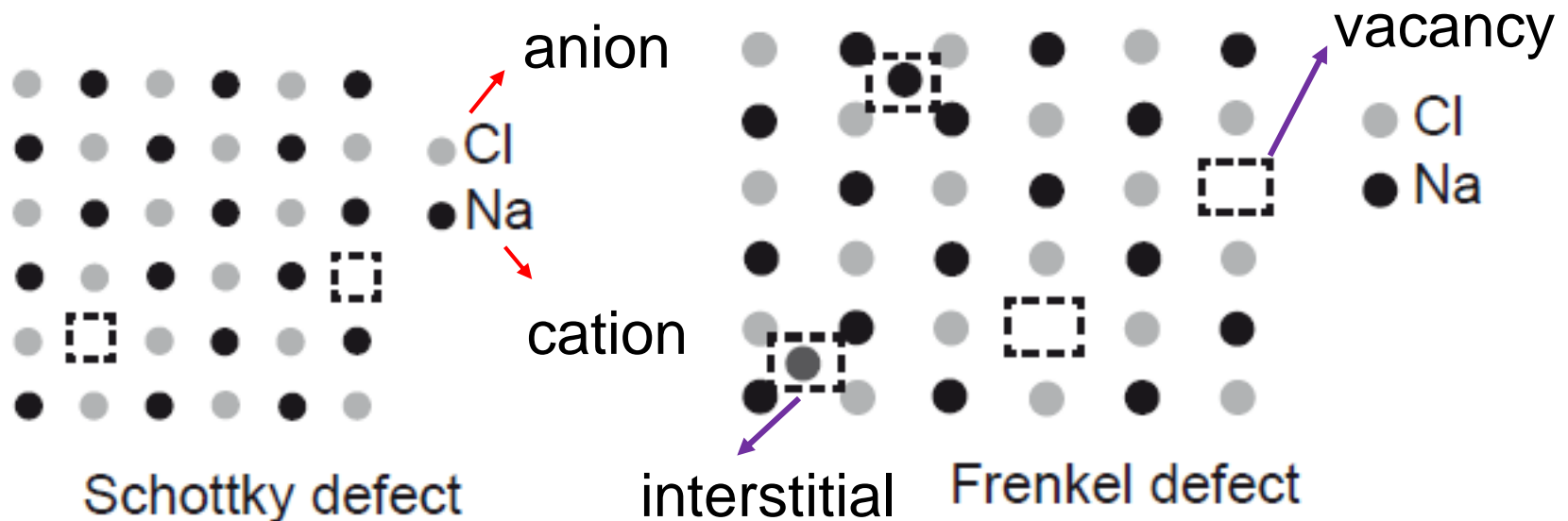
Microstructure and defects

Defects in Crystalline materials

How are defects classified?

– Point defects (0D)

- Metal and alloys → Vacancies, substitutional and interstitial
- Ionic solids → **Schottky** (anion-cation vacancy pairs) and **Frenkel** (vacancy-interstitial pairs) defects



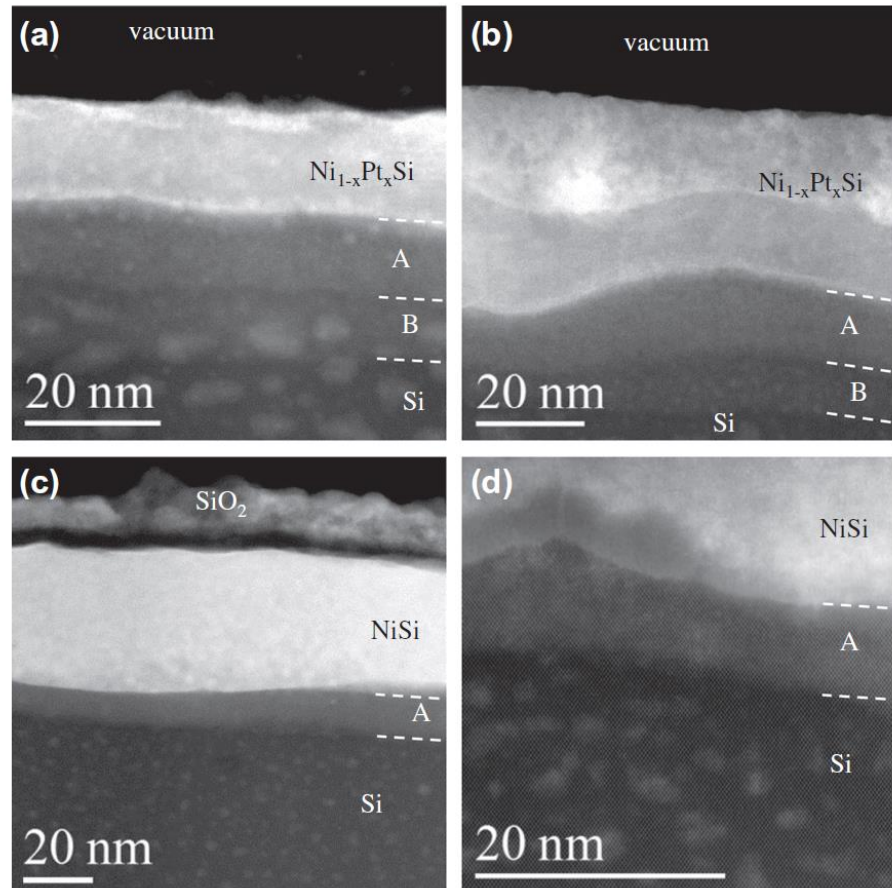
Microstructure and defects

Point defects (0D)

- Ionic solids → **Frenkel** (vacancy-interstitial pairs) defects

$\text{Ni}_{1-x}\text{Pt}_x\text{Si}$ films

Pt doping
alternative to NiSi
film for integrated
circuits



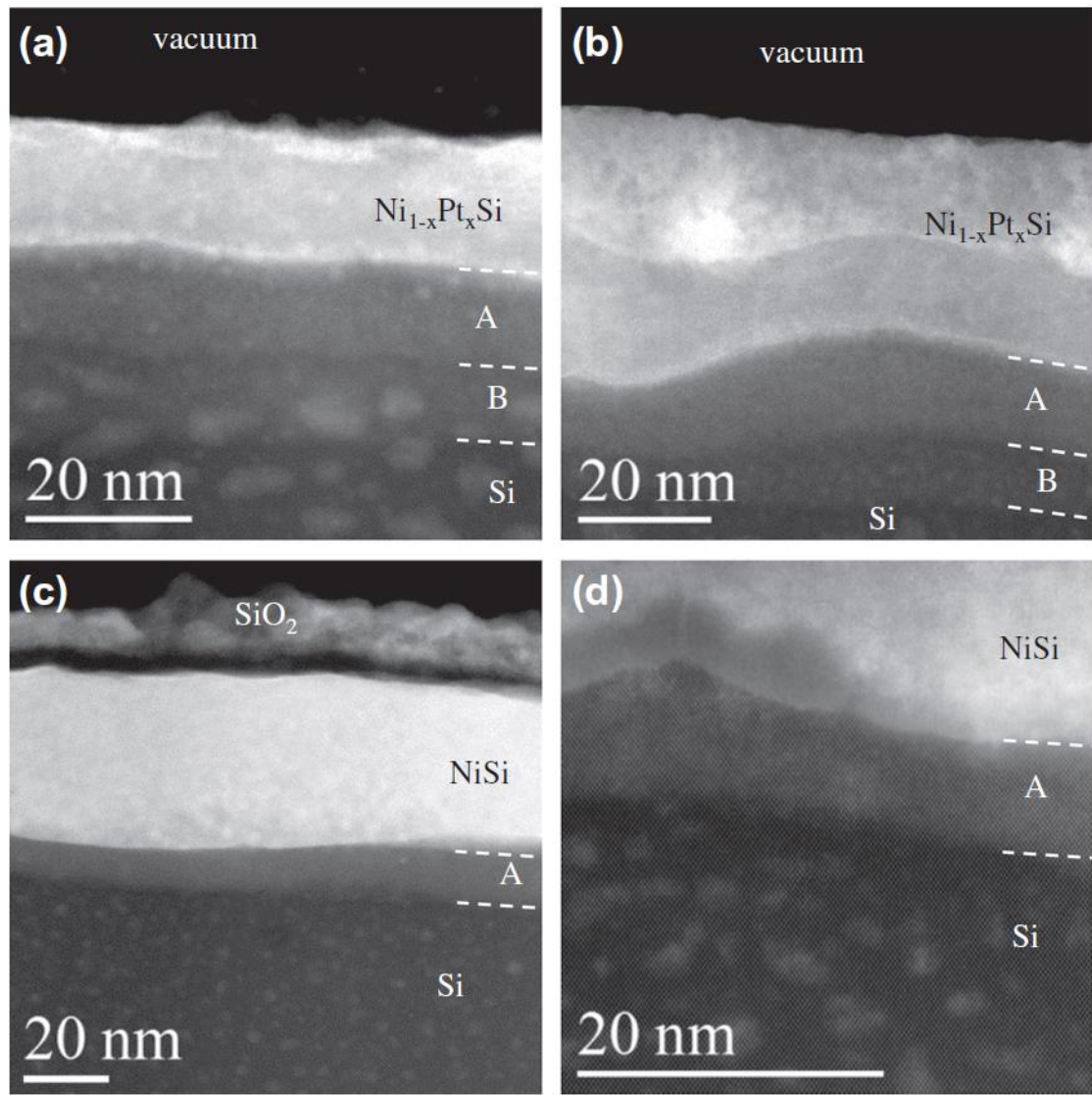


Fig. 1. HAADF STEM images of $\text{Ni}_{1-x}\text{Pt}_x\text{Si}$ films recorded in $\langle 110 \rangle$ zone-axis orientation for the silicon substrate. The interfaces in (a) and (b) correspond to samples alloyed with 10 at.% Pt and RTA1 temperatures of 280 °C and 330 °C, respectively. The HAADF STEM images in (c) and (d) show interface structures in the absence of platinum for RTA1 at temperatures of 280 and 330 °C, respectively.

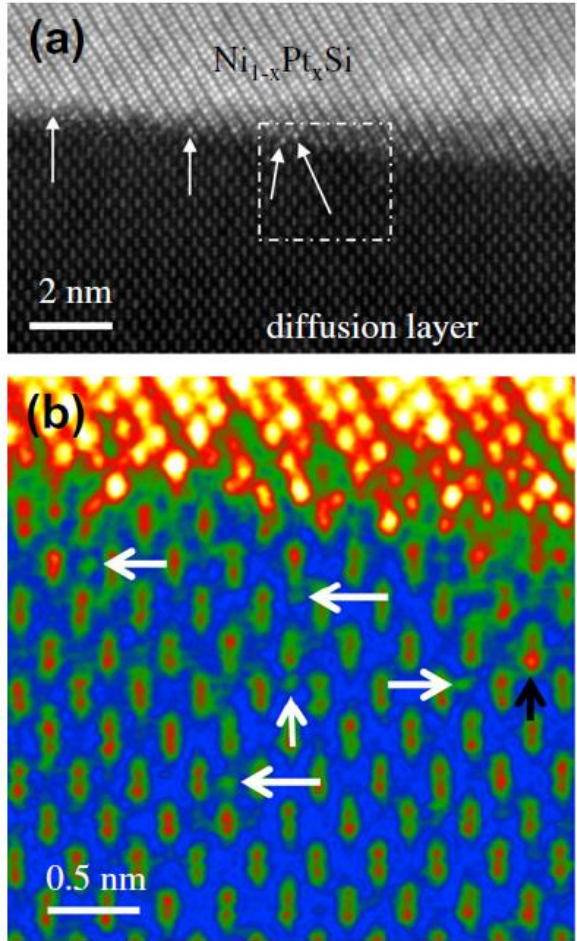


Fig. 3. (a) High-resolution HAADF STEM image of the NiSi/Si interface in $\langle 110 \rangle$ zone-axis orientation for the silicon substrate. Individual Pt atoms at the interface are observed as bright intensities marked by the white vertical arrows. (b) Magnified view of the area marked in (a). The white arrows highlight different Ni interstitial configurations observed below the interface. The black arrow highlights an example of a substitutional Ni atom below the interface.

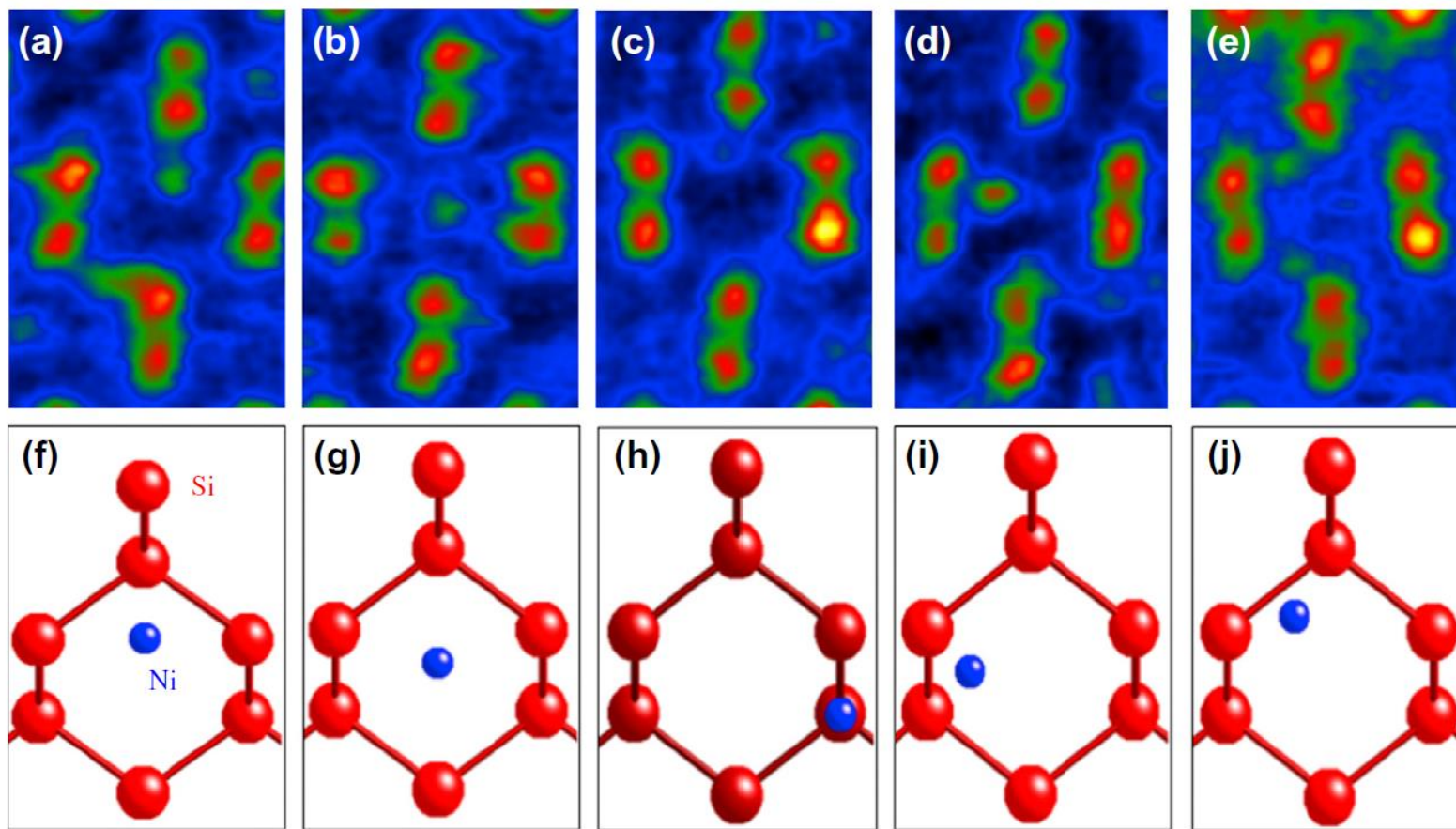
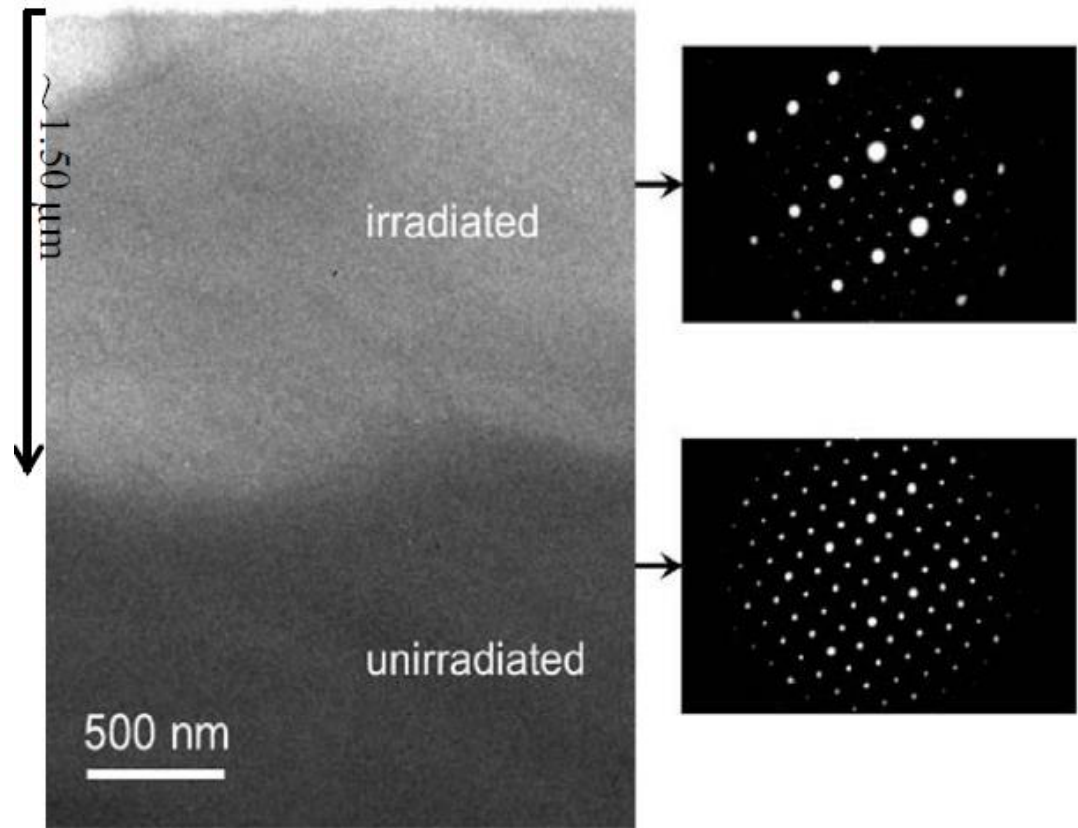
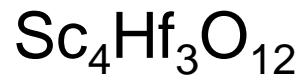


Fig. 4. (a–e) Selected areas extracted from the HAADF STEM image in Fig. 3b. (f–j) Ball-and-stick models of the corresponding experimental observations. Si atoms are red, Ni atoms are blue. The models are projected views along the $\langle 110 \rangle$ direction in silicon. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Microstructure and defects

Point defects (0D)

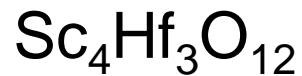
- Ionic solids → **Schottky** (anion-cation vacancy pairs)



Microstructure and defects

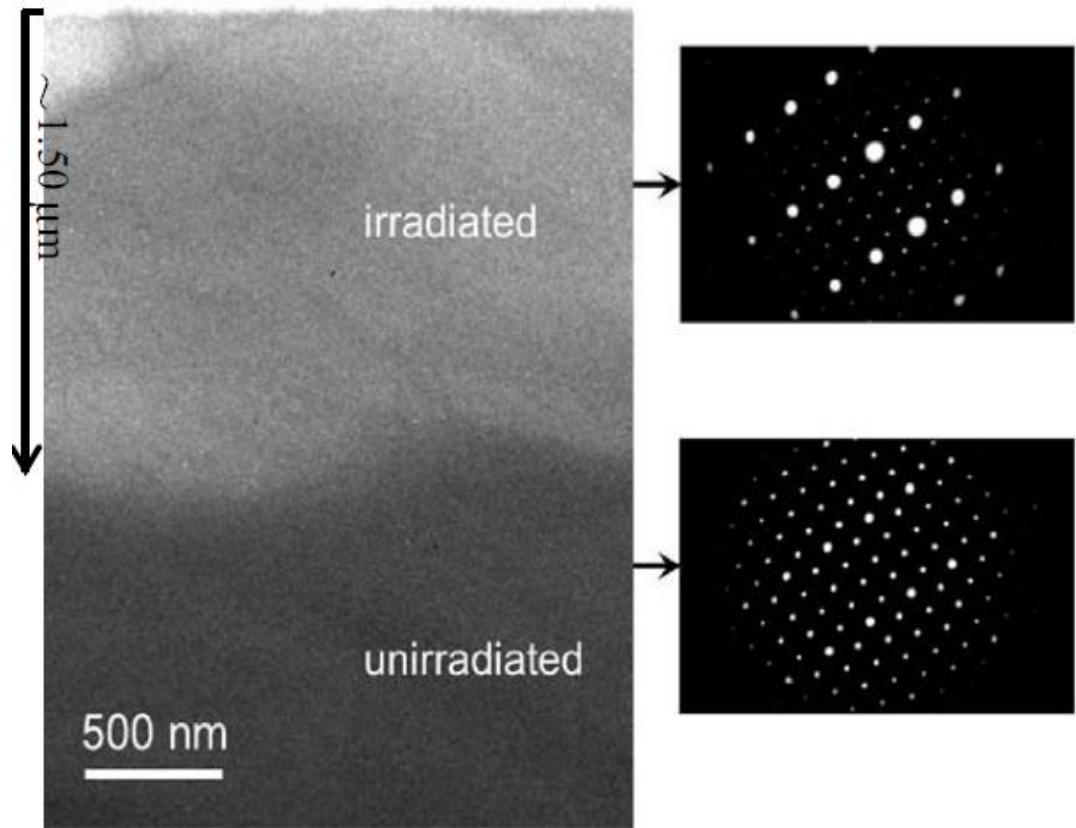
Point defects (0D)

- Ionic solids → **Schottky** (anion-cation vacancy pairs)



Oxygen vacancies

Problem B

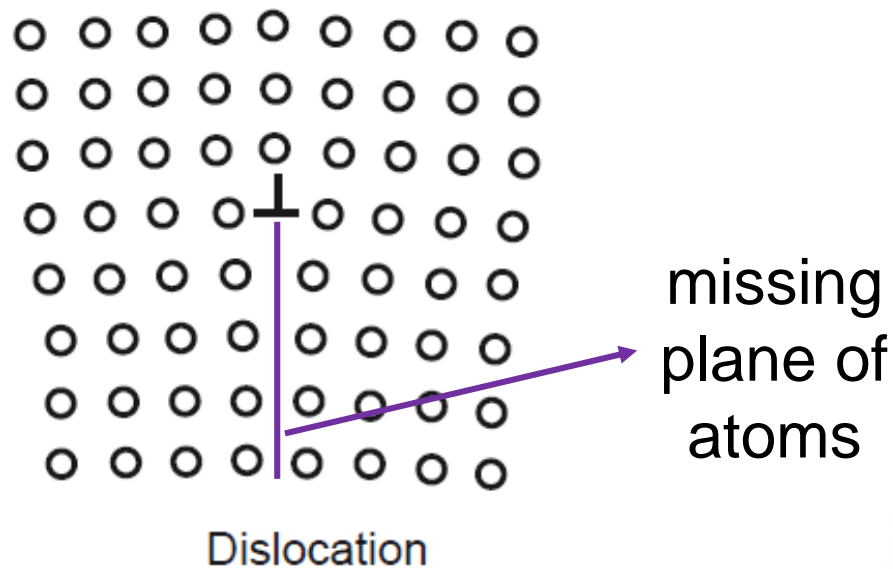


Microstructure and defects

Defects in Crystalline materials

How are defects classified?

- Point defects (0D)
- Line defects (1D)
 - Dislocations: missing plane of atoms



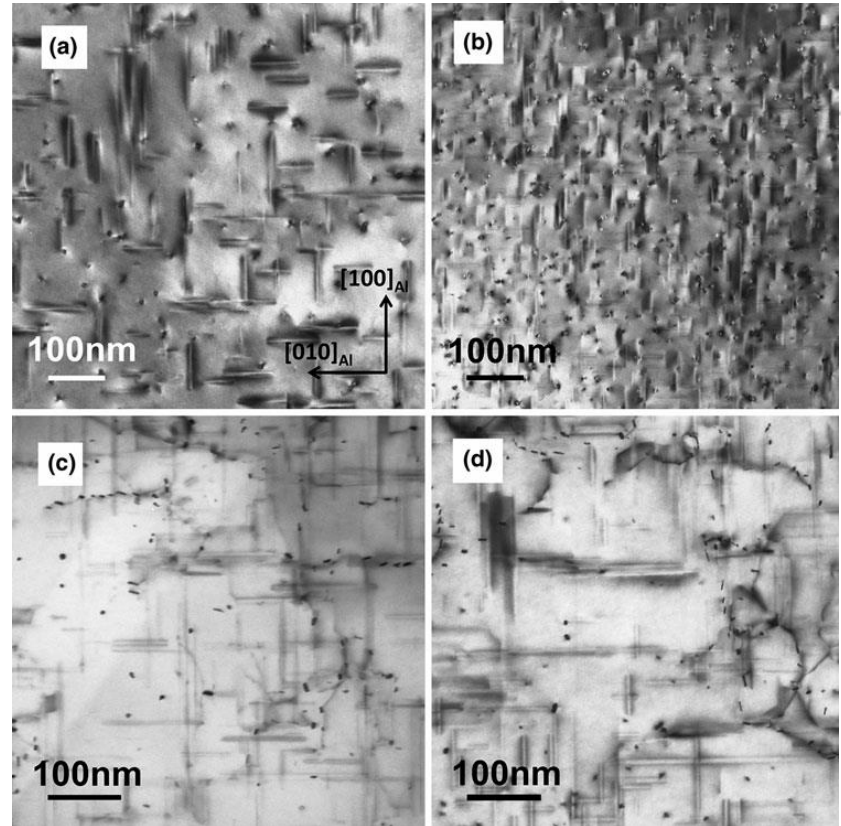
Microstructure and defects

Line defects (1D)

- Dislocations: missing plane of atoms

6060 Al-Mg-Si alloy

Department of Physics,
Norwegian University of
Science and Technology
(NTNU)



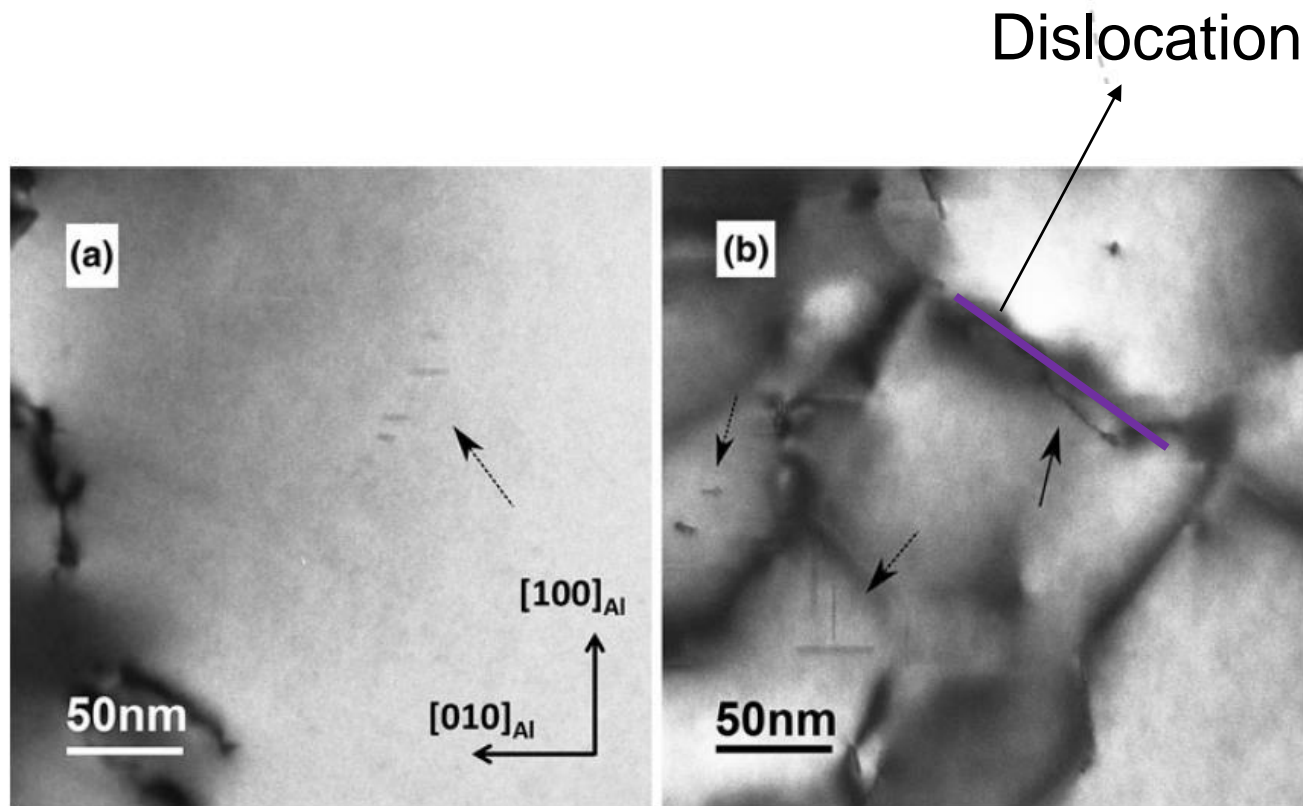


Fig. 5—Bright-field TEM images taken along $\langle 001 \rangle_{Al}$ directions for the alloy containing 0.10 wt pct Cu in (a) undeformed and (b) predeformed conditions, after an isothermal heat treatment at 463 K (190 °C) for 10 min. For a direct comparison, the images are recorded in areas with similar thicknesses (~ 110 nm). Crystallographic orientation for (b) is the same as that for (a). Some precipitate-types are indicated by arrows. Arrows with solid and dotted lines represent string-type and needle/lath-shaped precipitates, respectively. A large number of precipitates nucleated along dislocation lines can be observed in the predeformed condition.

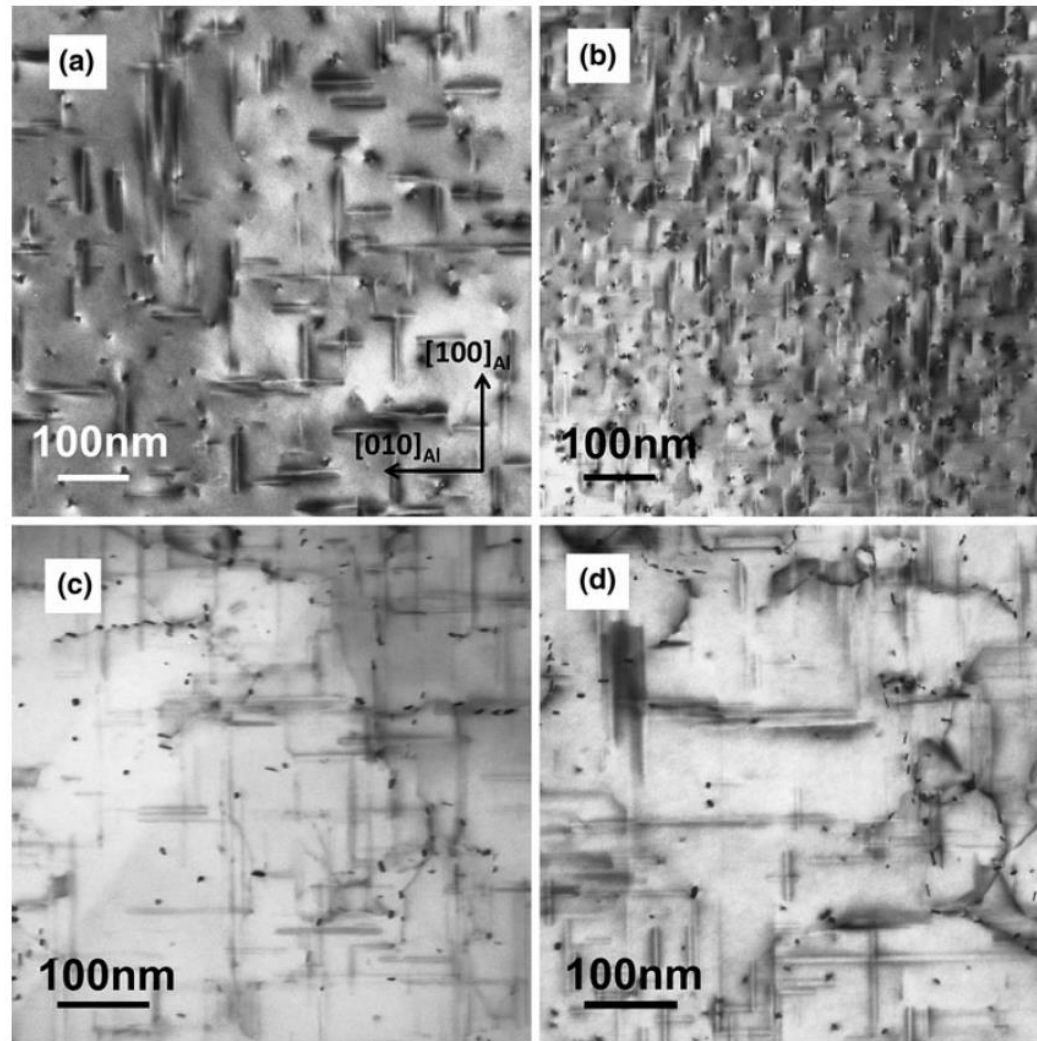


Fig. 6—Bright-field TEM images taken along $\langle 001 \rangle_{\text{Al}}$ directions for the alloys containing (a) 0.01 wt pct and (b) 0.10 wt pct Cu in undeformed conditions, and (c) 0.01 wt pct and (d) 0.10 wt pct Cu in 10 pct predeformed conditions, after an isothermal heat treatment at 463 K (190 °C) for 300 min, corresponding to Figs. 3 and 4 and Table II. For a direct comparison, the images are recorded in areas with similar thicknesses between 70 nm and 110 nm. Crystallographic orientation for (b) to (d) are the same as that for (a). The dark spots represent the cross sections of the needle-shaped precipitates in the viewing direction. The introduced dislocations in the predeformed conditions lead to coarser, inhomogeneous microstructures, while Cu addition produces a finer microstructure in the undeformed condition. See Table II for the microstructure quantification.

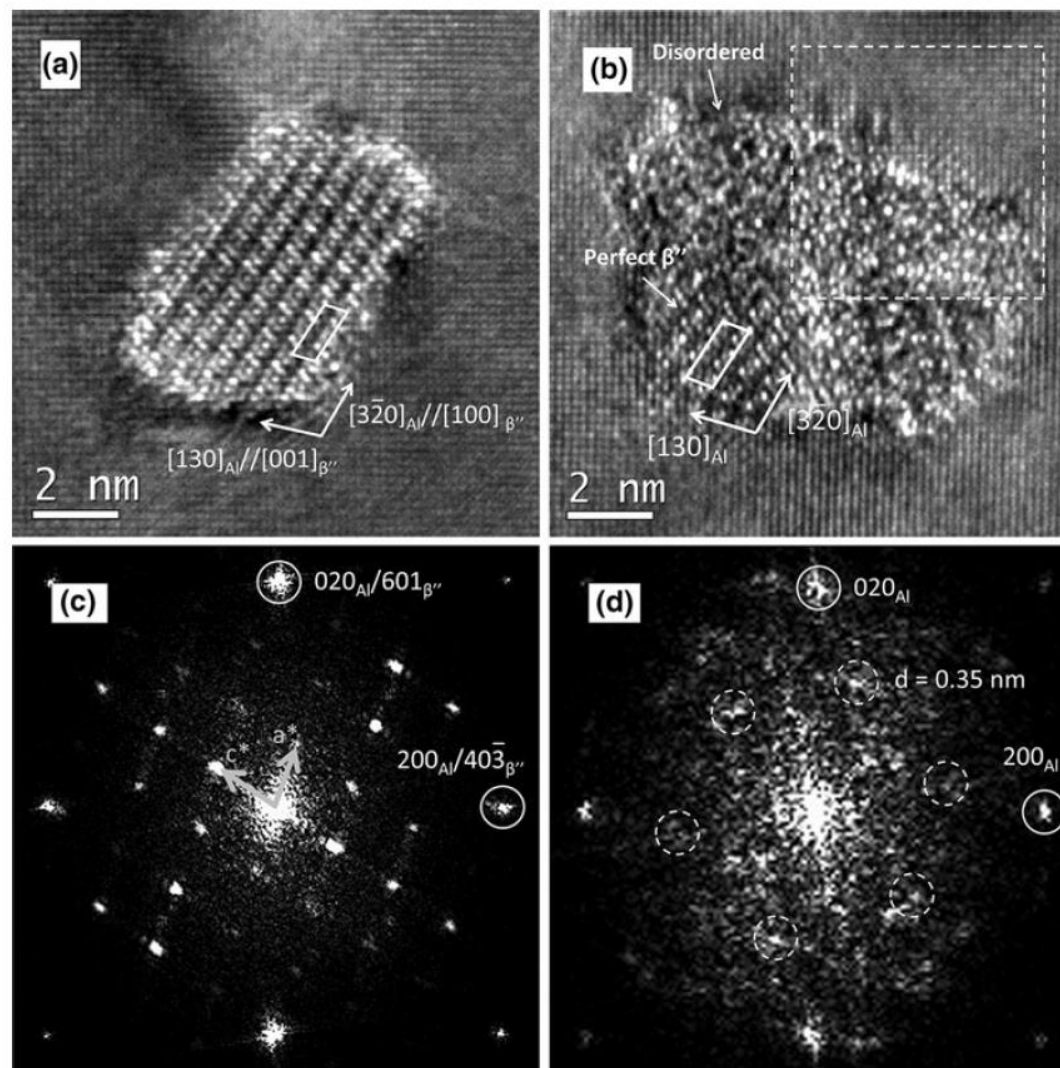


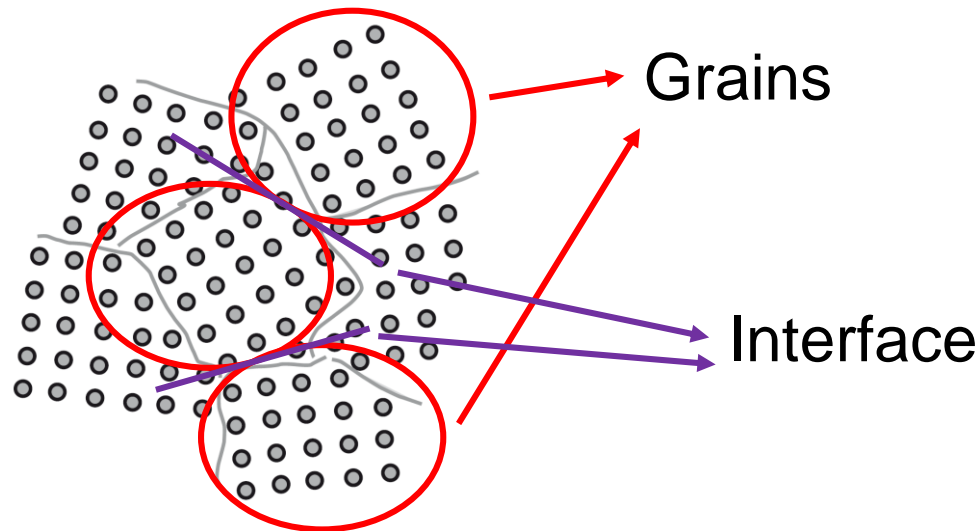
Fig. 7—HRTEM images of precipitate cross sections taken along $\langle 001 \rangle_{\text{Al}}$ for the alloy containing 0.10 wt pct Cu in the undeformed condition (b). (a) perfect β'' , (b) a combined β'' /disordered precipitate, (c) FFT of (a) and (d) FFT of disordered part of (b). A unit cell of the perfect β'' is shown by solid line in (a) and (b). The FFT of the disordered part was extracted from the area delimited by the dashed line square in (b). The d-spacing of 0.35 nm is the spatial period related to spots inside dashed circles in (d), and corresponds to a projected hexagonal periodicity of ~ 0.4 nm in real space, typical for the Si-network. It therefore indicates the presence of the network in the disordered part, where no unit cell could be identified in the HRTEM image (b).

Microstructure and defects

Defects in Crystalline materials

How are defects classified?

- Point defects (0D)
- Line defects (1D)
- Surface defects (2D)
 - Grain boundaries, twins and stacking faults



Grain boundary
(GB): the interface between two grains, or crystallites, in a polycrystalline material

Grain boundaries

Microstructure and defects

- Surface defects (2D)
 - Grain boundaries

BaZrO₃ ceramics

Our group's work



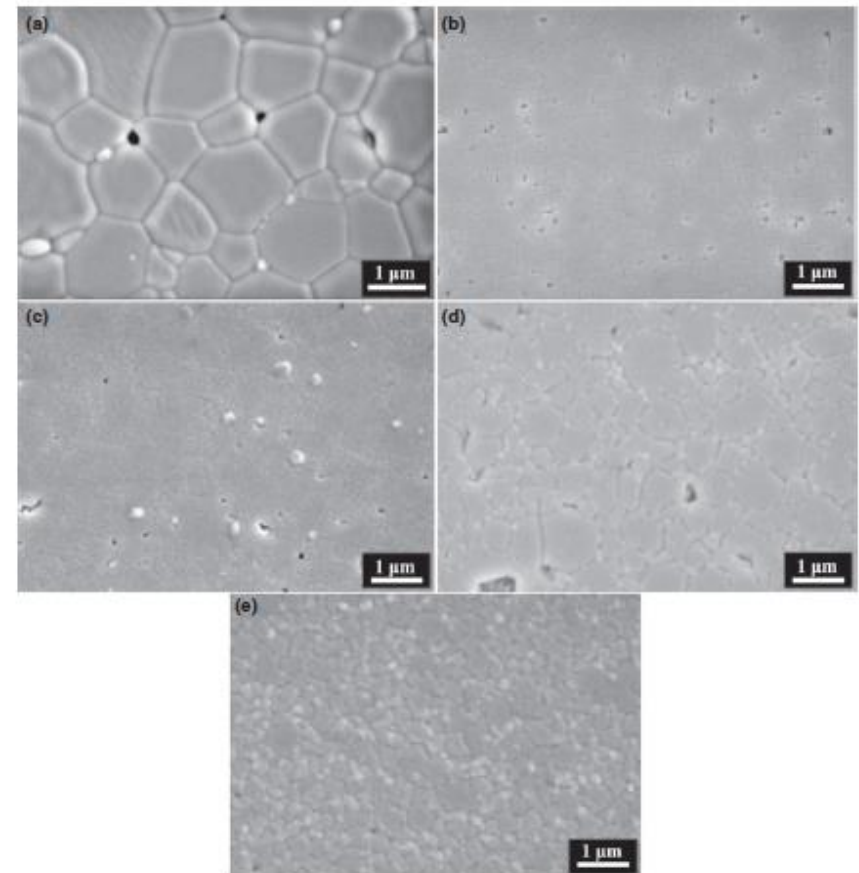
Journal

Effect of CO₂ Exposure on the Chemical Stability and Mechanical Properties of BaZrO₃-Ceramics

J. Am. Ceram. Soc., 1–11 (2016)
DOI: 10.1111/jace.14395
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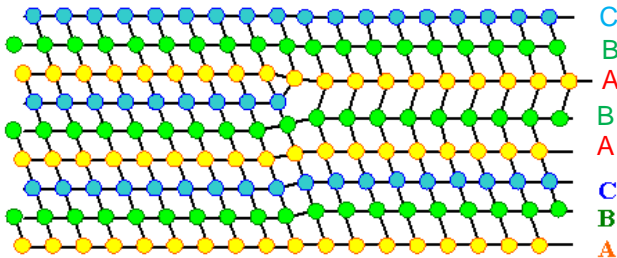
<http://onlinelibrary.wiley.com/doi/10.1111/jace.14395/abstract>

Microstructure and defects

Defects in Crystalline materials

How are defects classified?

- Point defects (0D)
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Stacking faults

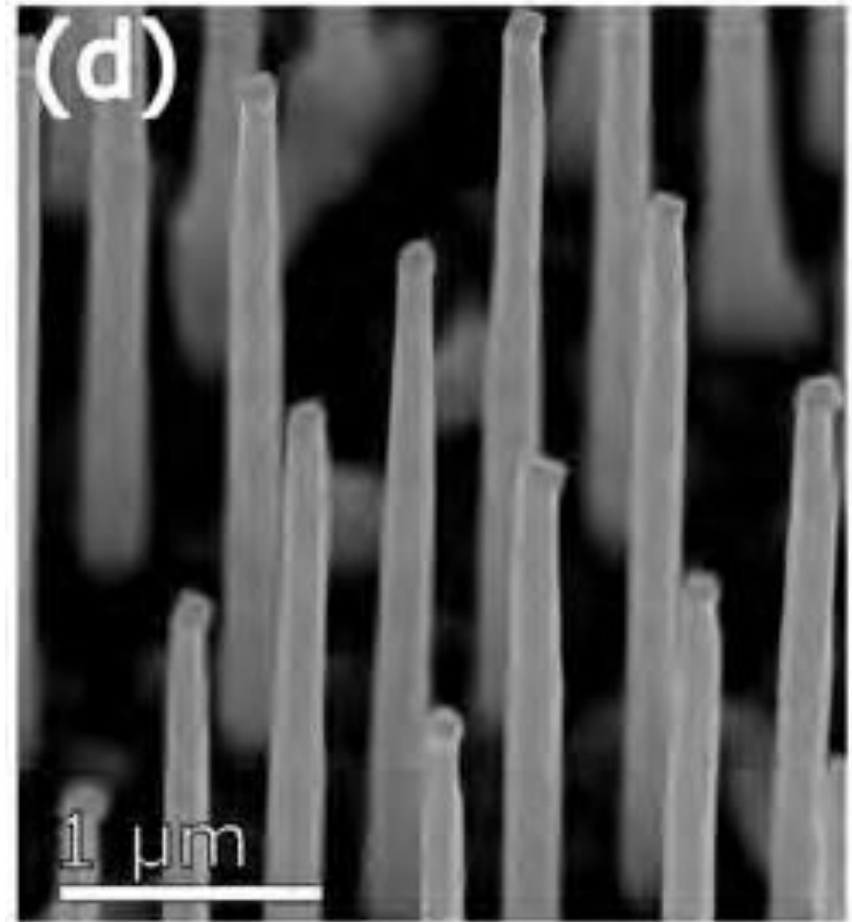
Stacking fault:
Incorrect stacking
of crystal planes
associated with
the presence of
partial dislocations

Microstructure and defects

- Surface defects (2D)
 - Stacking faults

GaAs/AlGaAs core-shell nanowires

As well in problem B

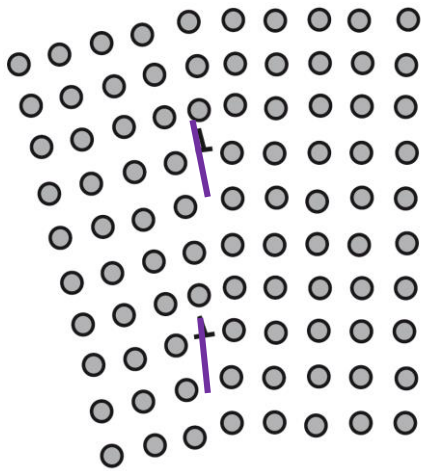


Microstructure and defects

Defects in Crystalline materials

How are defects classified?

- Point defects (0D)
- Line defects (1D)
- Surface defects (2D)
 - Grain boundaries, twins and stacking faults



Twin boundary: A
slight
misorientation that
exists between
two crystals

Twin boundary

Microstructure and defects

Surface defects (2D)

- Twin boundary

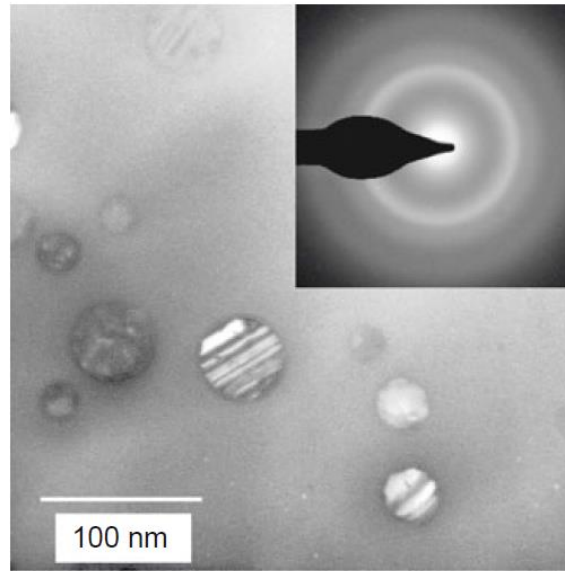
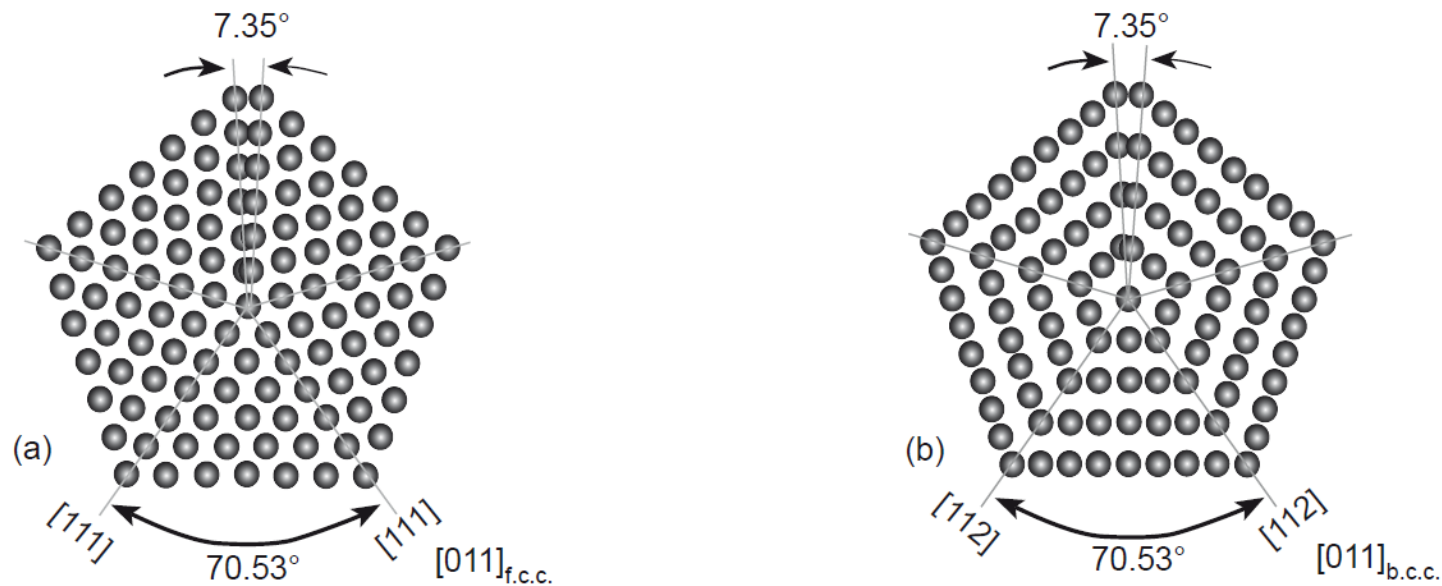


Fig. 2.1 Twins in nanocrystalline Cu particles embedded in amorphous Fe-Zr-B alloy. The twins formation is possibly due to strains developed at the amorphous Cu interface during solidification.
(Source: BS Murty, IIT Madras).

Microstructure and defects

Surface defects (2D)

- Twin boundary: multiply twinned nanoparticles

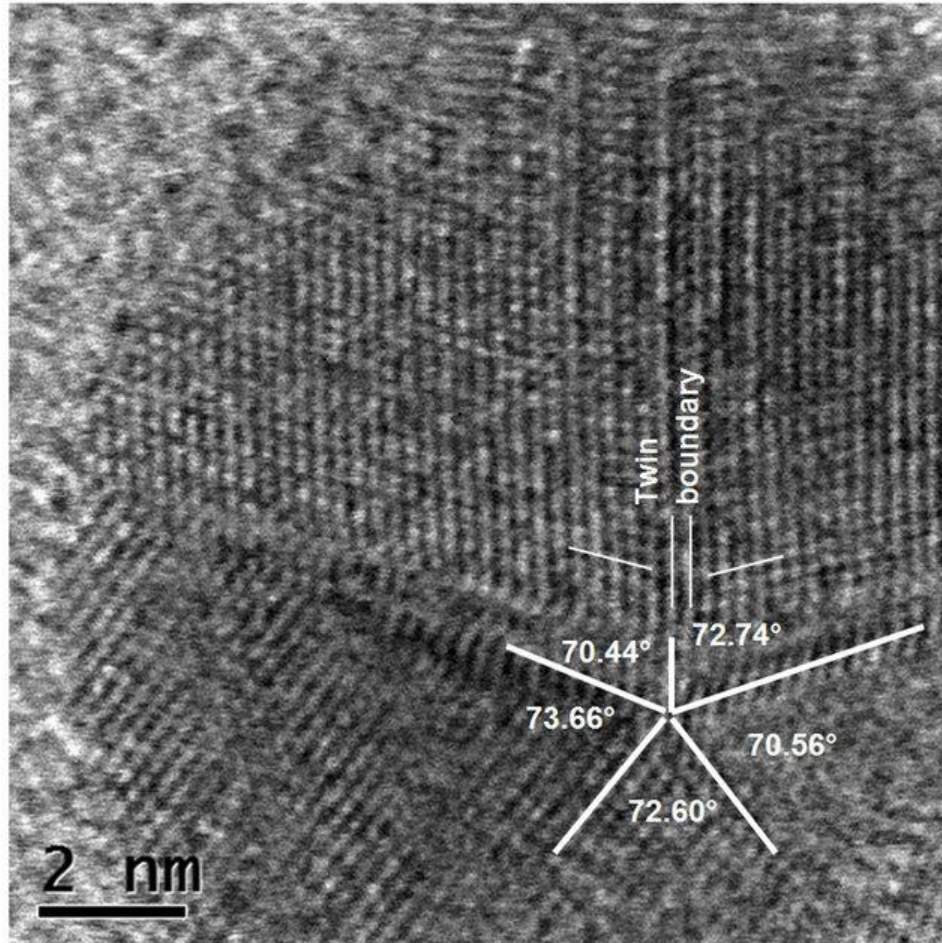


Schematic of five-fold twinned cubic lattice showing the annular mismatch created by repetitive twinning.

Microstructure and defects

Surface defects (2D)

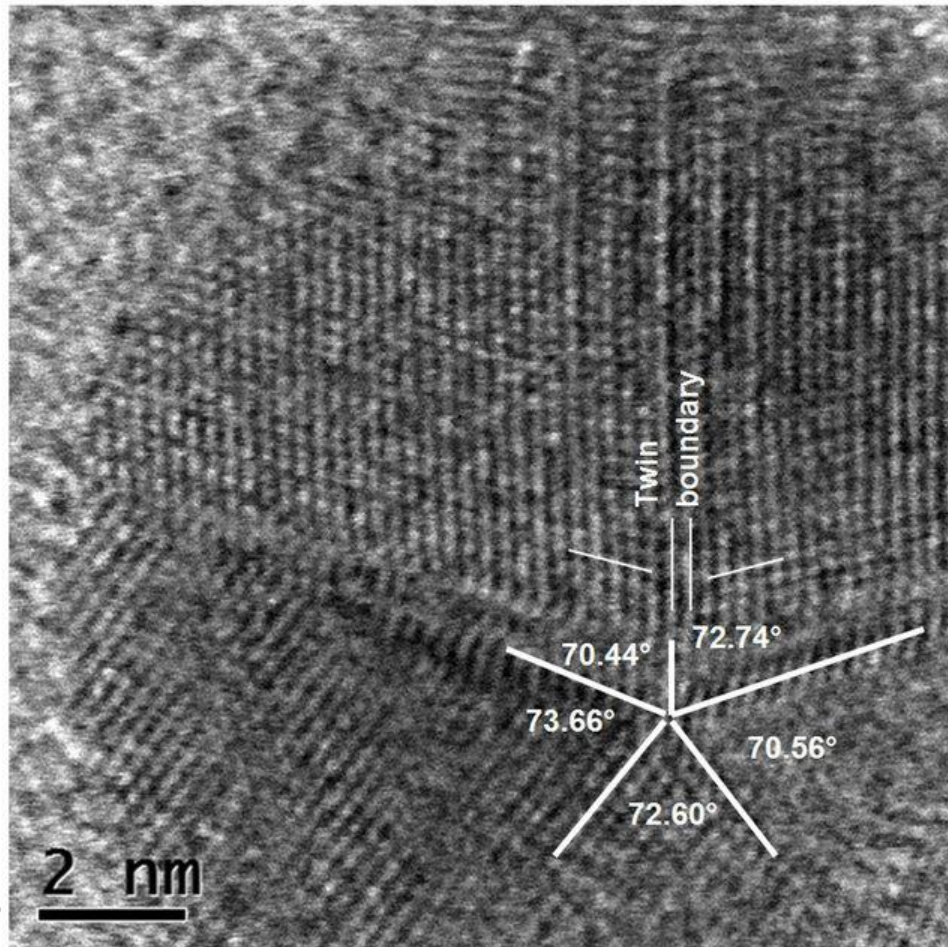
- Twin boundary: multiply twinned nanoparticles



Microstructure and defects

Surface defects (2D)

- Twin boundary: multiply twinned nanoparticles



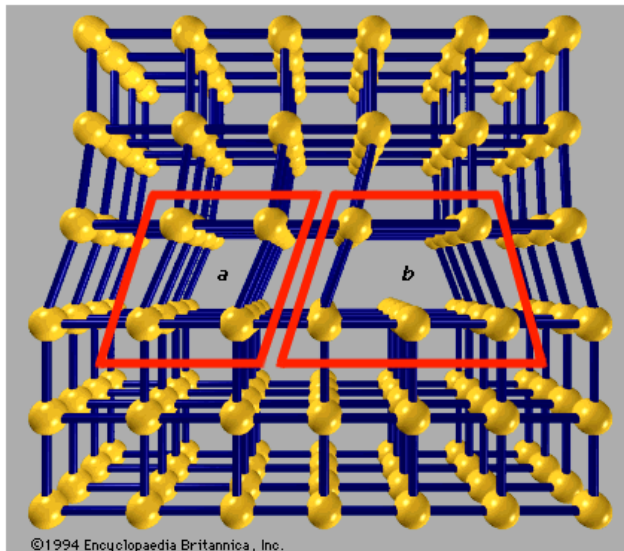
HR STEM image
of the center of the
Au nanoparticle

Microstructure and defects

Defects in Crystalline materials

How are defects classified?

- Point defects (0D)
- Line defects (1D)
- Surface defects (2D)
- Volume defects (3D)
 - Voids and microcracks.

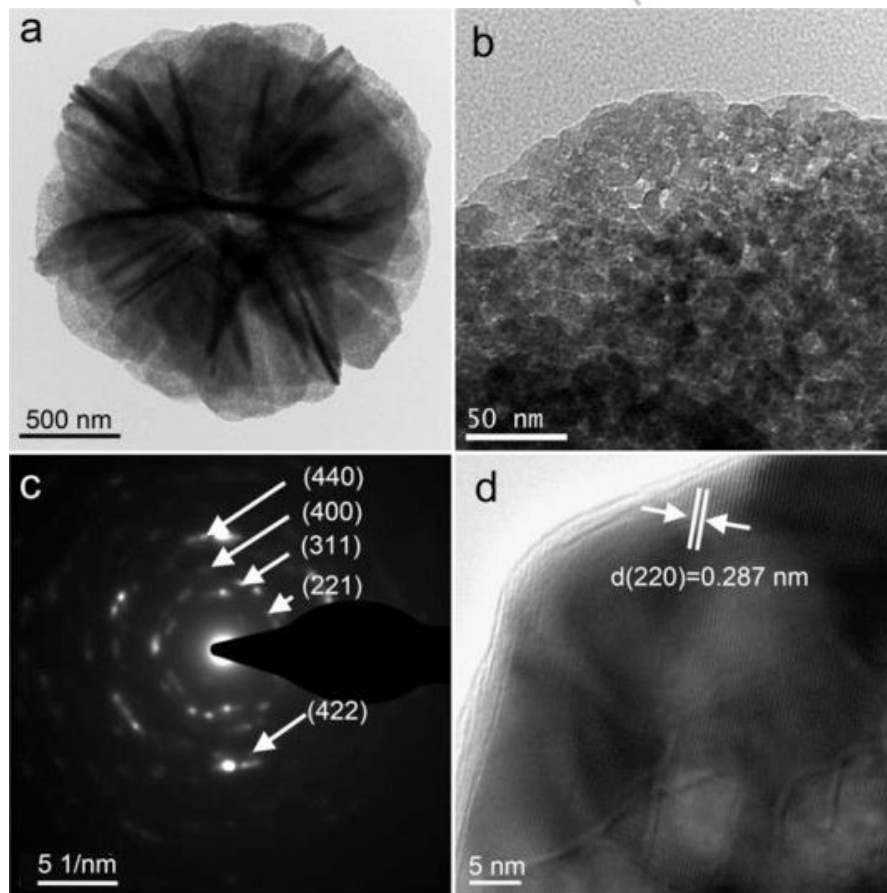


Void: The absence of a number of atoms to form internal surfaces in the crystal

Microstructure and defects

- Volume defects (3D)
 - Voids

Rose flower-like NiCo_2O_4



Microstructure and defects

- Volume defects (3D)
 - Voids

Rose flower-like NiCo_2O_4

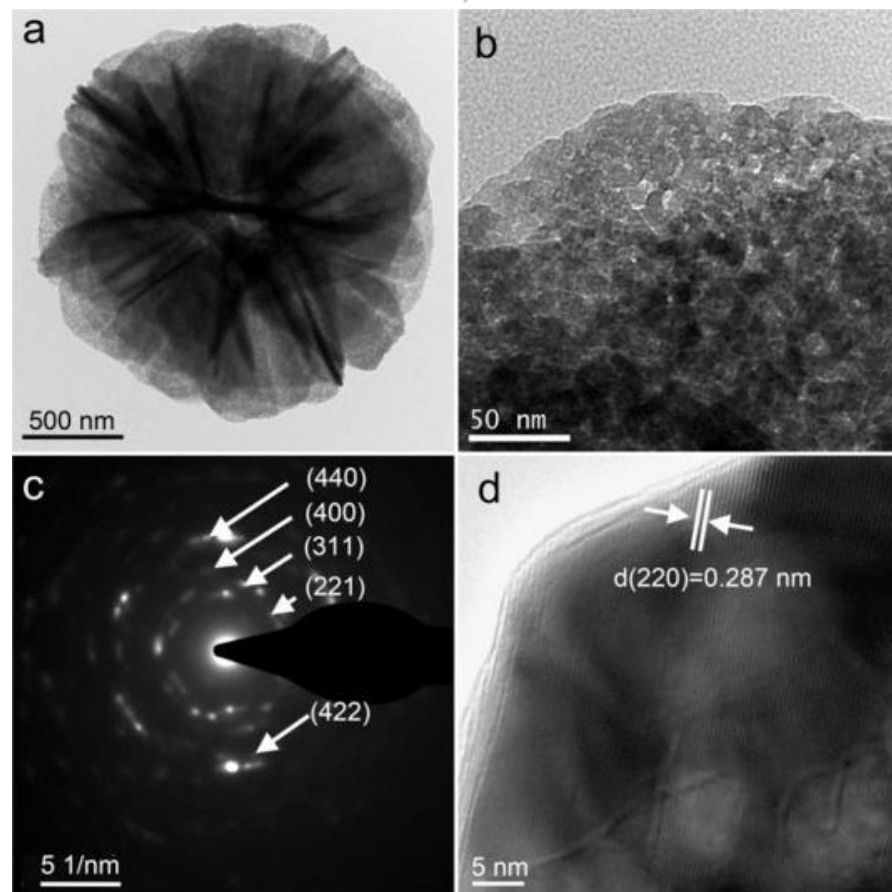


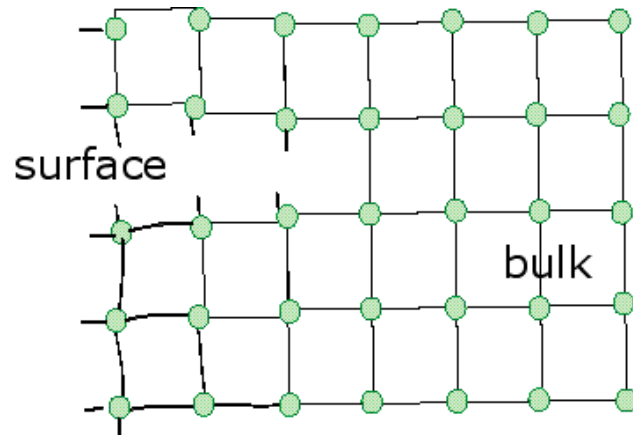
Fig. 3. (a and b) TEM images for the hierarchical porous flower-like NiCo_2O_4 . (c) Corresponding SAED pattern. (d) HRTEM image for NiCo_2O_4

Microstructure and defects

Defects in Crystalline materials

How are defects classified?

- Point defects (0D)
- Line defects (1D)
- Surface defects (2D)
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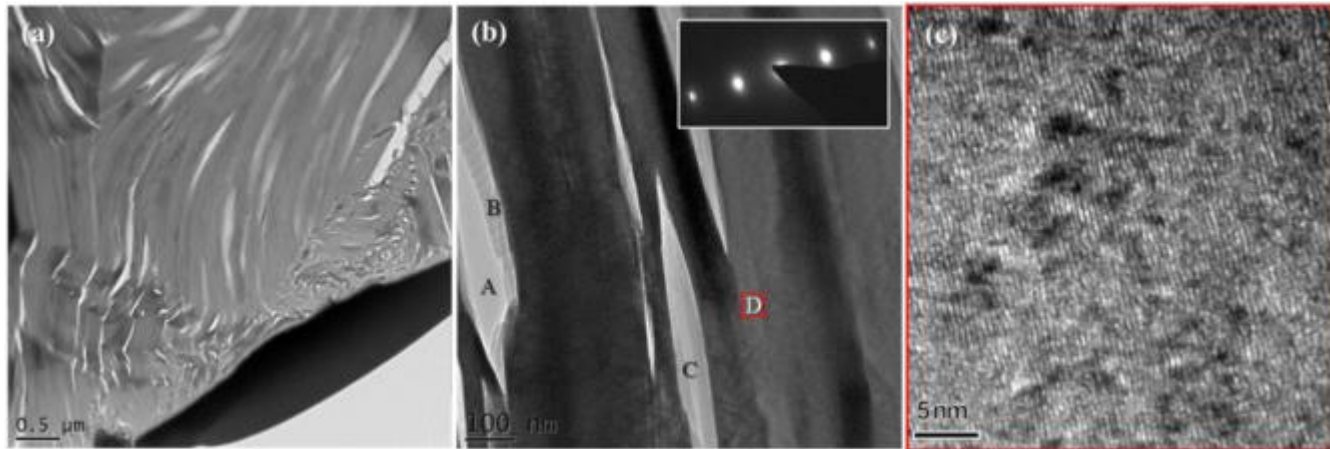
A **Microcrack** occurs where internal broken bonds create new surfaces.

Microstructure and defects

Volume defects (3D)

- Microcracks.

Graphite



Microstructure and defects

Volume defects (3D)

- Microcracks.

Graphite

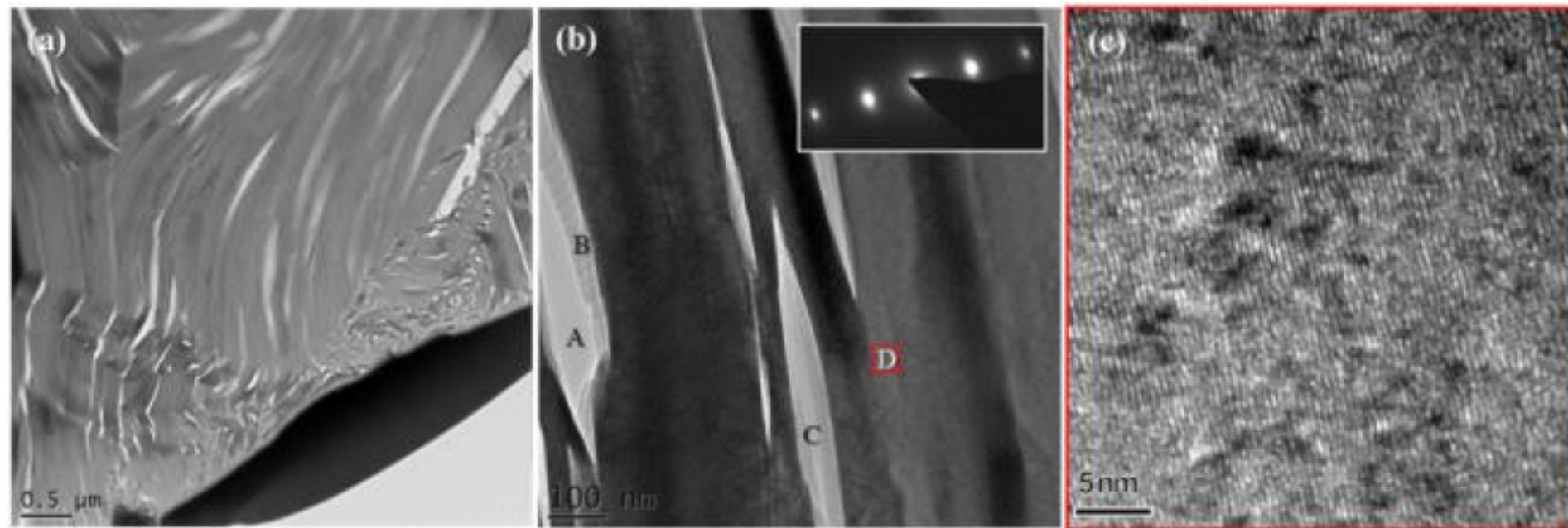


Fig. 3. (a) relatively low magnification and (b) medium magnification TEM images of intra-granular microcracks within the FIB section of specimen BEPO20. Labels in (b): A - no material within the crack; B - material at the crack interface; C - a completely filled crack; D - graphitic bulk material. Electron diffraction pattern of the area shown in micrograph (b) is presented in the top right corner. (c) shows a high magnification TEM image of region D to verify the basal plane orientation is parallel to cracks. (A colour version of this figure can be viewed online.)

Microstructure and defects

Problem B

Complex layered oxides materials are used as electrode materials in energy storage and conversion applications such as batteries and fuel cells due to their capacity to adopt various defects. In the following material, identify the defects (point defects (0D), line defects (1D), surface defects (2D) and volume defects (3D)) that this materials may present looking at the data that you have.

Complex layered oxide as anode for solid oxide cell

What is a solid oxide cell?

What temperature operates?

What can we obtain?

Complex layered oxide as anode for solid oxide cell

Intergrowth between $\text{Sr}_2\text{MgMoO}_{6-\delta}$ and $\text{Sr}_3\text{Mo}_2\text{O}_{7-\delta}$

What is an intergrowth?

What structure is $\text{Sr}_2\text{MgMoO}_{6-\delta}$? And $\text{Sr}_3\text{Mo}_2\text{O}_{7-\delta}$?

What does « δ » mean?

Complex layered oxide as anode for solid oxide cell

Intergrowth between $\text{Sr}_2\text{MgMoO}_{6-\delta}$ and $\text{Sr}_3\text{Mo}_2\text{O}_{7-\delta}$

What is an intergrowth?

The growing of one thing with or into another.

$\text{Sr}_2\text{MgMoO}_{6-\delta}$ in $\text{Sr}_3\text{Mo}_2\text{O}_{7-\delta}$ or viceversa

What structure is $\text{Sr}_2\text{MgMoO}_{6-\delta}$? And $\text{Sr}_3\text{Mo}_2\text{O}_{7-\delta}$?

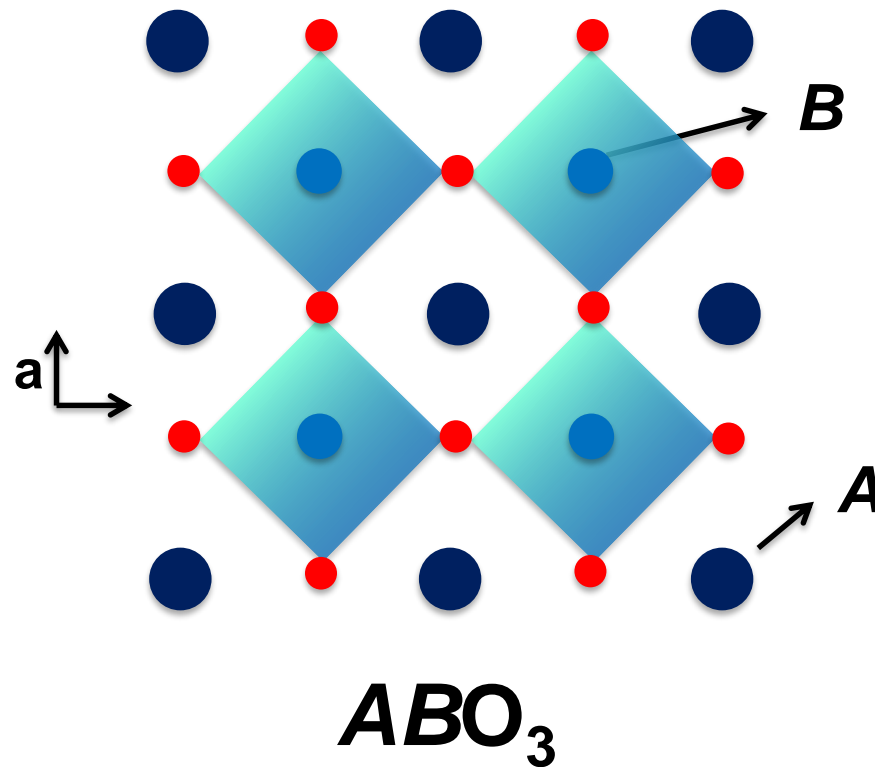
$\text{Sr}_2\text{MgMoO}_{6-\delta}$: Perovskite → Doble perovskite

$\text{Sr}_3\text{Mo}_2\text{O}_{7-\delta}$: Ruddlesden Popper $n=2$.

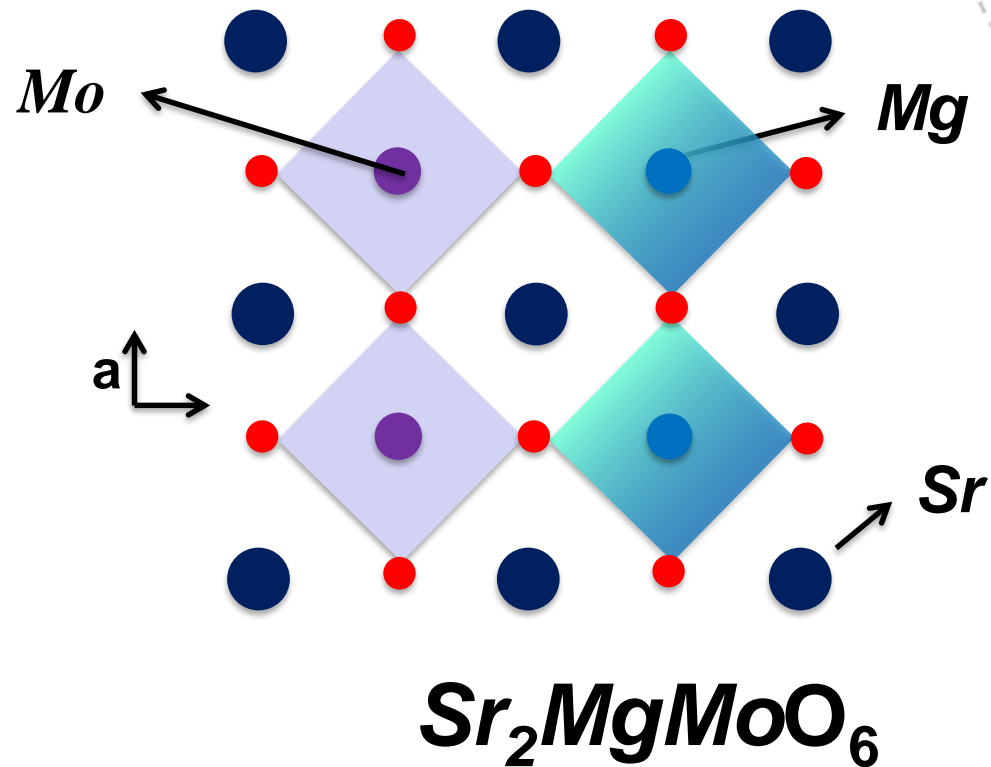
What does « δ »?

Oxygen vacancies

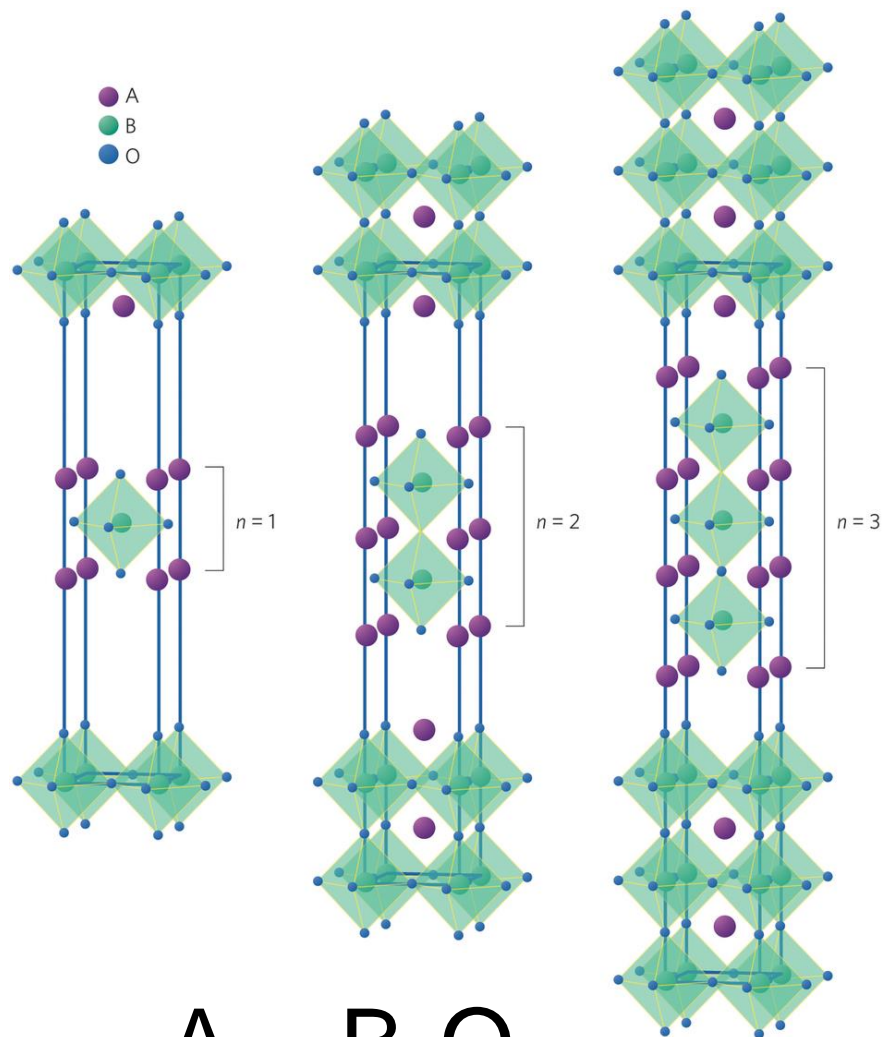
Perovskite



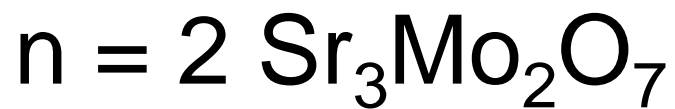
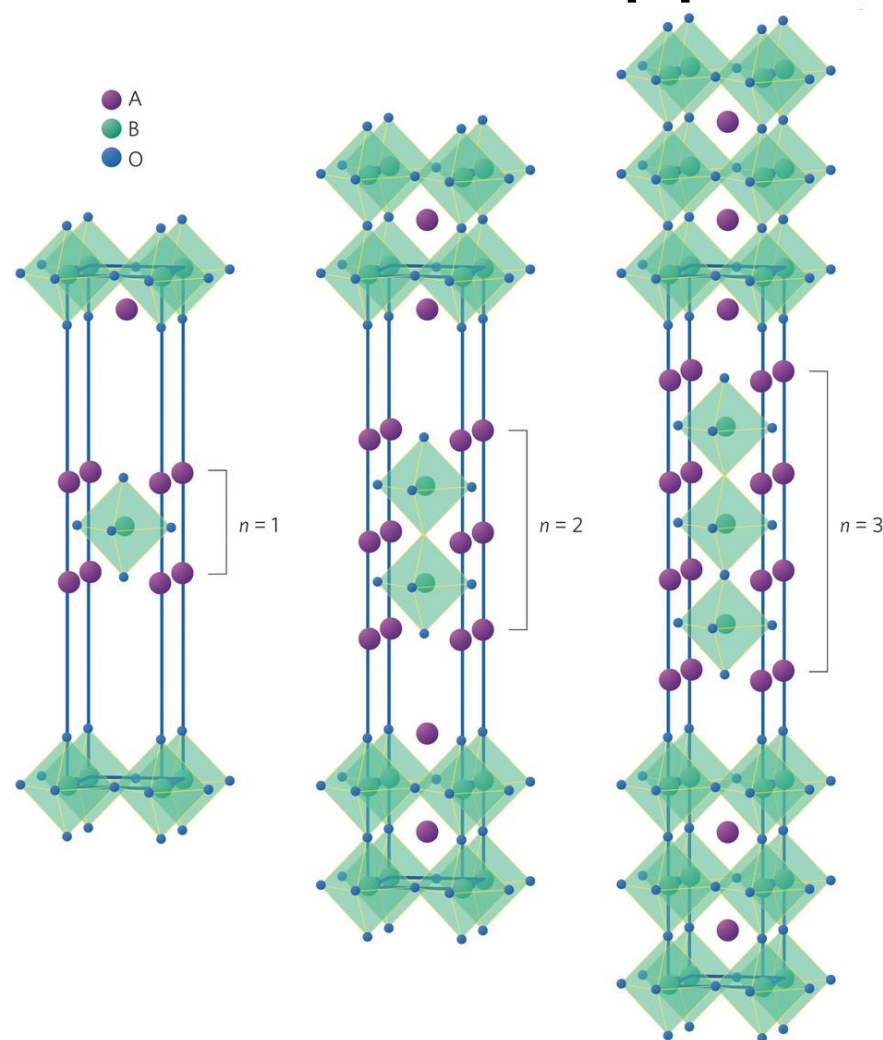
Double Perovskite



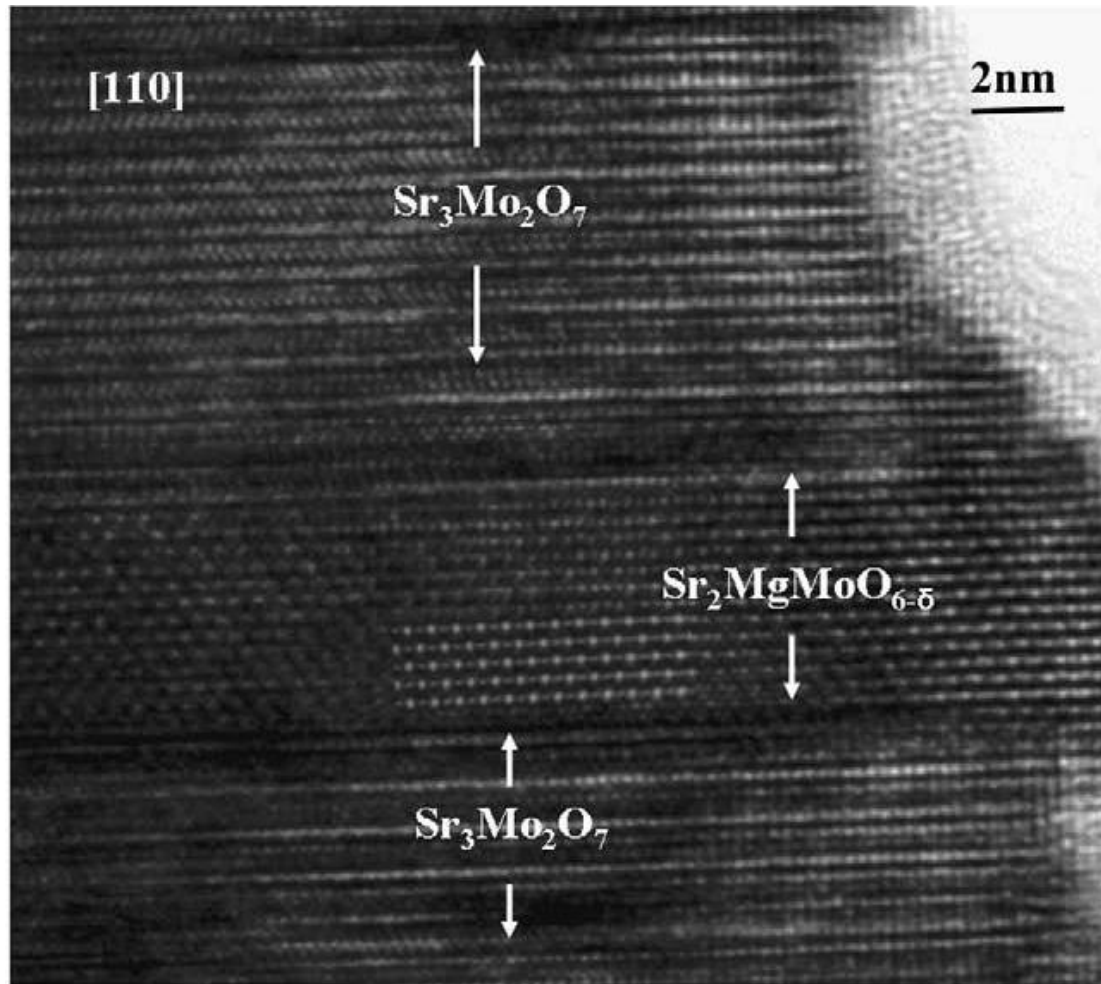
Ruddlesden-Popper



Ruddlesden-Popper

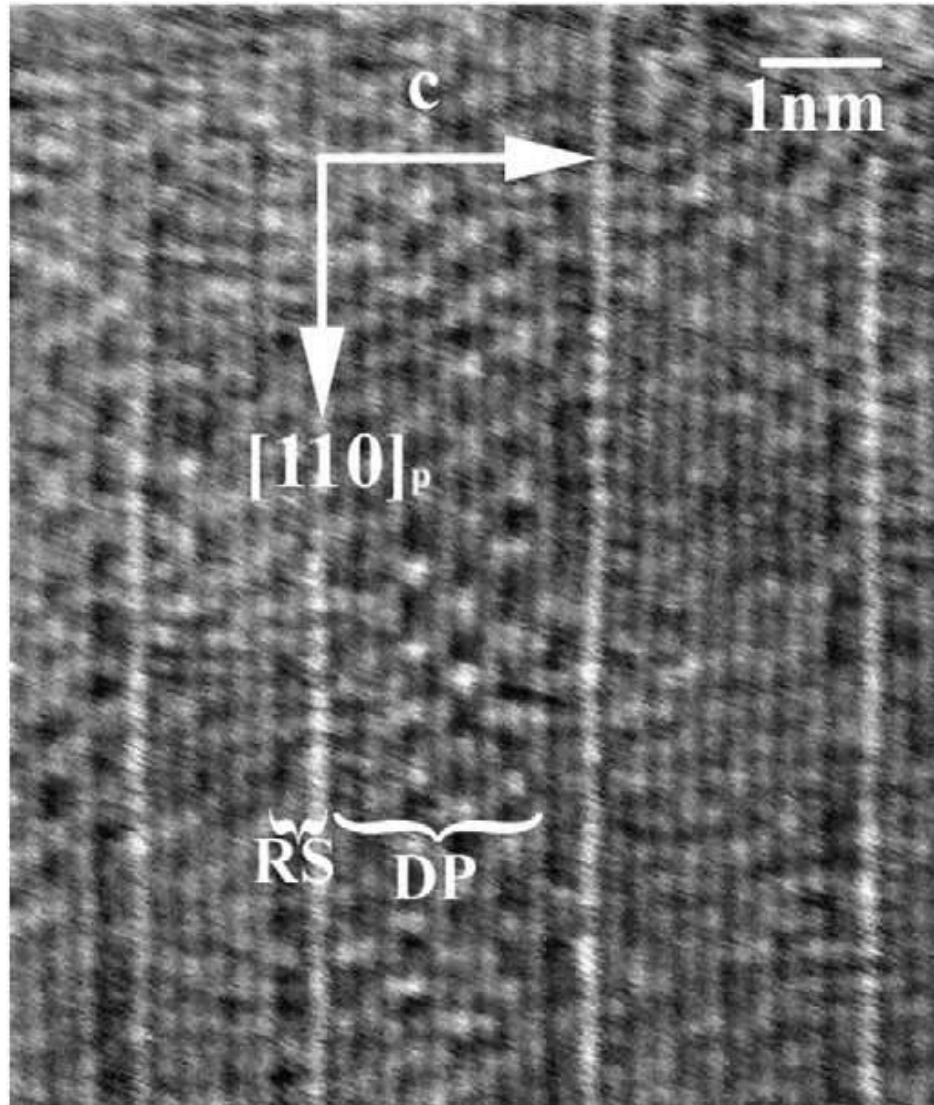


Complex layered oxide $\text{Sr}_3(\text{Mg}_x\text{Mo}_{1-x})_2\text{O}_{7-\delta}$ ($x \approx 0.25$) as anode for solid oxide cell

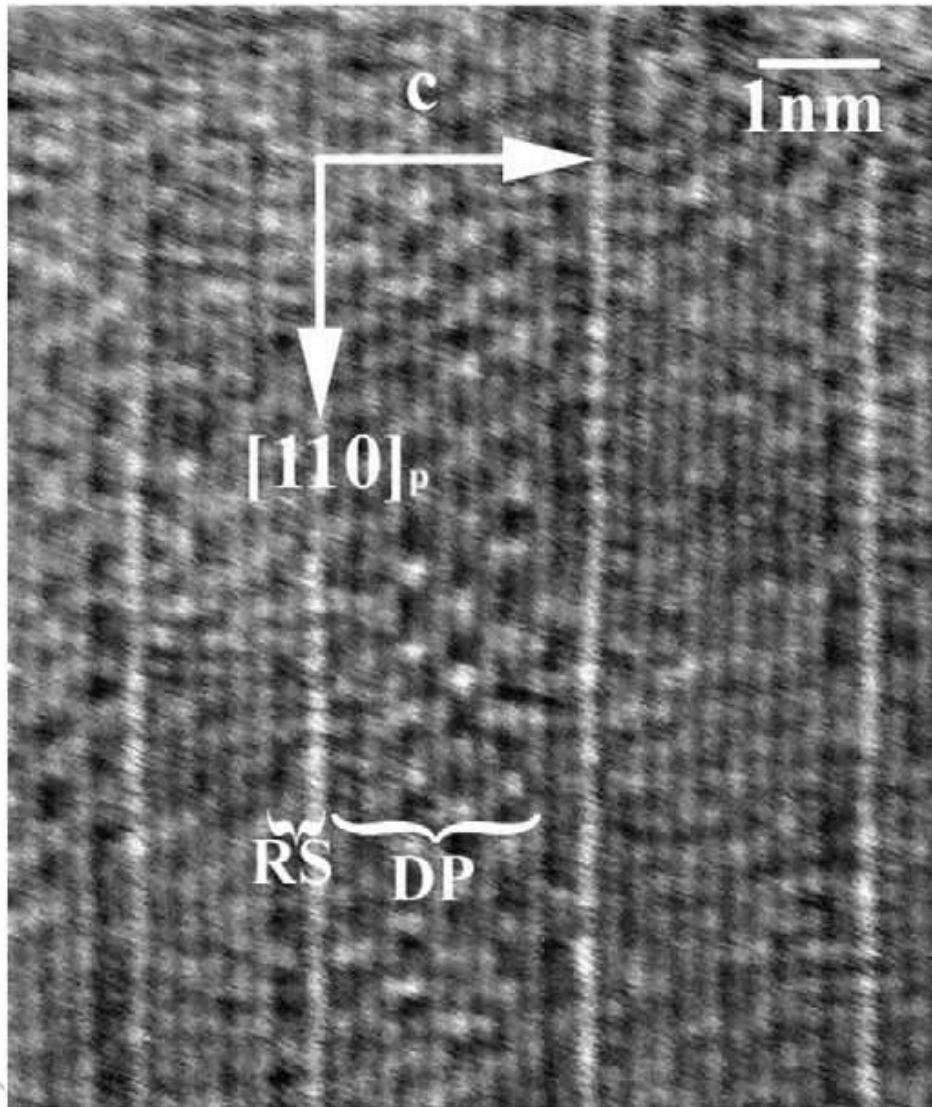


Intergrowth

Complex layered oxide $\text{Sr}_3(\text{Mg}_x\text{Mo}_{1-x})_2\text{O}_{7-\delta}$ ($x \approx 0.25$) as anode for solid oxide cell

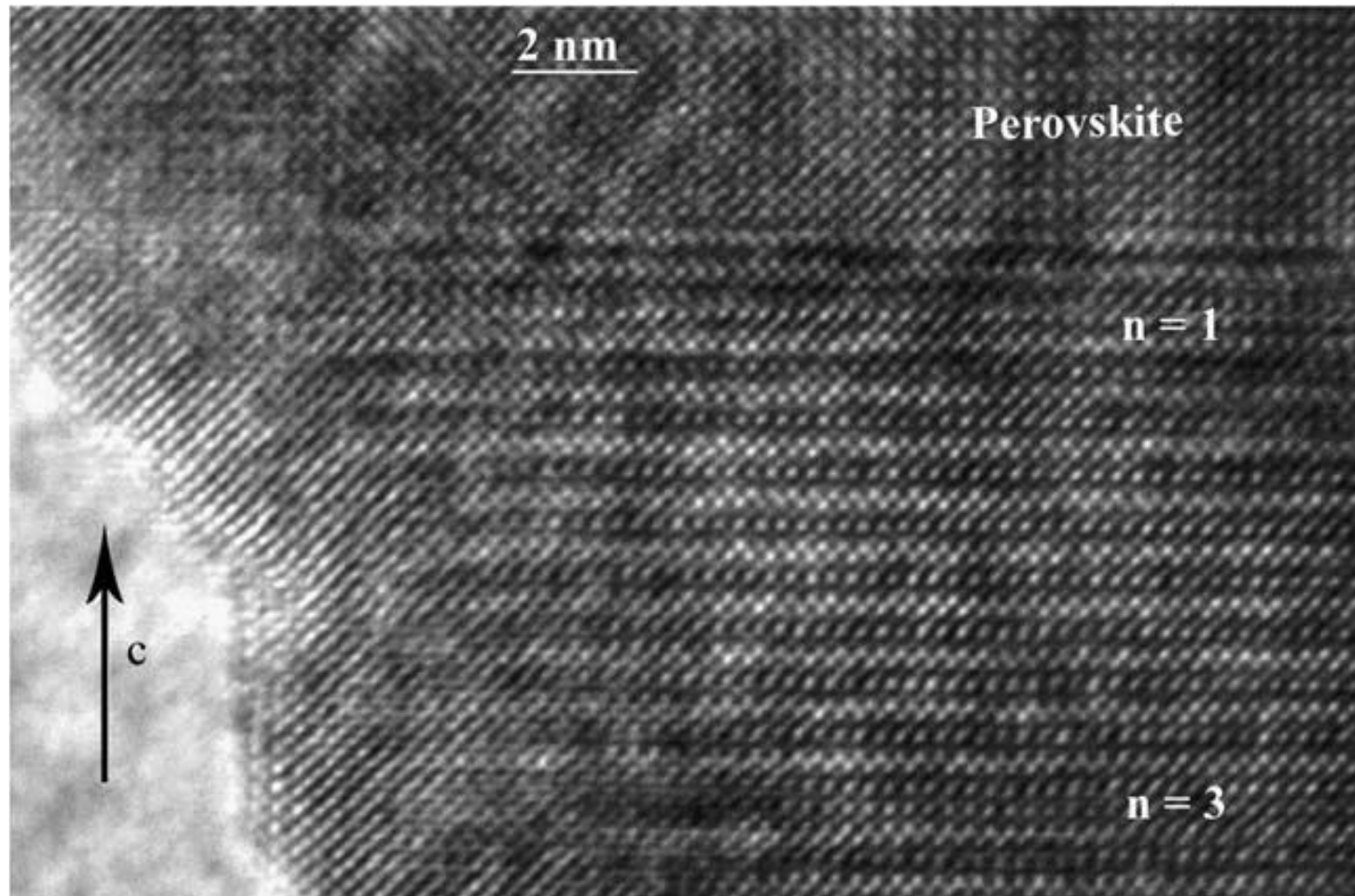


Complex layered oxide $\text{Sr}_3(\text{Mg}_x\text{Mo}_{1-x})_2\text{O}_{7-\delta}$ ($x \approx 0.25$) as anode for solid oxide cell

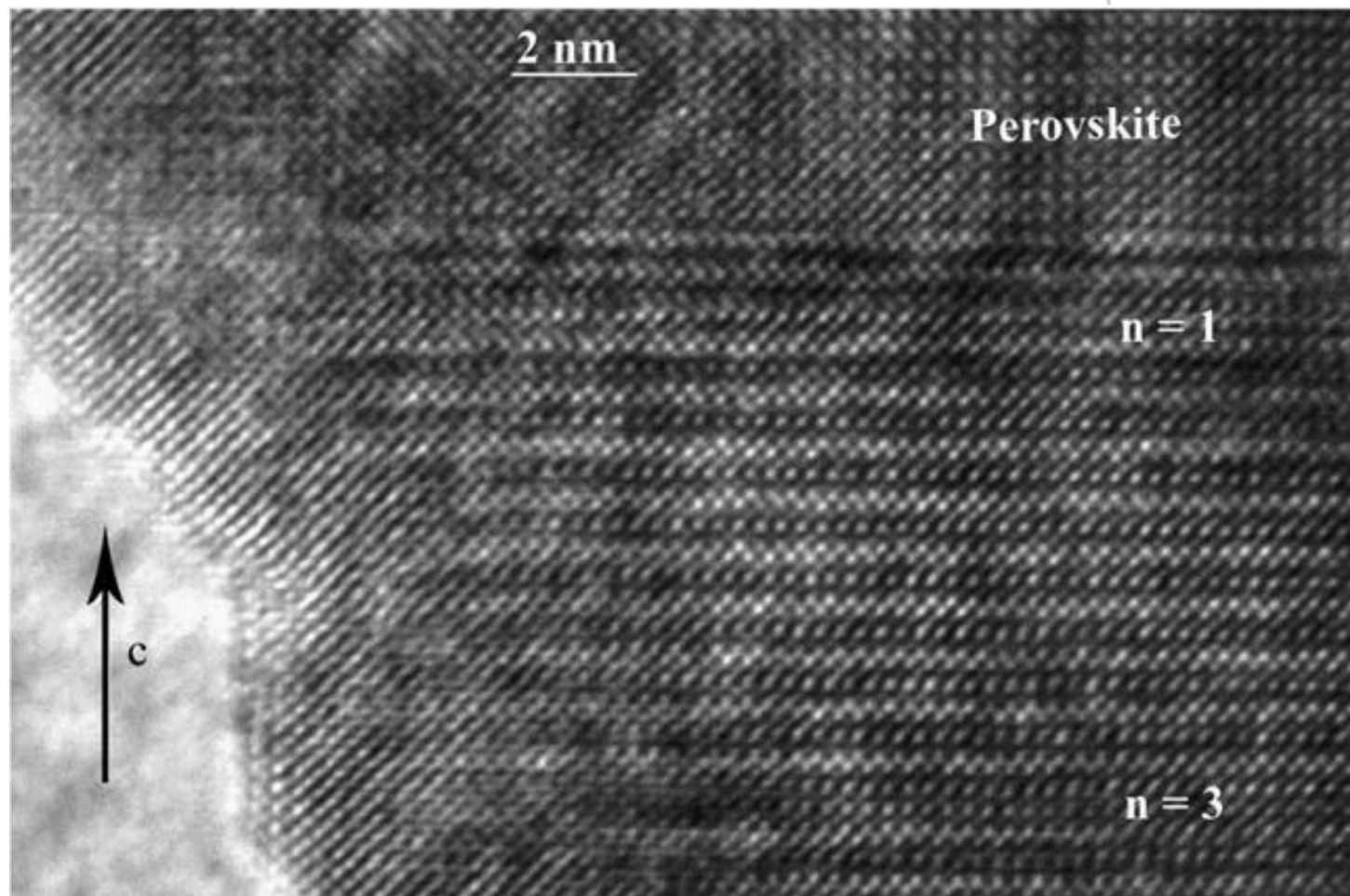


Point defects

Complex layered oxide $\text{Sr}_3(\text{Mg}_x\text{Mo}_{1-x})_2\text{O}_{7-\delta}$ ($x \approx 0.25$) as anode for solid oxide cell



Complex layered oxide $\text{Sr}_3(\text{Mg}_x\text{Mo}_{1-x})_2\text{O}_{7-\delta}$ ($x \approx 0.25$) as anode for solid oxide cell



Stacking faults

Today lecture

- Defects
 - Examples
 - Problem B
- Physical properties: size effects
 - Lattice constant
 - Melting point

Physical properties of Nanomaterials

Properties differ a lot between dimensions on an atomic scale and the dimensions which characterize bulk materials

- 1) Lower melting point or phase transition temperature and reduce lattice parameters due to a huge fraction of surface atoms in the total amount of atoms (More in deep today)
- 2) Mechanical properties may reach the theoretical strength (2 times more than single crystal) due to the reduce probability of defects.

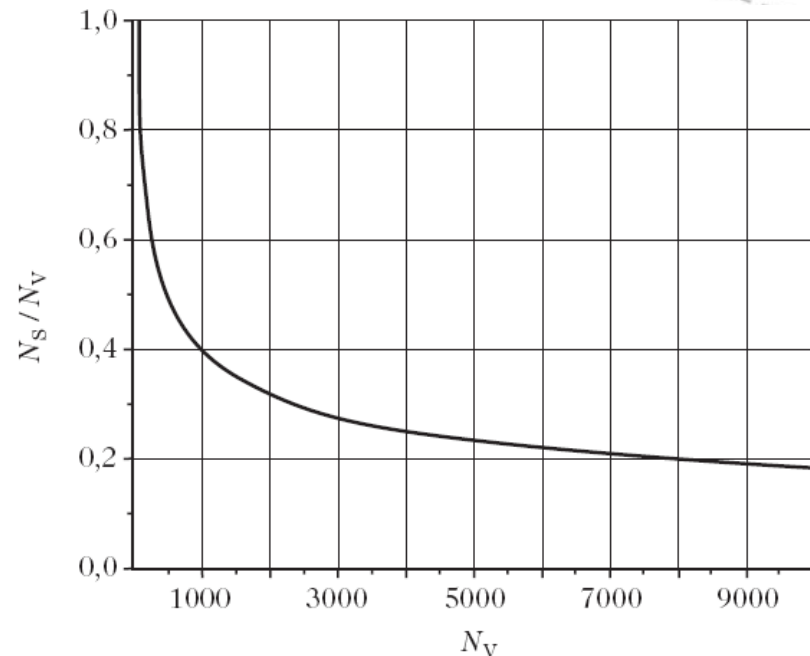
Physical properties of Nanomaterials

Properties differ a lot between dimensions on an atomic scale and the dimensions which characterize bulk materials

- 3) Optical properties. Optical absorption peak of a semiconductor nanoparticle shifts to a short wavelength due to an increased bandgap. Color of metallic nanoparticles may change with size due to surface plasmon resonance.
- 4) Electrical conductivity decreases with reduced dimension due to increased surface scattering. However, it could be enhanced due to better ordering in microstructure.
- 5) Magnetic properties. Ferromagnetism of bulk materials disappears and transfers to supermagnetism in the nanometer scale due to huge surface energy.

Fraction of surface atoms

- Surface atoms are important for chemical reactivity (catalysis) and crystal growth
- Nanoparticles
 - 5 nm: around 8000 atoms
 - 2 nm: around 500 atoms
- Surface/volume ratio



Proportion of surface atoms for a spherical particle comprising N_v atoms with N_s at the surface

Specific surface energy

- γ (J/m²)
- The energy produced by cleaving a crystal divided by the surface area thereby created
- In order to increase the surface area of an object by an amount dA , e.g., by changing the shape of the object, the work required to do this will be:

$$dW = \gamma dA$$

- Also expressed as:

$$\gamma = \left(\frac{\partial G}{\partial A} \right)_{n,T,P} \quad \text{or} \quad \gamma = N_b \left(\frac{1}{2} \varepsilon \right) = \frac{1}{2} n_a \cdot n_b \varepsilon$$

N_b = number of bonds broken per unit area
 n_a = number of surface atoms per unit area

ε = bond energy
 n_b = number of bonds broken per atom

Surface stress

- g_{ij} (J/m²)
- If one increase the area by stretching it, i.e., keeps the number of surface atoms constant, the work required to do this will be:

$$dW = g_{ij}dA$$

- The surface stress is related to the elastic stresses resulting from deformation of the surface (strain)

Surface curvature

- Surface energies given in handbooks are for flat surfaces
- Surface energy and hence chemical potential (μ) of the surface atoms change with curvature

$$\Delta\mu = \mu_c - \mu_\infty = 2\gamma \frac{\Omega}{r}$$

where μ_c = chemical potential of curved surface

μ_∞ = chemical potential of flat surface

Ω = atomic volume

r = radius of curvature (> 0 for convex surface, < 0 for concave surface, ∞ for flat surface)

- $\mu_{convex} > \mu_{flat} > \mu_{concave}$

Effects on the melting point

Decrease in bonding energy due to enhance surface and grain boundary area in nanocrystalline materials can reduce the enthalpy of fusion and the melting temperature

Melting point → Solid and liquid in equilibrium


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Thermodynamically: $\mu_i^l = \mu_i^s$

Chemical
potential



Effects on the melting point

Decrease in bonding energy due to enhance surface and grain boundary area in nanocrystalline materials can reduce the enthalpy of fusion and the melting temperature

Melting point → Solid and liquid in equilibrium →

Thermodynamically: $\mu_i^l = \mu_i^s$

Chemical
potential

Due to inherent internal pressure: $P_s > P_l$

Surface
energy

$$P_s = P_l + \frac{2\gamma}{r}$$

radius

Effects on the melting point

Chemical potential related with pressure: $d\mu = -SdT + VdP$

Effects on the melting point

Chemical potential related with pressure: $d\mu = -SdT + VdP$

$$\mu_i^l = \mu_i^s$$

Chemical
potential

$$S_l - S_s = \Delta S_m$$

Entropy at
melting point

$$\Delta S_m dT = \frac{-2V\gamma dr}{r^2}$$

Effects on the melting point

Chemical potential related with pressure: $d\mu = -SdT + VdP$

$$\mu_i^l = \mu_i^s$$

$$S_l - S_s = \Delta S_m$$

$$\Delta S_m dT = \frac{-2V\gamma dr}{r^2}$$

$$\Delta S_m = \frac{\Delta H_m}{T_m}$$

Bulk melting
temperature

$$\Delta T = \frac{2V\gamma T_m}{r\Delta H_m}$$

Change in melting point is inversely proportional to radius on the sphere → Grain size is reduced, melting point is lowered

Effects on the melting point

Chemical potential related with pressure: $d\mu = -SdT + VdP$

$$\mu_i^l = \mu_i^s$$

$$S_l - S_s = \Delta S_m$$

$$\Delta S_m dT = \frac{-2V\gamma dr}{r^2}$$

$$\Delta S_m = \frac{\Delta H_m}{T_m}$$

$$\Delta T = \frac{2V\gamma T_m}{r\Delta H_m}$$

Change in melting point is **inversely proportional** to **radius** on the sphere → **Grain size is reduced, melting point is lowered**

Effects on the melting point

Examples:

Nano-CdS of diameter ~ 2.5 nm melts at 600 K (bulk at 1675 K)

Single-walled carbon nanotube melts at 1600 K (bulk at 3800K)

Effects on the melting point

In contrast to nanoclusters and nano-agglomerates, nanoparticles within a matrix may experience an enhancement in the melting temperature. Matrix exerts pressure (p) that affects melting temperature

Effects on the melting point

In contrast to nanoclusters and nano-agglomerates, **nanoparticles within a matrix may experience an enhancement in the melting temperature**. Matrix exerts pressure (p) that affects melting temperature

$$p = \frac{2\mu\kappa\Delta V}{3V_o}$$

μ = shear modulus (rigidity/stiffness) of the matrix

κ = Dimensionless particle (presence of other particles)

ΔV = change of V due to thermal expansion

V_o = initial particle volume

Effects on the melting point

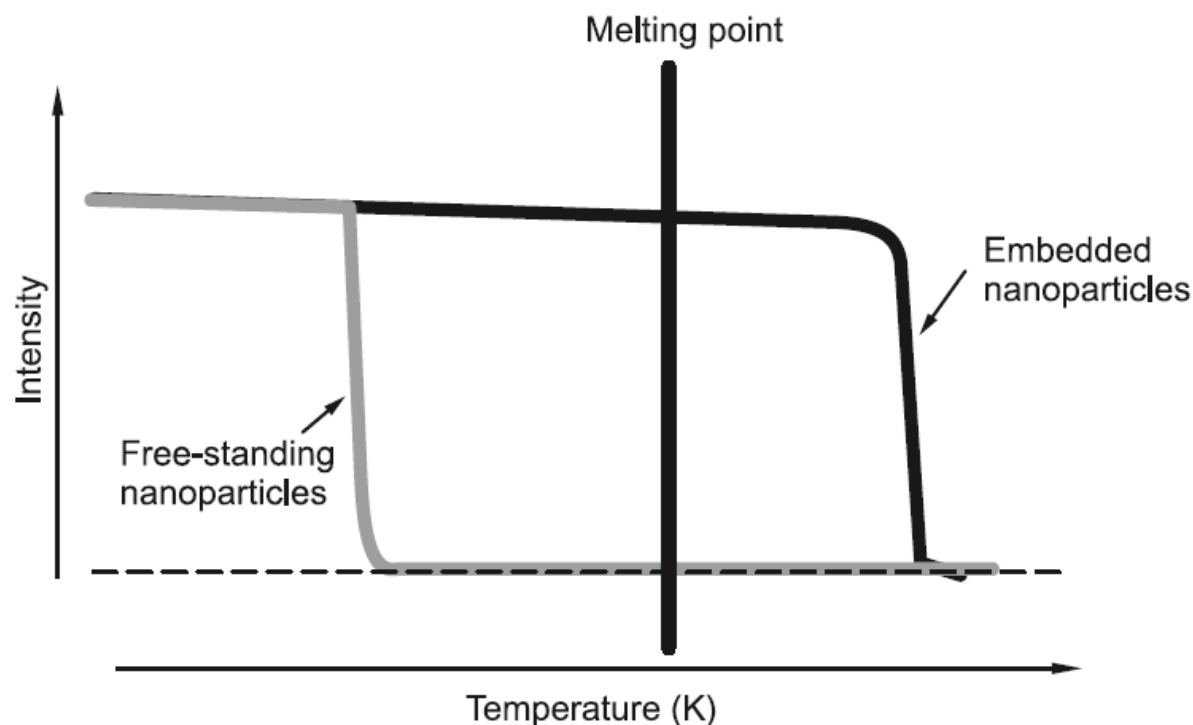
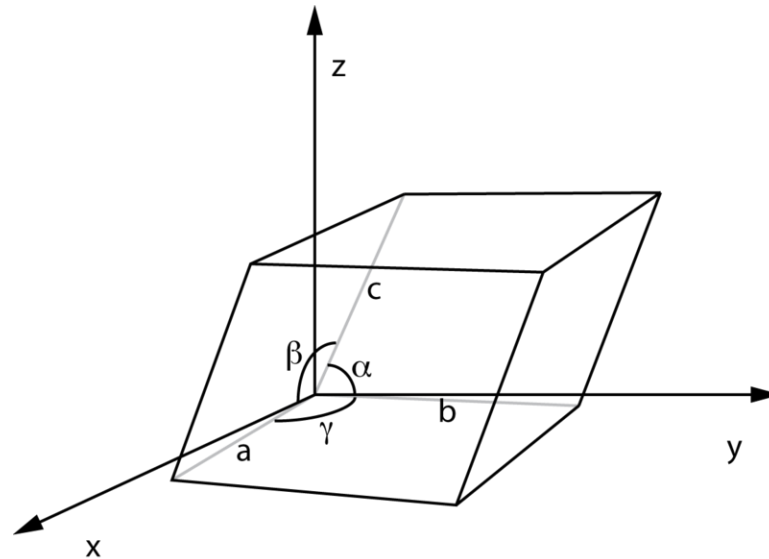


Fig. 2.4 Schematic diagram showing melting of free-standing nanoparticles and that of nanoparticles embedded in a higher melting matrix.

Effect on the lattice parameter

- The lattice parameter (or lattice constant) refers to the constant distance between unit cells in a crystal lattice



- Is the lattice parameter affected by increased surface/volume ratio (decreased nanoparticle size)?

Effect on the lattice parameter

- Ex.: A liquid sphere of diameter $2R$
 - Due to the curvature of the surface, a pressure is generated toward the inside of the sphere
 - The excess pressure ΔP inside the sphere, in the purely hydrostatic case, is given by the Laplace equation

$$\Delta P dV = \gamma dA$$

- dV is the volume change corresponding to a change dA in the area of the droplet
- In the case of a sphere, we obtain:

$$\Delta P = 2\gamma/R$$

- For a spherical solid:

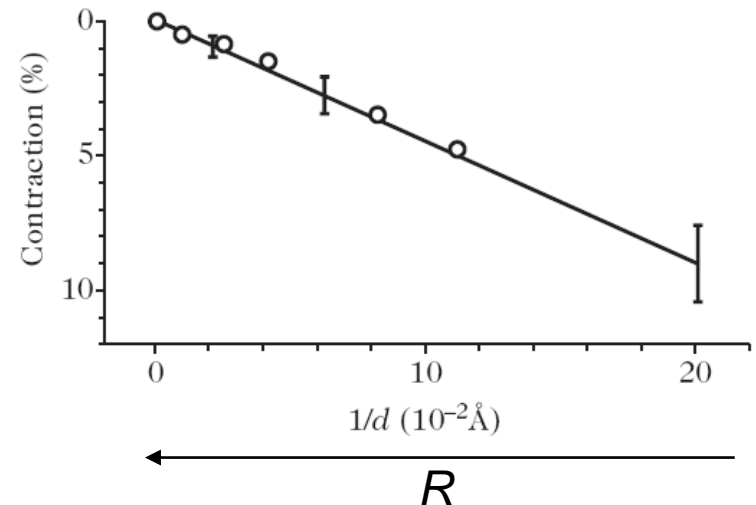
- γ replaced by g_{ij}
- χ = compressibility
- a = lattice parameter

$$\frac{\Delta a}{a} = -\frac{2}{3} \chi \frac{g}{R}$$

Effect on the lattice parameter (cont.)

- Contraction of the crystal lattice due to the pressure exerted toward the interior of the particle
 - Proportional to the surface stress
 - Inversely proportional to the particle size
- NB: Here metal nanoparticles

$$\frac{\Delta a}{a} = -\frac{2}{3} \chi \frac{g}{R}$$



Contraction of the lattice parameter of copper clusters as a function of the reciprocal of their diameter

Summary

- Defects
 - Examples
 - Problem B
- Physical properties: size effects
 - Lattice constant
 - Melting point

Next lecture

- Physical properties: size effects
 - Mechanical and optical properties
 - Electrical properties
 - Ferroelectrics
 - Dielectrics
 - Superparamagnetism