

고체내의 결함

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학습 목표

- 공공(vacancy)와 침입형 (interstitial) 결함 (defect)
- 특정 온도에서 재료가 가지는 공공(vacancy)의 개수 계산
- 세라믹 화합물에서 관찰되는 점 결함 (point defect)
- 두 고용체 (치환형 substitutional, 침입형 interstitial)
- 합금에서 요소들의 질량과 원자 무게가 주어질 때, 각 요소의 질량 비율과 원자 비율 계산하는 방법
- 전위 (dislocation)
 - 칼날, 나선 (edge, screw dislocation)
 - 전위 그리기, 전위 분석
- 결정립계 (grain boundary)
- 쌍정 (twin; twinning)



Imperfections in Solids

- 앞서 다룬 결정질(crystalline) 재료내에는 원자가 ‘완벽한’ 정렬로 배열되어 있다고 가정하였다.
- 하지만 실제 많은 재료는 이러한 ‘이상적인’ 배열에서 벗어나 있다.
- 결함(defect)과 불완전성(imperfection)
 - 1차원적
 - 2차원적
 - 3차원적
- 그러한 결함을 탐지하기 위한 몇몇 현미경 검사 기술

- Can the number and type of defects be varied and controlled?
- How do defects affect material properties?
- Are defects undesirable? Can we make use of them?
- 많은 재료들은 상온에서 고체상태로 쓰인다. 제조 공정에서 액체 상태의 물질이 고체로 바뀌면서 결함이 나타나기도 한다 (다음 장)



Patterns in Knives



왕좌의 게임의 Valyrian
steel은 허구이다.



하지만 Damascus steel?



https://en.wikipedia.org/wiki/Damascus_steel



Defects

□ 재료공학에서 “결함”은 가치 중립적 용어

- 나쁠 수도, 좋을 수도 있음.
- 결함은 제거해야 할 수도, 혹은 결함은 이끌어내야 할 수도 있음 (결함의 성질 이용)

□ 중요한 재료공학적 결함에 대해서 다루자.

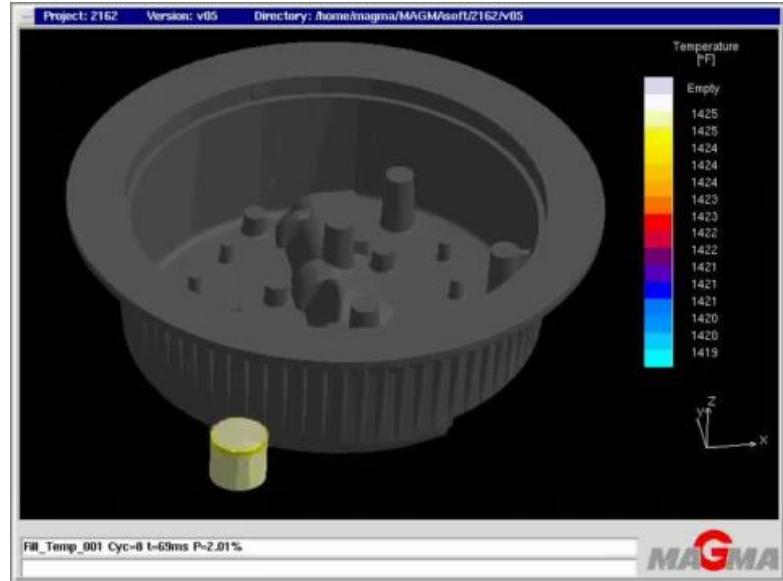
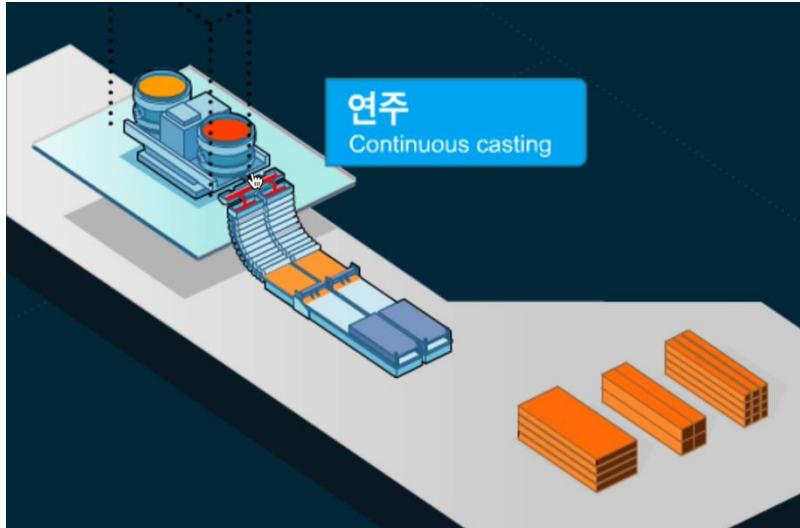
- 결정립과 결정립계
- 원자공공 (Vacancy) 불순물 (impurities) ...
- 전위 (dislocation)

□ 결함 관찰 방법

- 광학 현미경
- 전자 현미경



Solidification



<http://ekkinc.com>

<http://www.magmasoft.com.sg/en/>

포스코 철강 생산 공정 웹페이지:

<http://www.posco.co.kr/homepage/docs/kor5/html/product/exper/s91c5000103p.html>

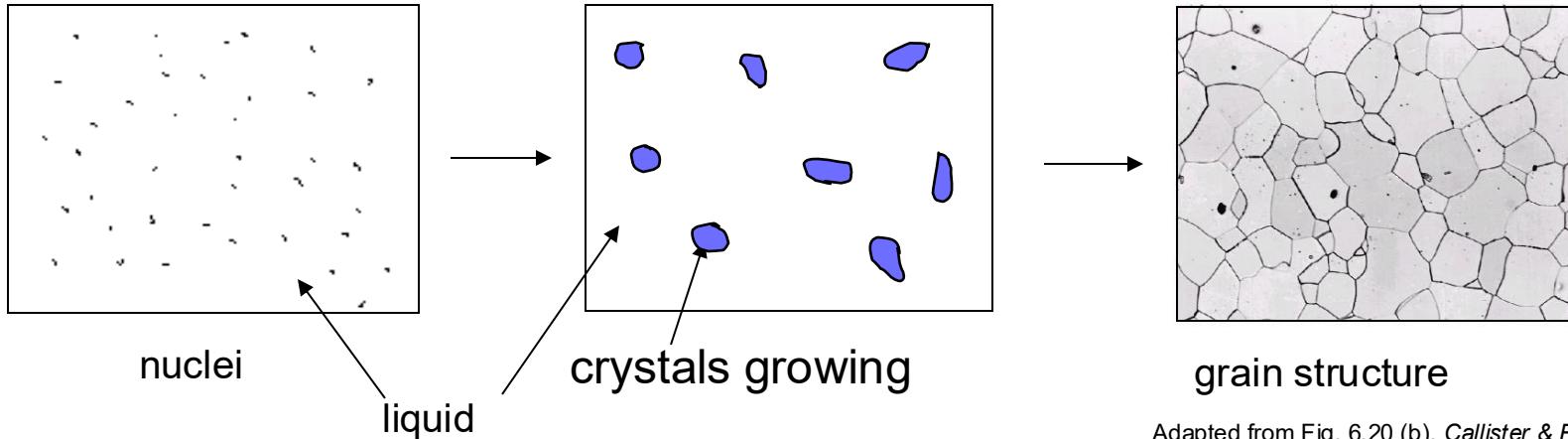


Imperfections in Solids

□ Solidification- result of casting of molten material

➤ 2 steps

- ❖ Nuclei form (핵생성)
- ❖ Nuclei grow to form crystals – grain structure (쌀알 무늬)



[Photomicrograph courtesy of L. C. Smith and C. Brady, the National Bureau of Standards, Washington, DC (now the National Institute of Standards and Technology, Gaithersburg, MD.)]

Adapted from Fig. 6.20 (b), Callister & Rethwisch 9e.

Crystals grow until they meet each other
(boundary; 경계)



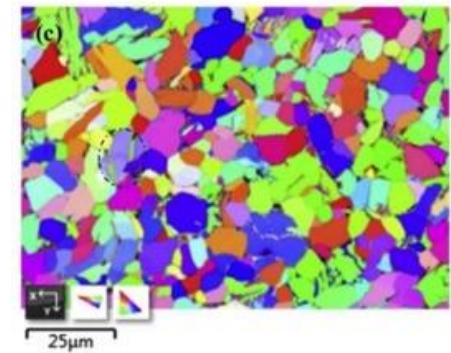
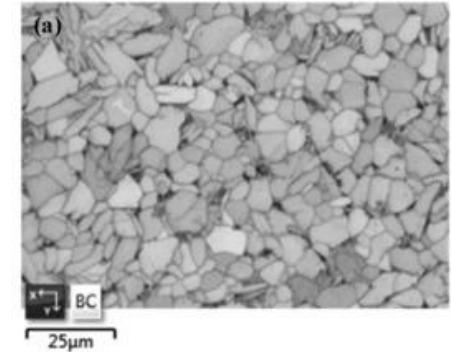
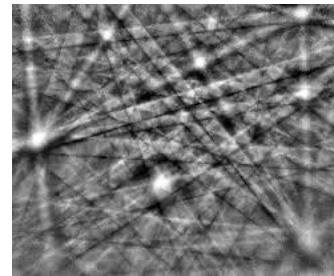


구조를 파악하기 위해 색입히기

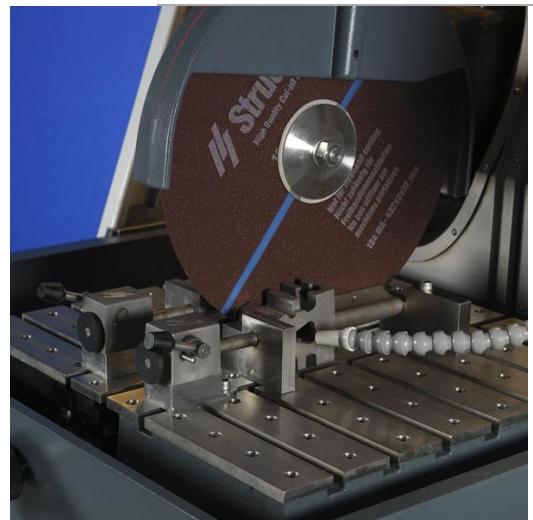


가시
광선

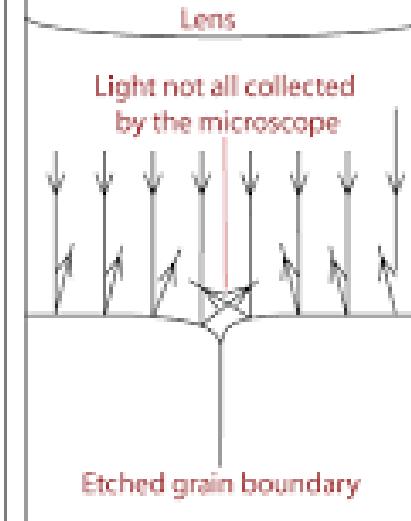
적외
선



결정립 관찰을 위한 시편준비 과정

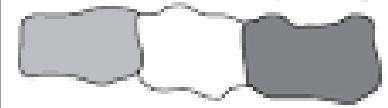


Examples of how contrast can arise

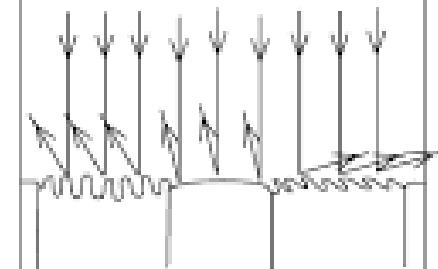


Etched grain boundary

Schematic of microscope image:



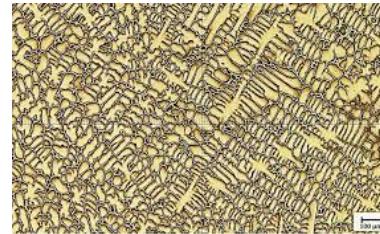
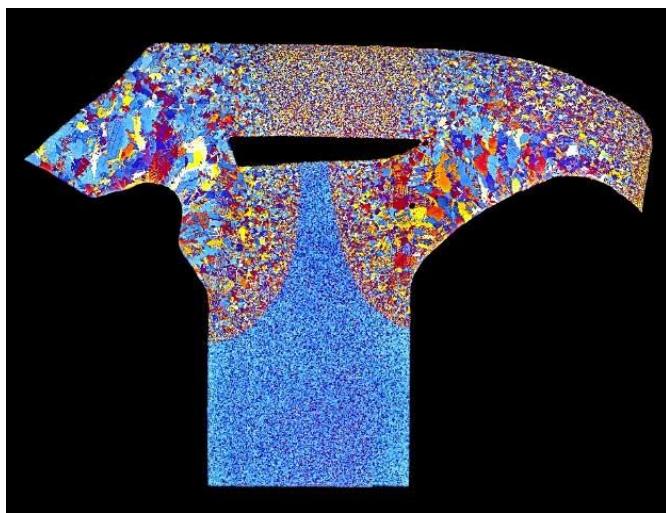
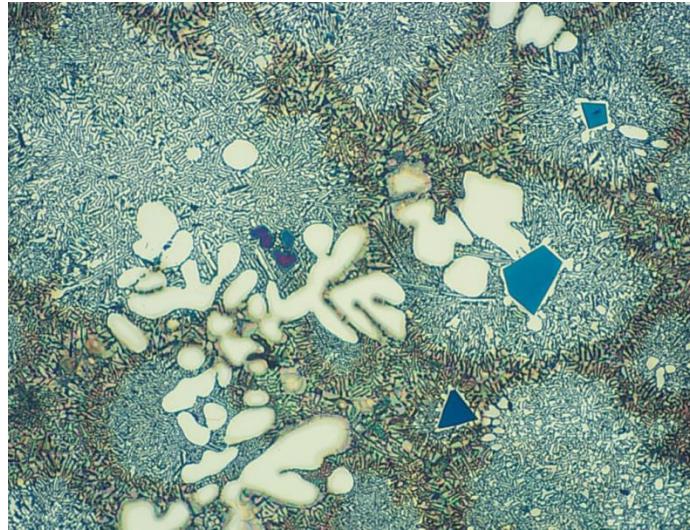
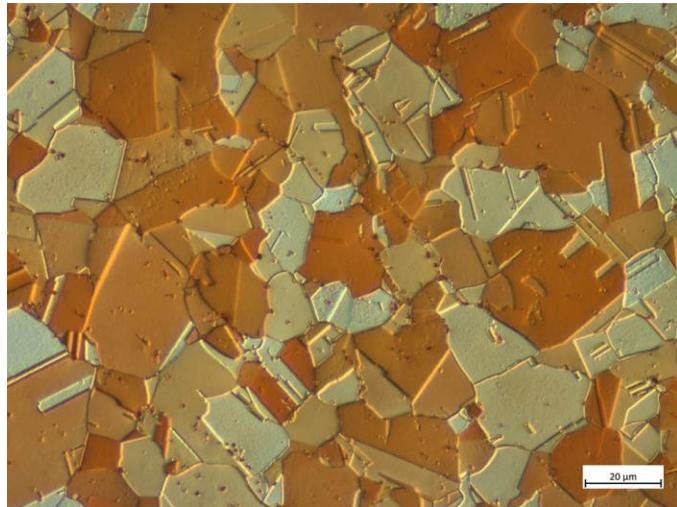
Different amounts of light are collected by the lens



Grains with different orientations are etched differently



OM examples.



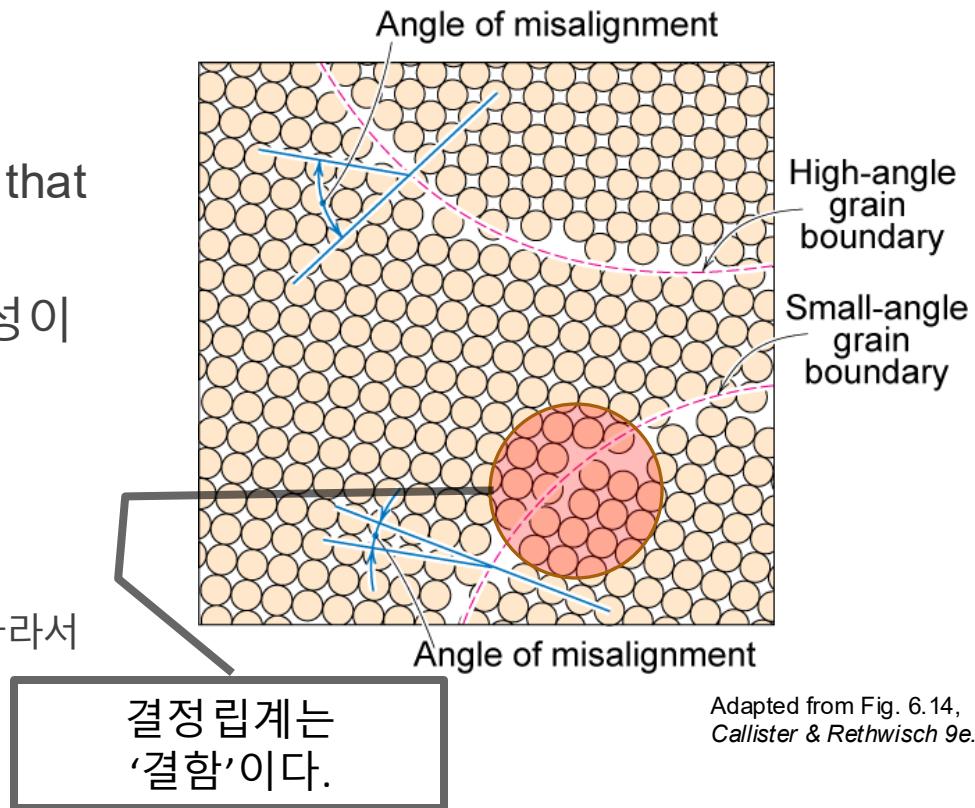
Welded
part



Polycrystalline Materials

Grain Boundaries

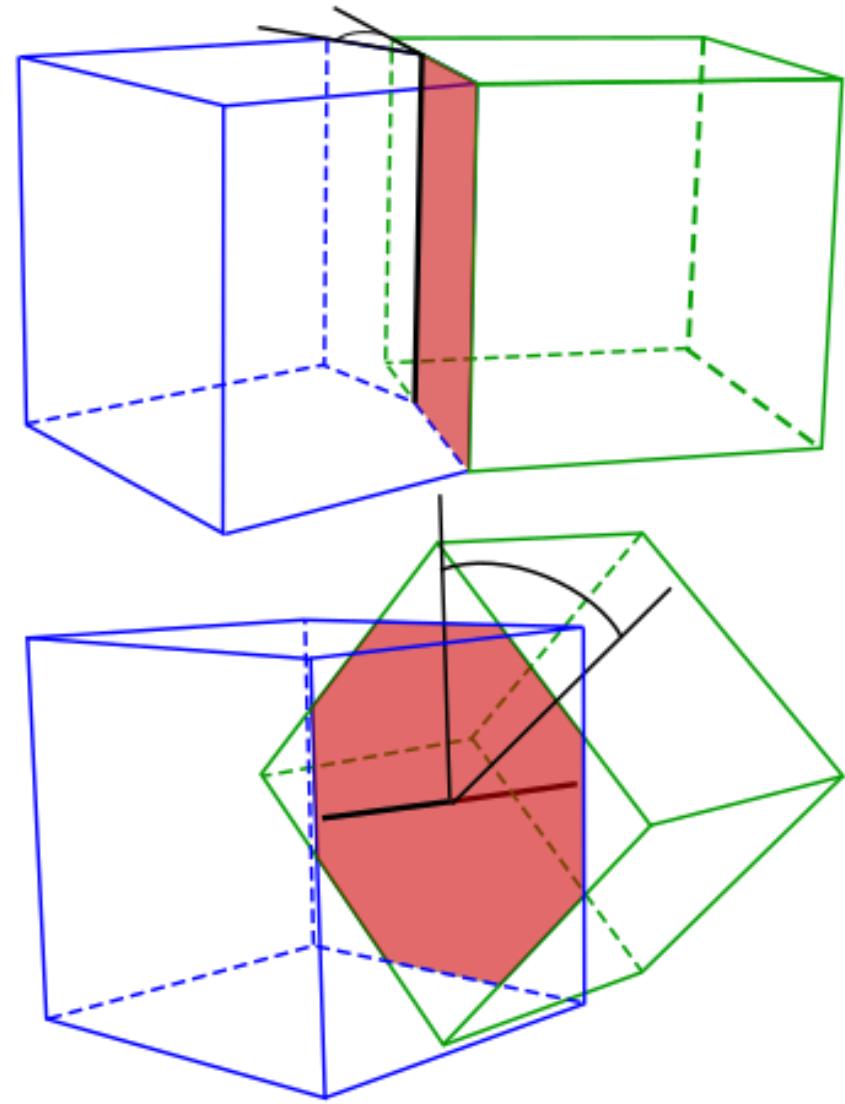
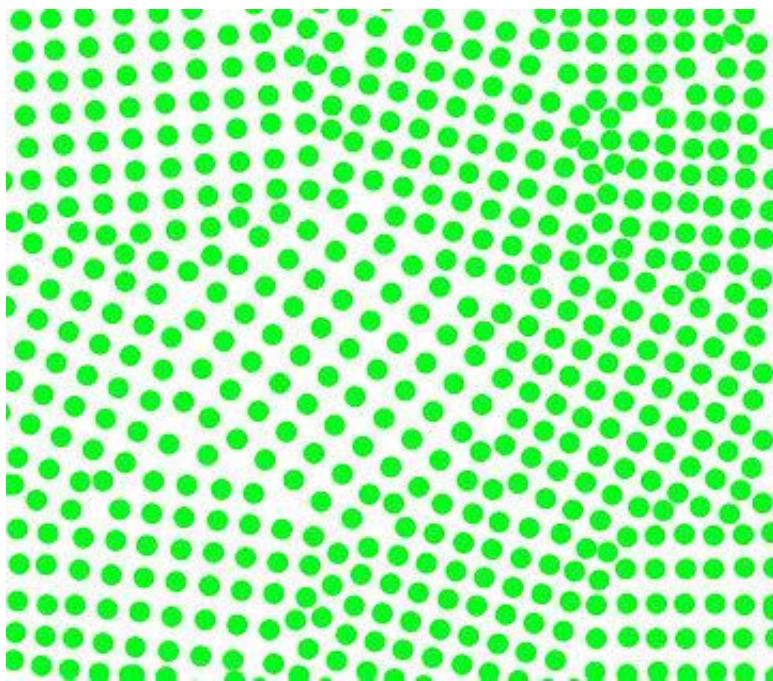
- Regions between crystals
- Transition from lattice of one region to that of the other
- Slightly disordered (원자 배열의 규칙성이 ‘약해진’ 구간)
- Low density in grain boundaries
 - high mobility (다른 원자들의 움직임이 빠름)
 - high diffusivity (확산이 빠름)
 - high chemical reactivity (온전하지 않은 결합-따라서 결합에 참여할 가능성 높음)



Adapted from Fig. 6.14,
Callister & Rethwisch 9e.

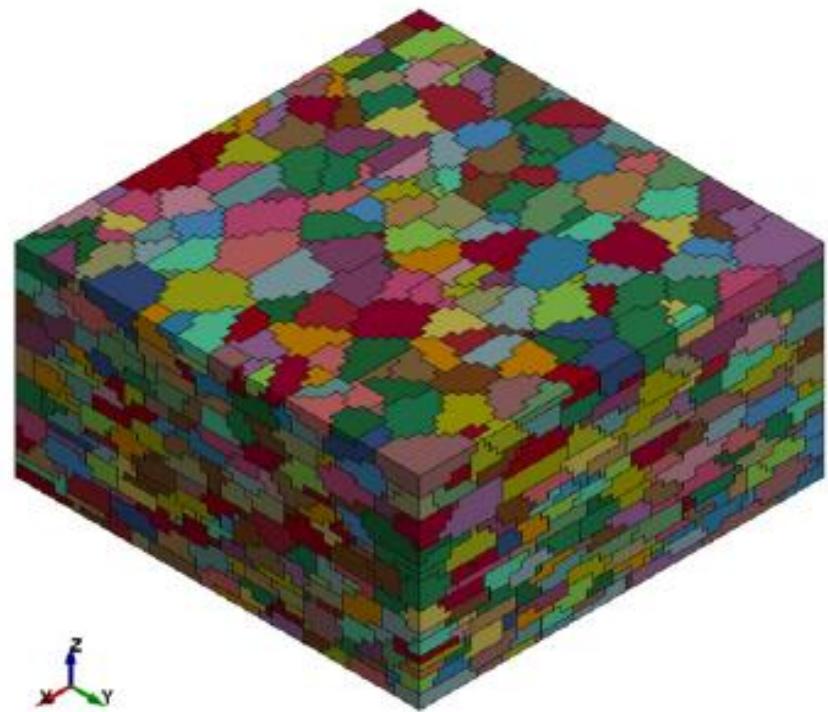
이렇듯, 재료의 응고 과정 중에서 결정 구조내에 자연스레 결함이 생겨날 수 있다. 다양한 결함에 대해 더 알아보자.







(a)



(b)



Types of Imperfections

Point defects (점결함)

- Vacancy atoms (원자공공)
- Interstitial atoms (침입형)
- Substitutional atoms (치환형)

Line defects (선결함)

- Dislocations (전위)

Area defects (면결함)

- Grain Boundaries (결정립계)

Volume defects (부피결함)

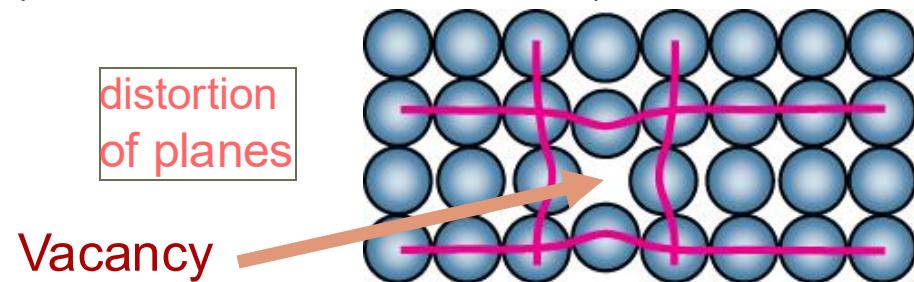
- Void (기포; 빈 공간)
- 2nd Phase (다른 상)



Point Defects in Metals

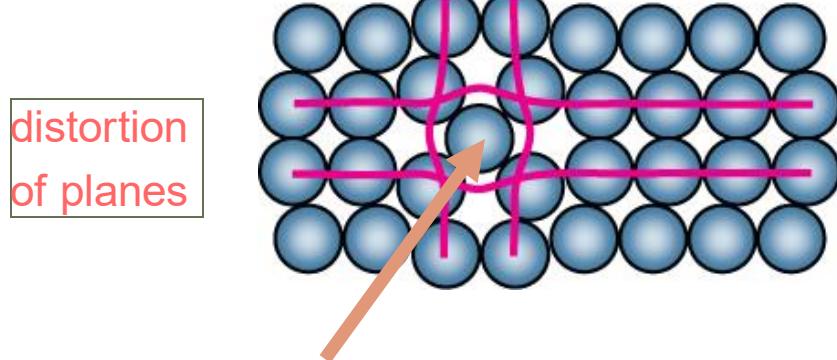
- Vacancies (공공):

-vacant atomic sites in a structure
(완벽했다면 채워졌을 빈 공간)

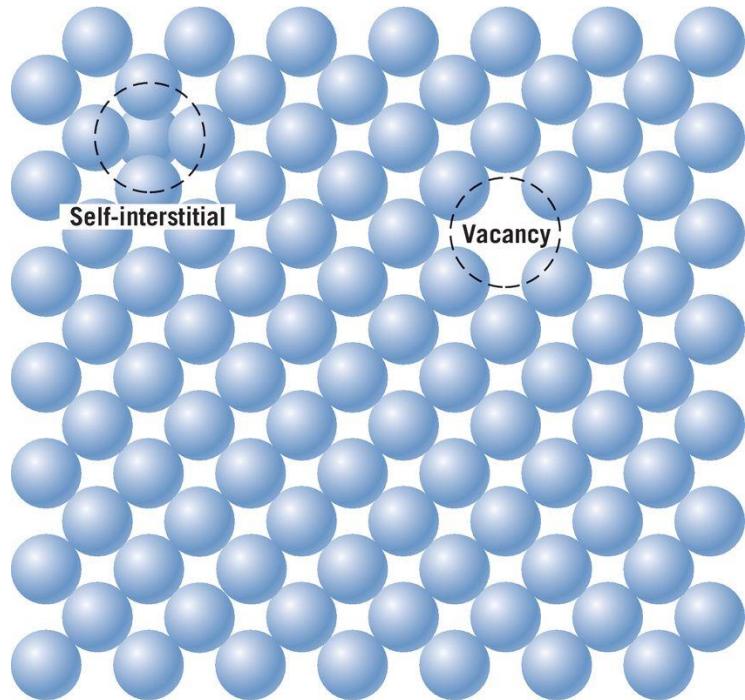


- Self-interstitials:

-"extra" atoms positioned between atomic sites
(완벽했다면 비워져있어야 할 곳에 끼워진..)



self-interstitial: 금속은 대개 close-packed 따라서 self-interstitial이 생길 가능성 낮다



Adapted from W. G. Moffatt, G. W. Pearsall, and J. Wulff, The Structure and Properties of Materials, Vol. I, Structure, p. 77.
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Equilibrium Concentration: Point Defects

- 원자 공공(vacancy)이 없는 결정질 고체를 만드는 것은 ‘거의’ 불가능에 가깝다.
- 온도에 따라서 단위 부피당 존재하는 (평형; equilibrium) 공공의 개수가 달라진다.

No. of defects

Activation energy (활성화 에너지)

No. of potential
defect sites

$$\frac{N_v}{N} = \exp\left(\frac{-Q_v}{kT}\right)$$

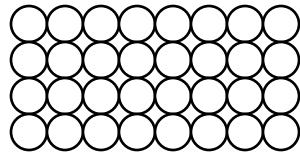
Temperature (Kelvin scale)

Boltzmann's constant

$$(1.38 \times 10^{-23} \text{ J/atom-K})$$

$$(8.62 \times 10^{-5} \text{ eV/atom-K})$$

Svante
Arrhenius



Each lattice site
is a potential
vacancy site

$$\frac{N_v}{N} = \exp\left(-\frac{Q_v}{kT}\right)$$

에서 평형 공공 농도 $\left(\frac{N_v}{N}\right)$ 와 온도(T)의 관계?

$T \uparrow$

$\frac{Q_v}{kT} \downarrow$

$-\frac{Q_v}{kT} \uparrow$

$\exp\left(-\frac{Q_v}{kT}\right) \uparrow$

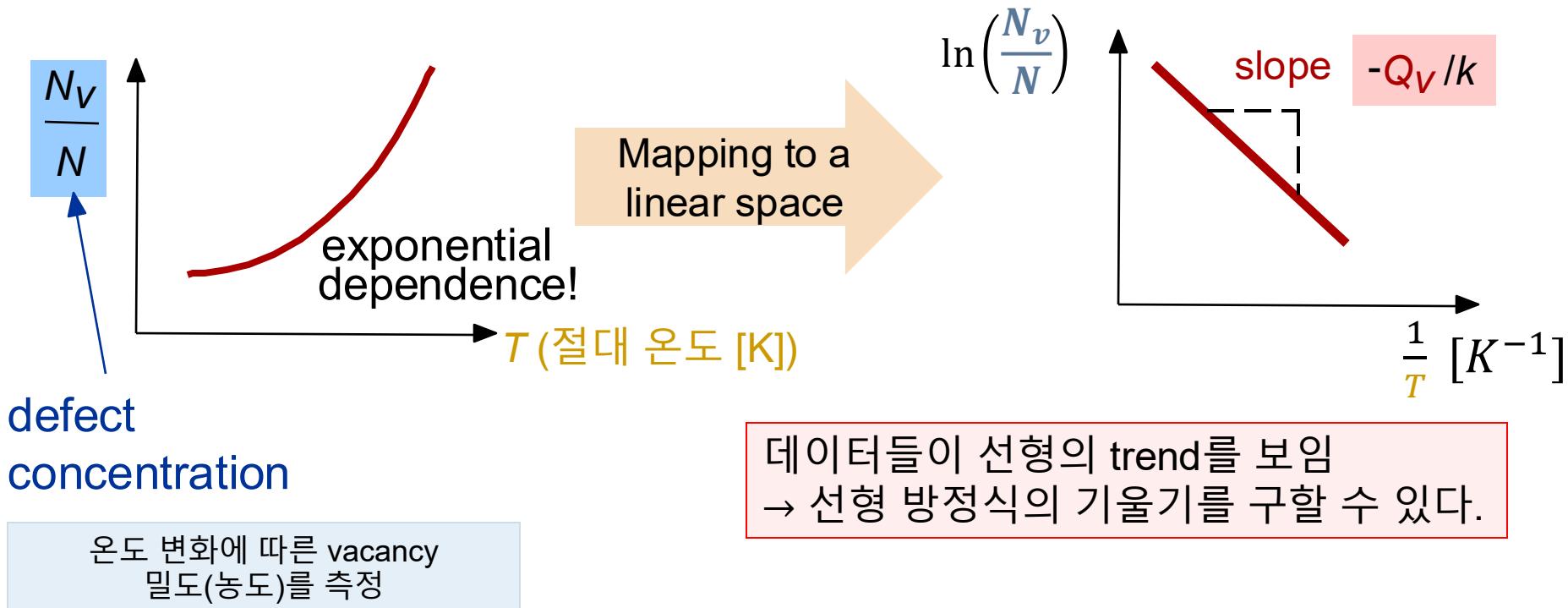
온도 증가에 의해 지수적으로(exponentially)
공공 농도 증가



Measuring Activation Energy

$$\frac{N_v}{N} = \exp\left(\frac{-Q_v}{kT}\right)$$

- How can we measure Q_v ?



Estimating Vacancy Concentration (단위 변환 주의)

- Find the Equil. # of vacancies (N_v) in **1 m³** of Cu at **1000°C**.

- 주어진 조건: $N_A = 6.02 \times 10^{23}$ atoms/mol $\rho = 8.4 \text{ g/cm}^3$

$$A_{\text{Cu}} = 63.5 \text{ g/mol}$$

$$Q_v = 0.9 \text{ eV/atom}$$

$$\frac{N_v}{N} = \exp\left(-\frac{Q_v}{kT}\right) = 2.7 \times 10^{-4}$$

1273 K

**1 m³ of copper
속 공공의 밀도(농도)**

$$8.62 \times 10^{-5} \text{ eV/(atom · K)}$$

$$\frac{N_v}{N} = 2.7 \times 10^{-4}$$

1 m³ 속 copper atoms 수:
 $N = (\text{몰수}) \times N_A$

$$\text{몰수} = (\text{부피당 몰수}) \times \text{부피}$$

$$(\text{부피당 몰수}) = \left(\frac{\text{질량} \times \text{부피}}{\text{질량}} \right) \text{당 몰수}$$

$$(\text{부피당 몰수}) = \frac{\text{몰수}}{\left(\frac{\text{질량} \times \text{부피}}{\text{질량}} \right)} = \text{밀도} \times \frac{\text{몰수}}{\text{질량}} = \text{밀도} \times \frac{1}{\frac{\text{질량}}{\text{밀도}}} = \frac{\text{밀도}}{\text{원(분)자량}}$$

$$N = \left(\frac{\text{밀도}}{\text{원(분)자량}} \times \text{부피} \right) \times N_A$$

$$N = \frac{8.4[\text{g/cm}^3]}{63.5[\text{g/mol}]} \times 1[\text{m}^3] \times 6.02 \times 10^{23} \left[\frac{\text{atoms}}{\text{mol}} \right] = \frac{8.4}{63.5} \times 6.02 \times 10^{23} [\text{mol/cm}^3] \cdot [\text{m}^3] \cdot \left[\frac{\text{atoms}}{\text{mol}} \right]$$

$$= \frac{8.4}{63.5} \times 6.02 \times 10^{23} \left[\frac{\text{mol}}{\text{cm}^3} \cdot \text{m}^3 \cdot \frac{\text{atoms}}{\text{mol}} \right]$$

$$= 7.96 \times 10^{22} \left[\frac{\text{m}^3}{\text{cm}^3} \cdot \text{atoms} \right]$$

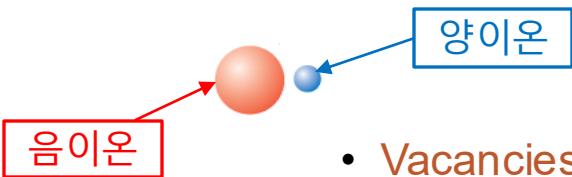
$$= 7.96 \times 10^{28} [\text{atoms}]$$

$$N_v = 2.7 \times 10^{-4} \times 7.96 \times$$

$$\frac{1}{10^{28} [\text{atoms}]}$$



Point Defects in Ceramics (i)



- Vacancies (원자 공공)
 - vacancies exist in ceramics for both cations (양이온) and anions (음이온)
- Interstitials (침입형 결함)
 - interstitials exist for cations
 - interstitials are not normally observed for anions because anions are large relative to the interstitial sites

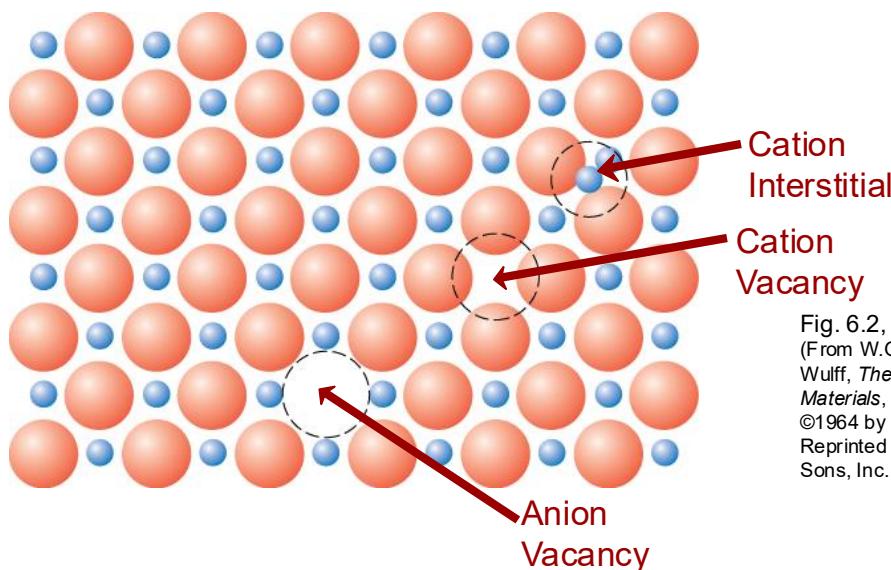


Fig. 6.2, Callister & Rethwisch 9e.
(From W.G. Moffatt, G.W. Pearsall, and J. Wulff, *The Structure and Properties of Materials*, Vol. 1, *Structure*, p.78. Copyright ©1964 by John Wiley & Sons, New York.
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Point Defects in Ceramics (ii)

세라믹 결합에 참여하는 원자들은 이온상태 (전하를 띤다). 따라서 결합 구조를 고려할 때, 전기중성도(electroneutrality)를 고려해야 한다. 결합이 짹 (pair)로 나타난다.

- Frenkel Defect
 - a cation vacancy (양이온 공공) - cation interstitial (양이온 침입) pair (狎).
- Shottky Defect
 - a paired set of cation and anion vacancies – (양이온 공공-음이온 공공 짹)

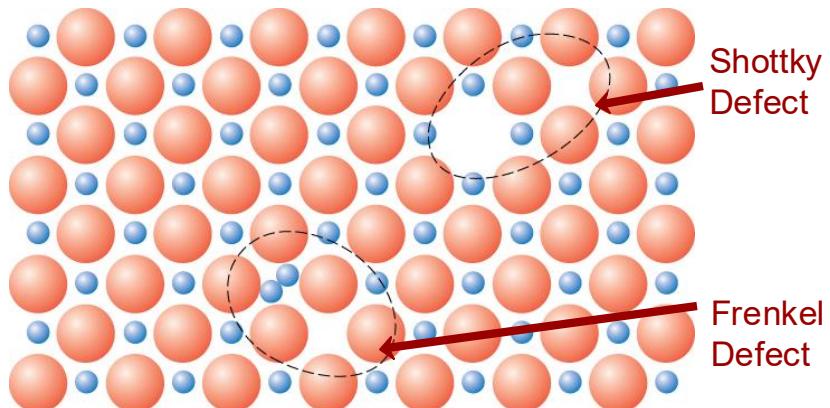


Fig. 6.3, Callister & Rethwisch 9e.
(From W.G. Moffatt, G.W. Pearsall, and J.
Wulff, *The Structure and Properties of
Materials*, Vol. 1, *Structure*, p.78. Copyright
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Sons, Inc.)

- Equilibrium concentration of defects $\propto e^{-Q_D/kT}$

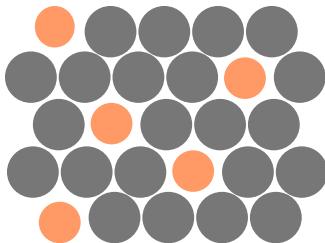


금속의 불순물

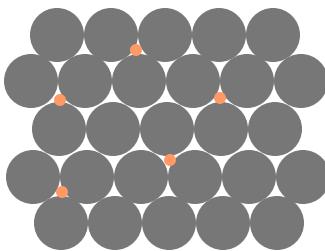
- 결함이 없는 금속이 존재하기 힘들 듯, 한 종류의 원자로만 이루어진 순수한 금속이 존재하기도 매우 힘들다. 대부분 불순물 (impurity) 혹은 외부 원자들이 존재한다.
- 공학적 용도로 사용되는 금속의 대부분은 ‘합금(alloy)’ – 특정한 성질을 갖기 위해서

불순물 B 를 모재 (host material)에 첨가하면 고용체(solid solution)나 제2의 상(second phase)이 형성된다

- Solid solution of B in A (B가 A 속에 균일하게 섞여있는 고용체)



OR



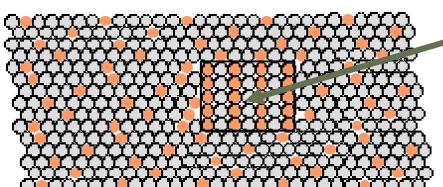
Interstitial solid solution
(e.g., C in Fe)

Substitutional solid solution
(e.g., Cu in Ni)

□ 용어 정리

- Solution (용체)
 - Liquid, Gas, Solid
- Solvent (용매)
- Solute (용질)

- Solid solution of B in A plus particles of a new phase (usually for a larger amount of B)



Second phase particle
-- different composition
-- often different structure.

용질 원자가 용매원자에
고용되는 정도?



Hume-Rothery rule (흄-로더리 법칙)

Conditions for substitutional solid solution (S.S.)

□ Hume – Rothery rule

- 1. Δr (atomic radius) < 15%
- 2. Same crystal structure for pure metals
- 3. Proximity in periodic table
 - ❖ i.e., similar electronegativities (비슷한 전기 음성도)
 - ❖ Atoms with dissimilar electronegativites tend to form a chemical compound (화합물; 주로 ionic 결합방식을 따름)
- 4. Valency (원자가)
 - ❖ 용질과 용매가 같은 원자가(valency)를 가진다면 완전히 녹게 된다. 금속은 더 높은 원자가의 금속을 용해할 가능성이 그 역의 경우보다 더 높다

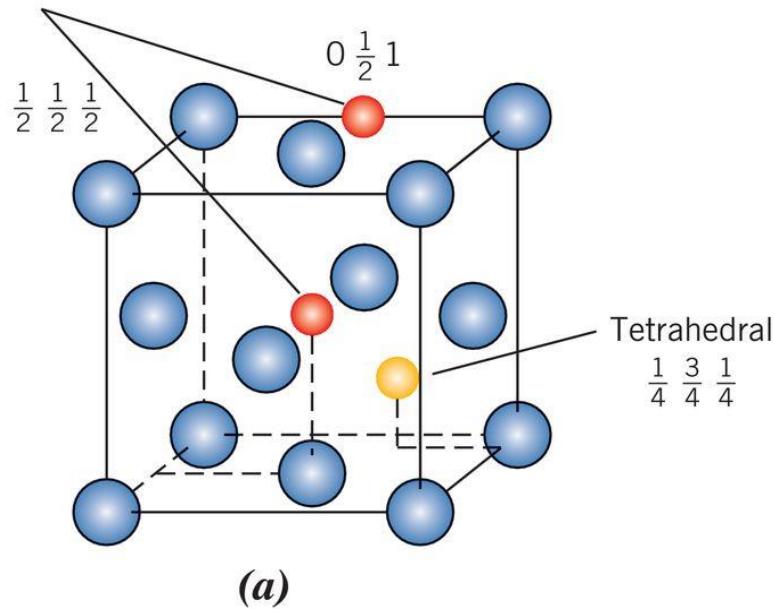


William Hume-Rothery OBE FRS
(15 May 1899 – 27 September 1968) was an English metallurgist and materials scientist who studied the constitution of alloys.



고용체내의 침입형 불순물 원자의 위치

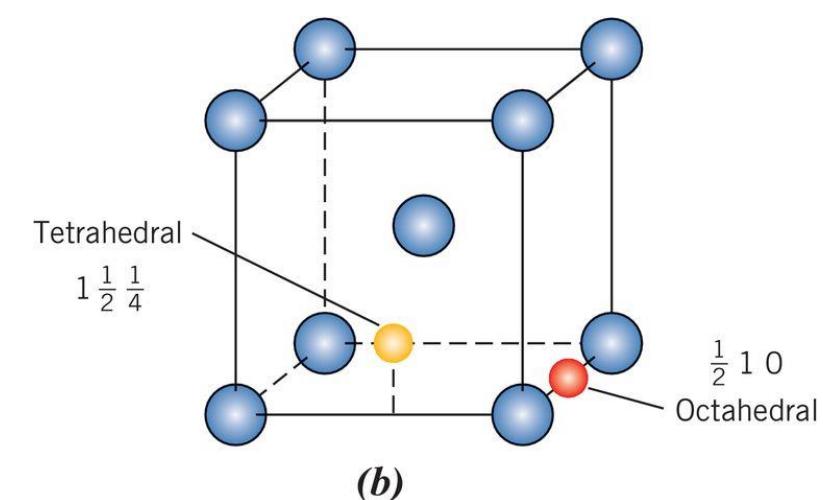
Octahedral



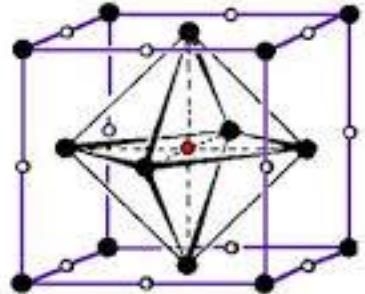
(a)

Tetrahedral

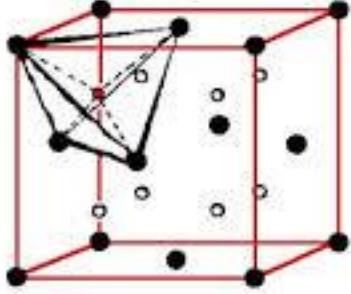
$1, \frac{1}{2}, \frac{1}{4}$



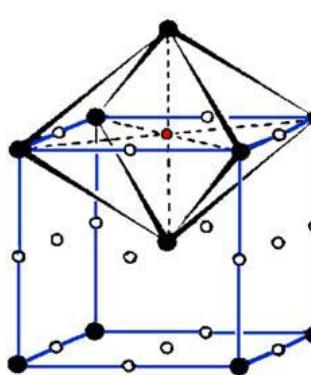
(b)



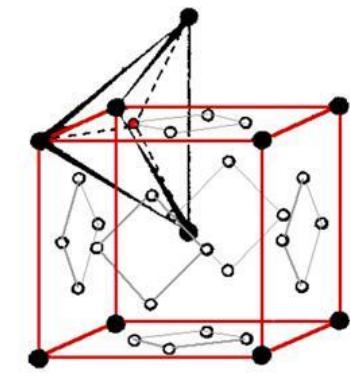
Octahedral Site



Tetrahedral Site



Octahedral Site



Tetrahedral Site



조성 (chemical composition) 표기법

합금의 조성 (composition – 혹은 농도)를 표기 하는 방법.

무게비 (weight percent, wt\%)

전체 합금 무게에 대한 특정 원소 무게(질량)비)

원자비 (atom percent, at\%)

(합금 전체 원소의 총 몰수 vs. 특정 원소 몰수비)

Q. 어떤 합금이 a 와 b 원소로 이루어 져 있을 때...

$$n_a = m_a/A_a$$

$$C_a = \frac{m_a}{m_a + m_b} \times 100 \ (\text{wt\%})$$

$$C'_a = \frac{n_a}{n_a + n_b} \times 100 \ (\text{at\%})$$

무게비 \Leftrightarrow 원자비 변환

$$C'_a = \frac{n_a}{n_a + n_b}$$

$$C'_a = \frac{m_a/A_a}{m_a/A_a + m_b/A_b}$$

$$= \frac{m_a \cdot A_b}{m_a \cdot A_b + m_b \cdot A_a}$$

$$= \frac{\left(\frac{m_a}{m_a + m_b}\right) \cdot A_b}{\left(\frac{m_a}{m_a + m_b}\right) \cdot A_b + \left(\frac{m_b}{m_a + m_b}\right) \cdot A_a} = \frac{C_a \cdot A_b}{C_a \cdot A_b + C_b \cdot A_a}$$

$$C'_a = \frac{C_a \cdot A_b}{C_a \cdot A_b + C_b \cdot A_a}$$



예제

wt % → at %로의 조성 변환

97 wt % Al & 3 wt % Cu로 이루어진 alloy의 at % ?

$$A_{\text{Cu}} = 63.55 \text{ g/mol}, A_{\text{Al}} = 26.98 \text{ g/mol}$$

$$C_{\text{Al}} = 97, C_{\text{Cu}} = 3$$

$$C'_{\text{Al}} = \frac{C_{\text{Al}} \cdot A_{\text{Cu}}}{C_{\text{Al}} \cdot A_{\text{Cu}} + C_{\text{Cu}} \cdot A_{\text{Al}}}$$

$$C'_{\text{Cu}} = \frac{C_{\text{Cu}} \cdot A_{\text{Al}}}{C_{\text{Al}} \cdot A_{\text{Cu}} + C_{\text{Cu}} \cdot A_{\text{Al}}}$$

$$= \frac{97\% \cdot A_{\text{Cu}}}{97\% \cdot A_{\text{Cu}} + 3\% \cdot A_{\text{Al}}}$$

$$= \frac{97\% \cdot 63.55 \text{ g/mol}}{97\% \cdot 63.55 \text{ g/mol} + 3\% \cdot 26.98 \text{ g/mol}}$$

$$C'_{\text{Al}} = 98.7 \text{ at\%}$$

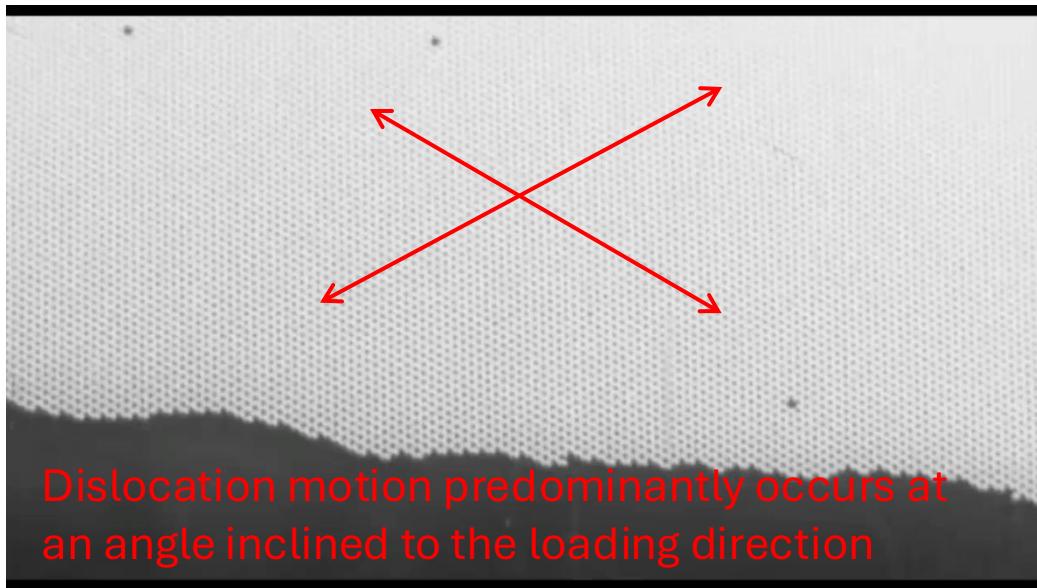
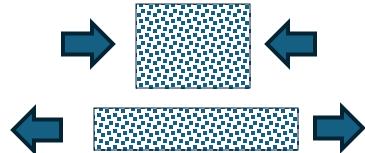
$$C'_{\text{Cu}} = 100 \text{ at\%} - C'_{\text{Al}}$$



Plastic anisotropy and dislocations

Bubble raft model

(A Dynamical Model of a Crystal Structure, Lawrence Bragg;
John F. Nye, Proc. Roy. Soc. Lon.)



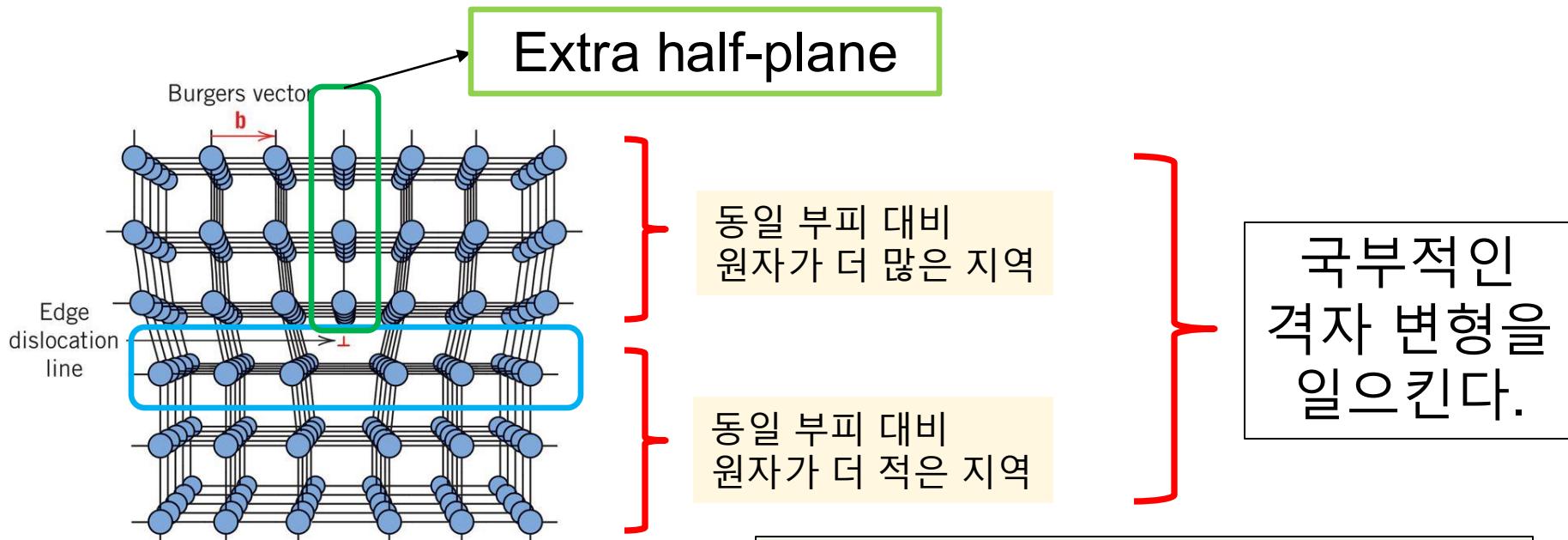
<https://youtu.be/UEB39-jlmdw>

Experiments with the Bubble Model of Metal Structure 1952
Sir Lawrence Bragg, W.M Lomer, J.F. Nye

전위 (dislocation; 대표적인 선결함)

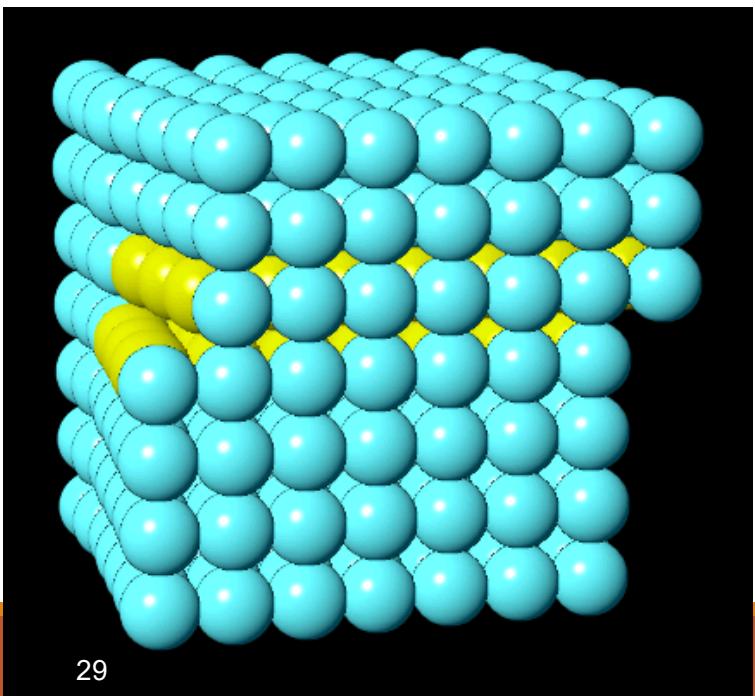
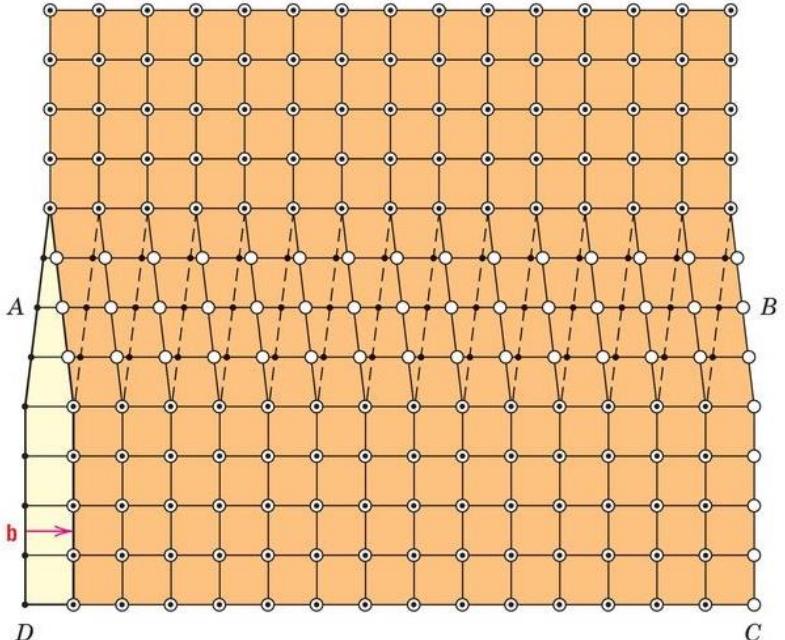
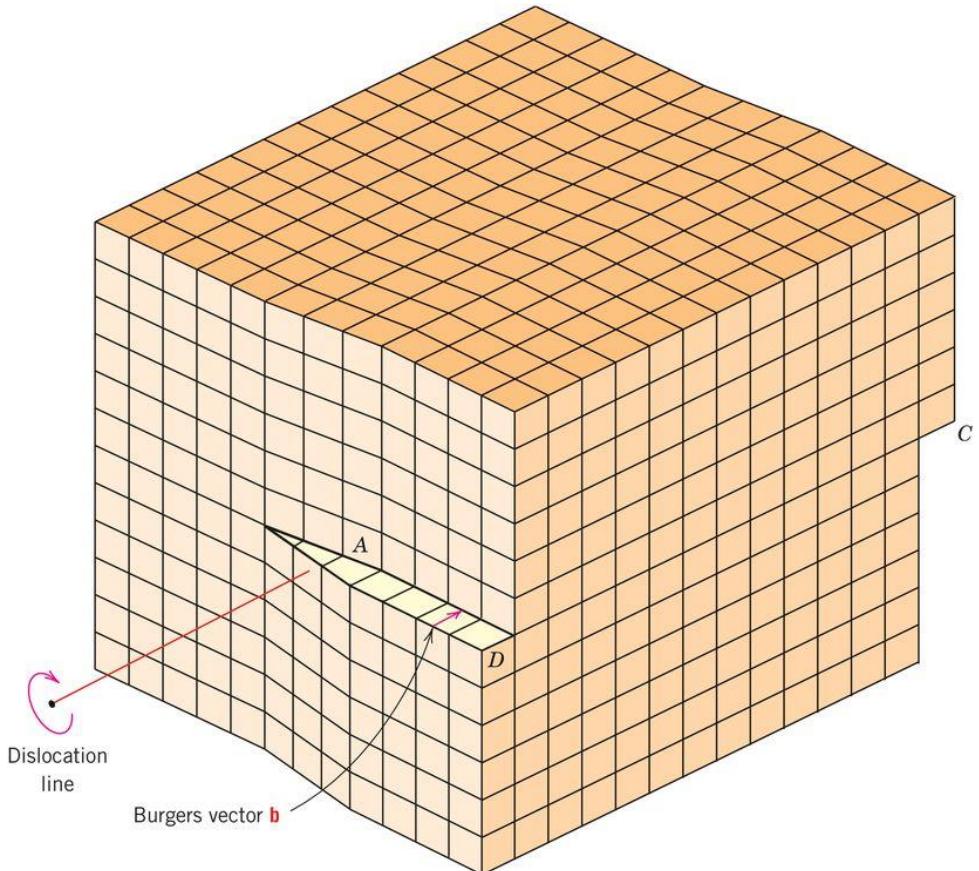
전위 (dislocation)

- 칼날 전위 (edge dislocation)
- 나선 전위 (screw dislocation)

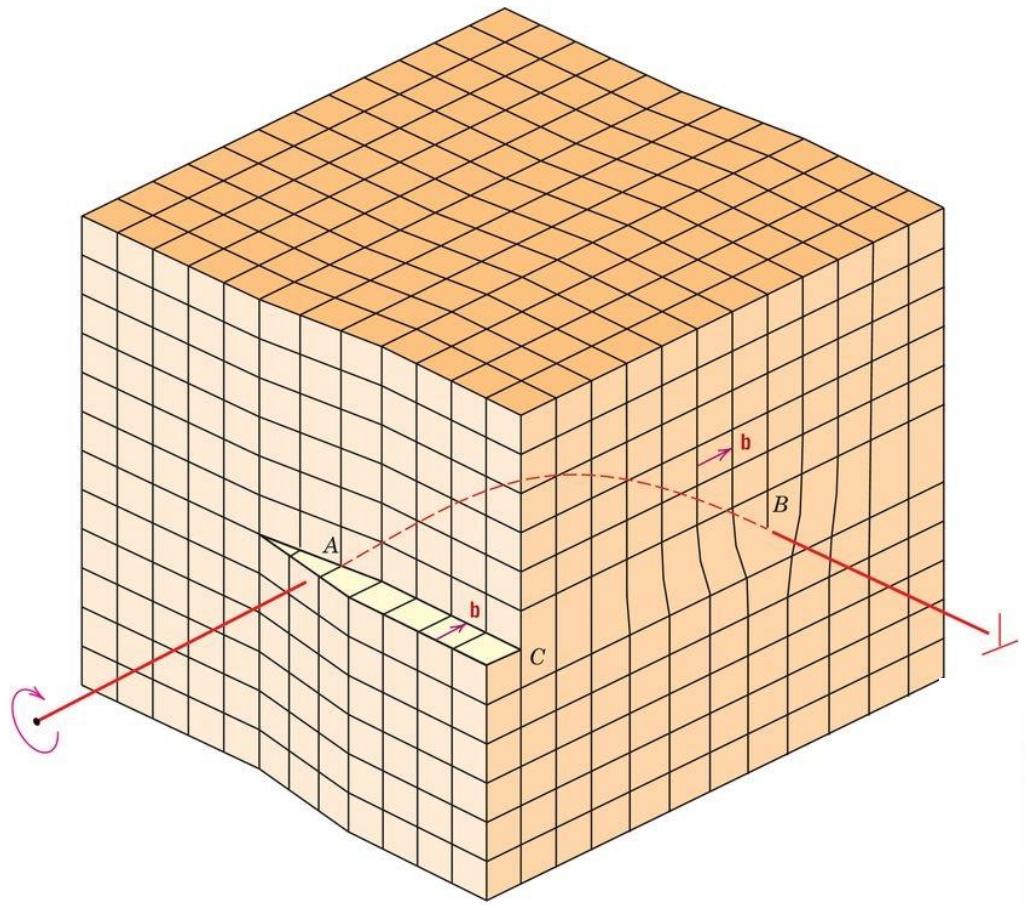


Burgers vector (b):
전위에 의해 발생하는 격자 뒤틀림(lattice distortion)의 크기 및 방향을 나타냄

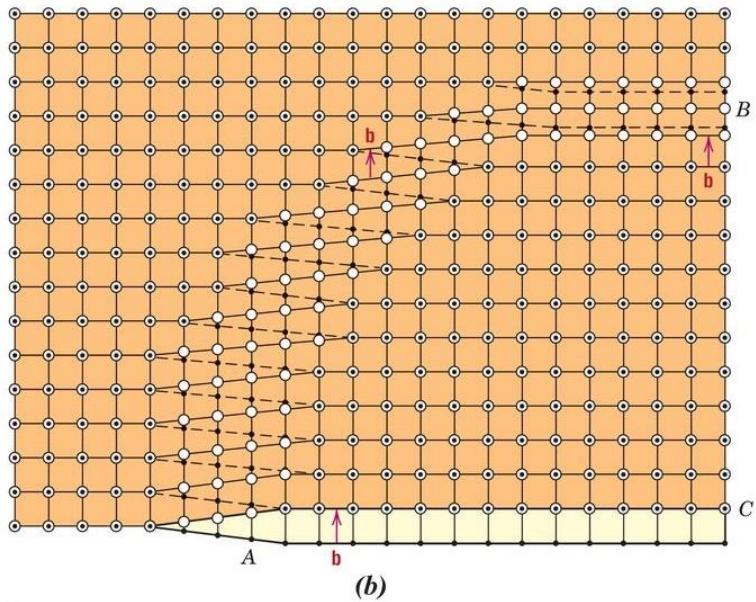




Screw dislocation



Edge와 screw dislocation이
연속적으로 나타난 모형



Imperfections in Solids

전위는 전자현미경(electron microscope)으로 관찰 가능.

아래 사진에서 검은 '선'이 전위이다.

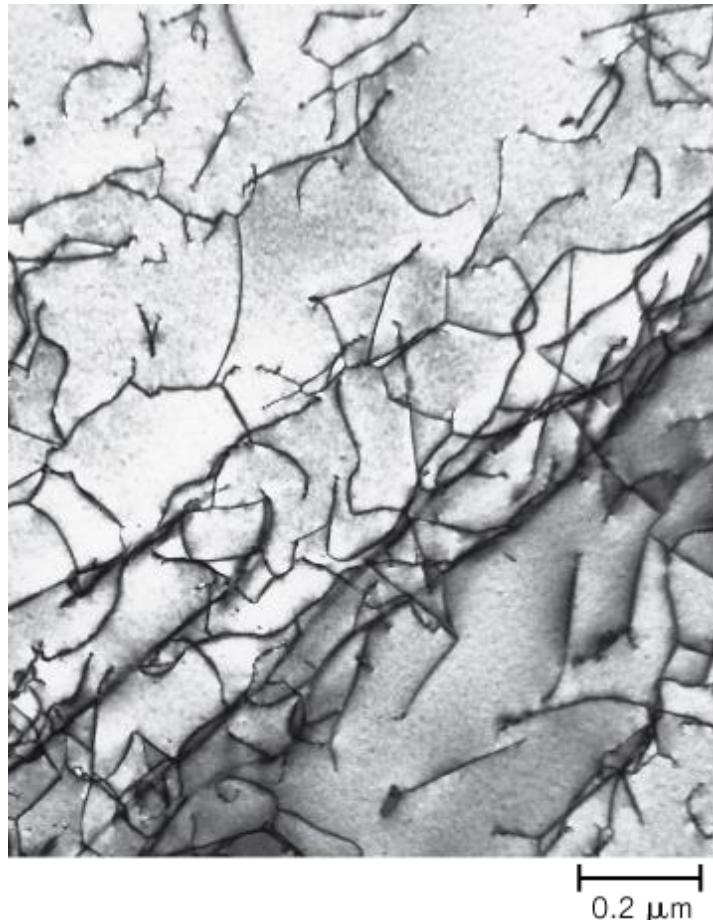


Fig. 6.12, Callister & Rethwisch 9e.
(Courtesy of M. R. Plichta, Michigan
Technological University.)



계면 결함 (interfacial defect)

▣ 계면 결함 (다른 결정 구조, 또는 다른 결정 방향을 분리하는 경계면)

- 외부 표면 (surface)
- 결정립계 (grain boundary)
- 상계면 (phase boundary)
- 쌍정립계 (twinning)
- 적층 결함 (stacking faults)

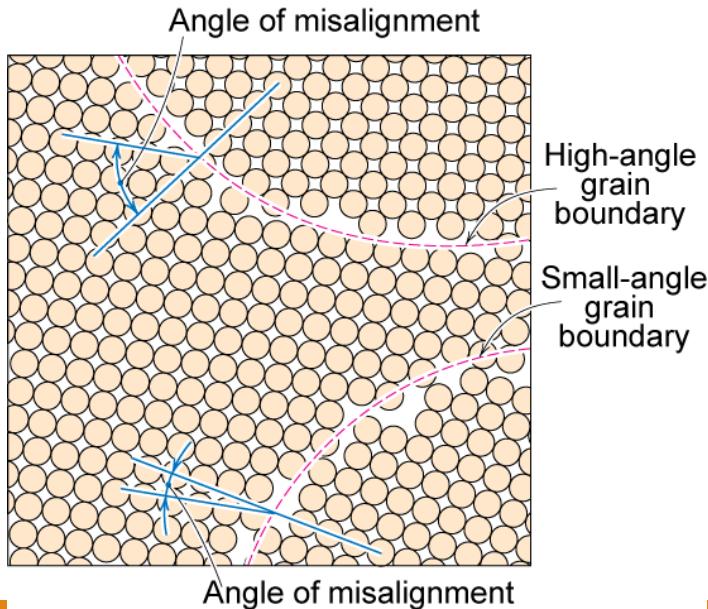
- 외부 표면에 드러난 원자는 내부에 원자들과 비교했을 때 최대한의 결합수를 만족하지 못한다.
- 불완전한 결합 상태는 표면 에너지를 유발하고, 재료는 이러한 표면 에너지를 최소화하려 한다.



결정립계 (grain boundary)

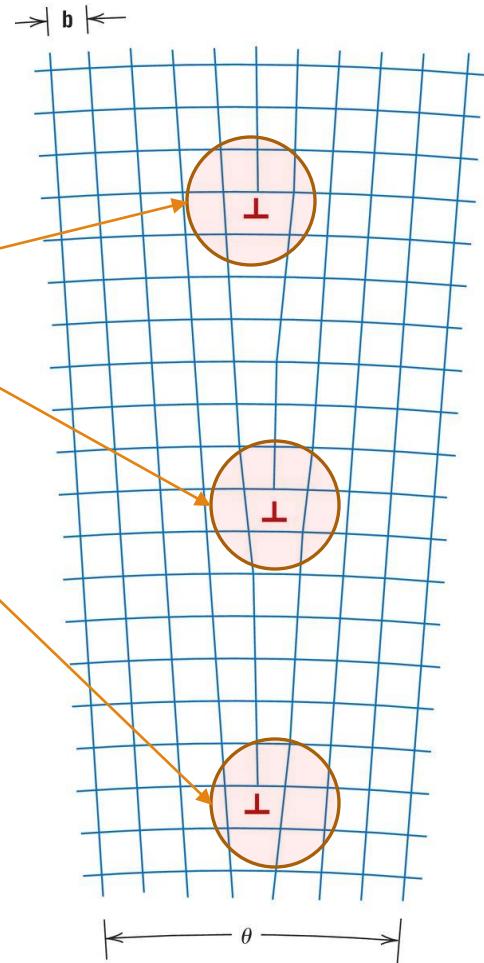
□ 계면 결함 (다른 결정 구조, 또는 다른 결정 방향을 분리하는 경계면)

- 외부 표면 (surface)
- 결정립계 (grain boundary)
- 상계면 (phase boundary)
- 쌍정립계 (twinning)
- 적층 결함 (stacking faults)



규칙적인
전위의 배열로
설명 가능

Small-angle grain
boundary



상의 계면 (phase boundary)

□ 계면 결함 (다른 결정 구조, 또는 다른 결정 방향을 분리하는 경계면)

- 외부 표면 (surface)
- 결정립계 (grain boundary)
- **상계면 (phase boundary)**
- 쌍정립계 (twinning)
- 적층 결함 (stacking faults)

회주철 (gray cast iron)
상계면



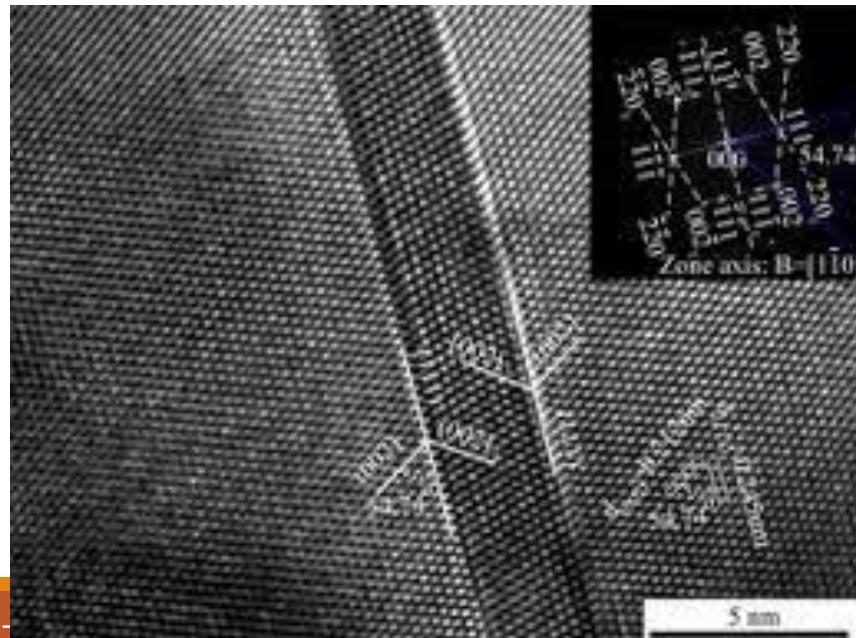
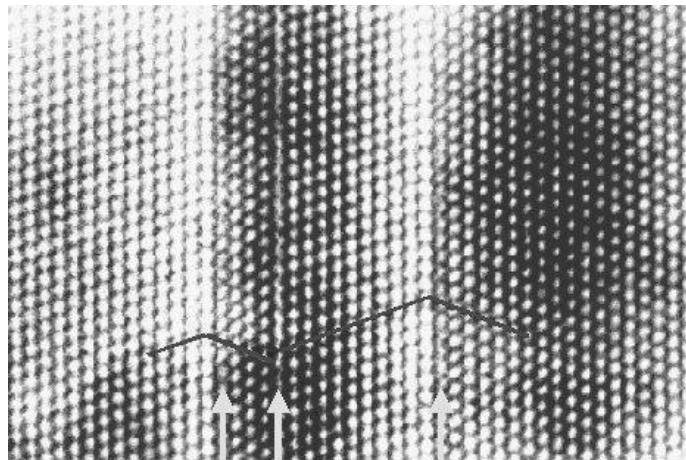
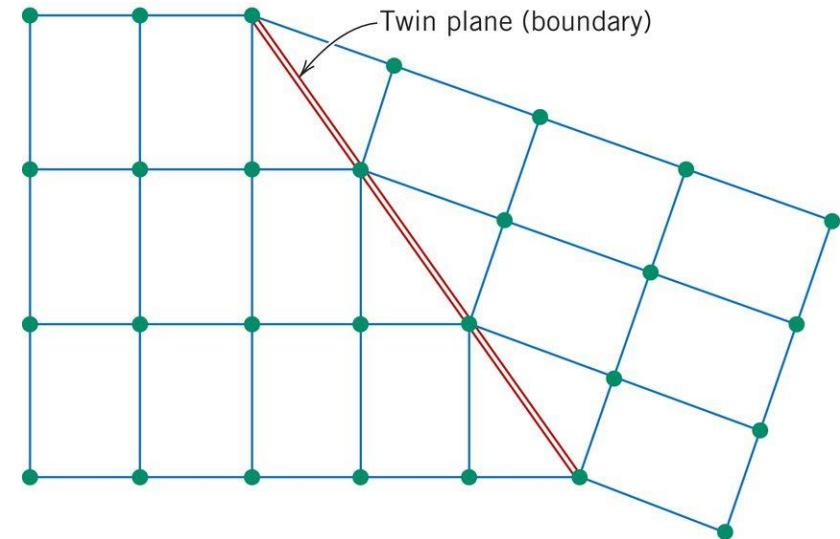
courtesy of C. H. Brady and L. C. Smith, National Bureau of Standards, Washington, DC (now the National Institute of Standards and Technology, Gaithersburg, MD).



상계면 (phase boundary)

□ 계면 결함 (다른 결정 구조, 또는 다른 결정 방향을 분리하는 경계면)

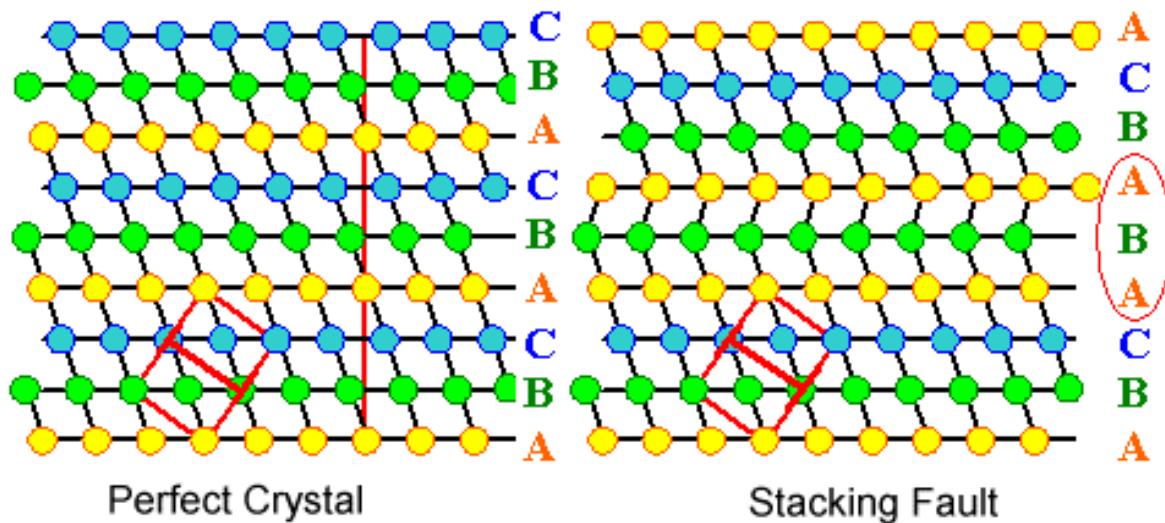
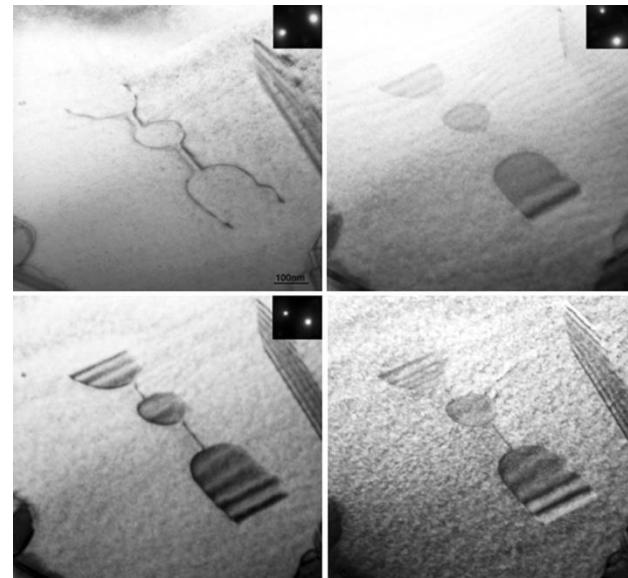
- 외부 표면 (surface)
- 결정립계 (grain boundary)
- 상계면 (phase boundary)
- 쌍정립계 (twinning)
- 적층 결함 (stacking faults)



Stacking Faults

□ 계면 결함 (다른 결정 구조, 또는 다른 결정 방향을 분리하는 경계면)

- 외부 표면 (surface)
- 결정립계 (grain boundary)
- 상계면 (phase boundary)
- 쌍정립계 (twinning)
- 적층 결함 (stacking faults)



<http://faculty.virginia.edu/teamhowe/Gallery/TEM/image5.html>



부피/체적 결함

- Void
- Crack
- (unwanted) 2nd phase

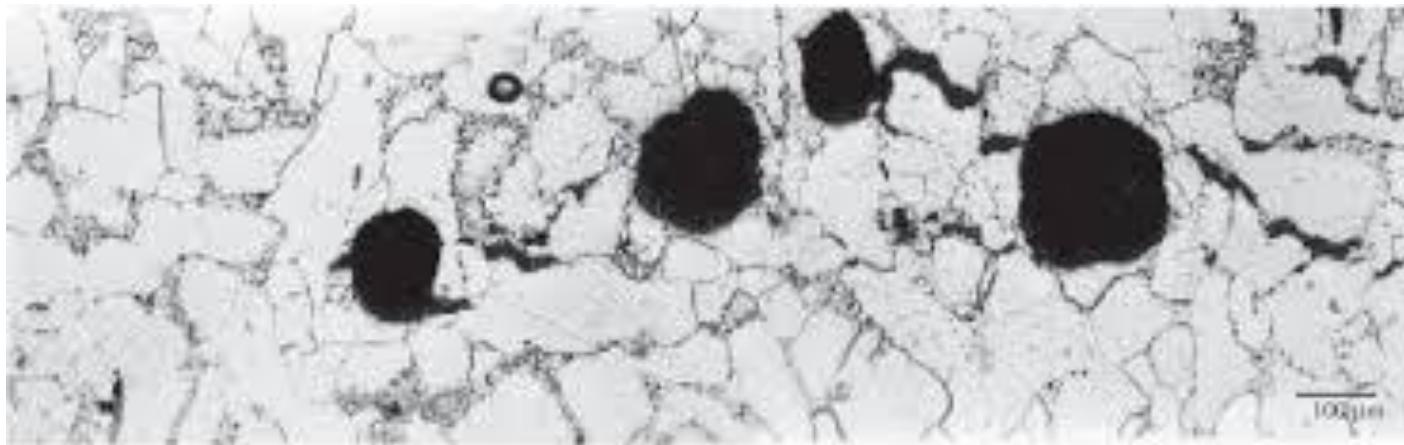
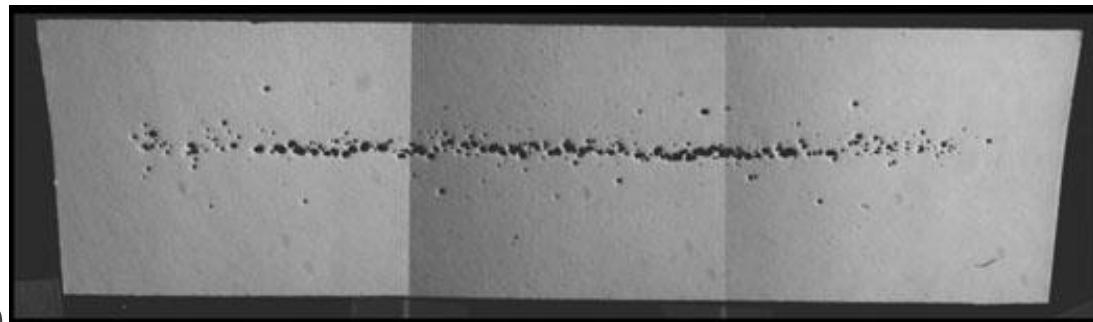


Figure 5. Voids formed by methane in a carbon steel exposed to a hydrogen atmosphere at high temperature. The carbides in the pearlite have been eliminated by reaction with the hydrogen. Photograph courtesy of T.L. da Silveira.

<https://str.llnl.gov/str/JulAug02/Becker.html>

http://www.scielo.br/scielo.php?script=sci_arttext&pid=S1516-14392004000100015



현미경 관찰

□ 기초 원리, 용어

- 거시적 (macroscopic): 육안으로 식별할 수 있을 정도로 큰 스케일
- 미시적 (microscopic): 현미경 (microscope)을 사용해야 식별 가능한 작은 스케일
- micron: 10^{-6} m (마이크론, 마이크로미터, micro meter)
- Microstructure (미세조직): 현미경을 사용해야 식별 가능한 재료의 구조
 - ❖ Grain, texture, 석출물 등등.

□ 다결정 재료의 성질에 영향을 주는 미세조직?

- Grain size
- Texture – grain의 방위
- Grain의 형태
- 석출물

□ 미세조직은 현미경(microscope)을 사용하여 관찰 가능하다.

□ 현미경의 종류

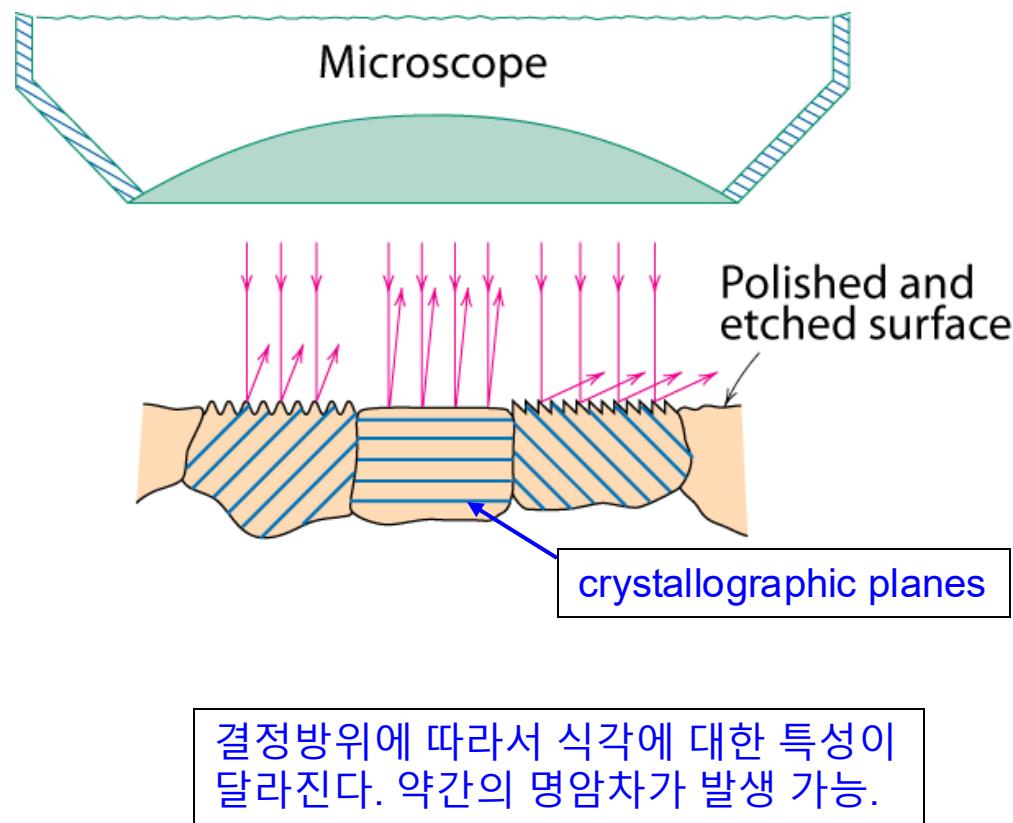
- 광학 현미경
- 전자 현미경
- 주사탐침 현미경

□ 현미경 관찰 법 (microscopy)

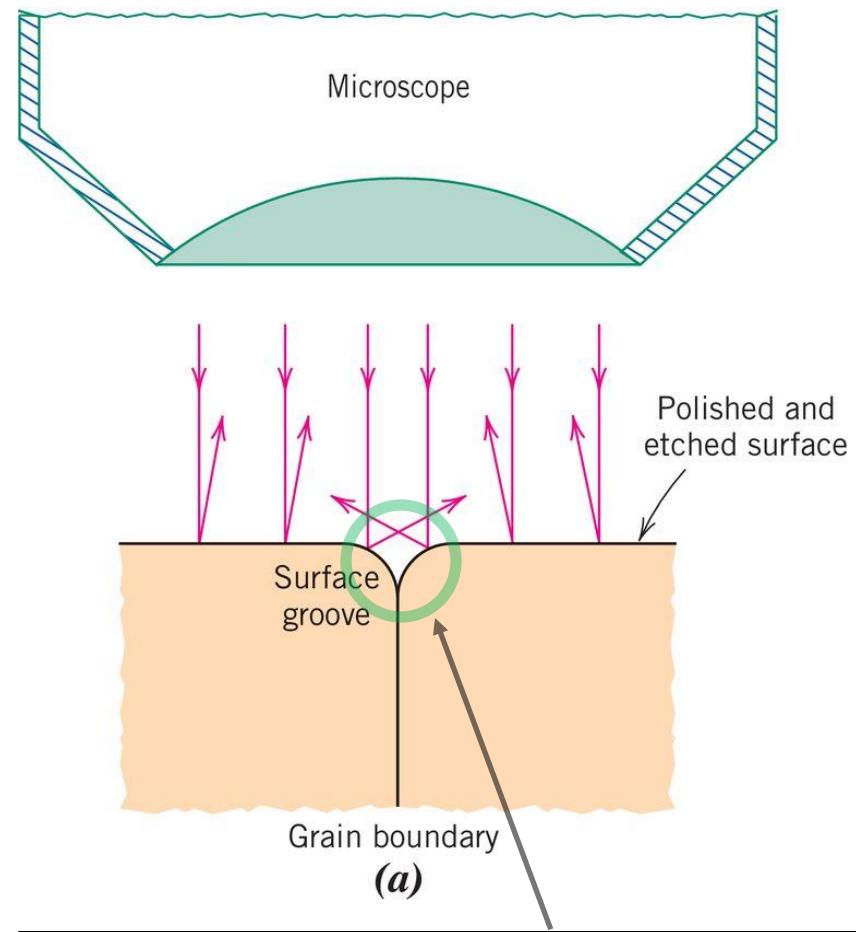


광학현미경 관찰법 (optical microscopy)

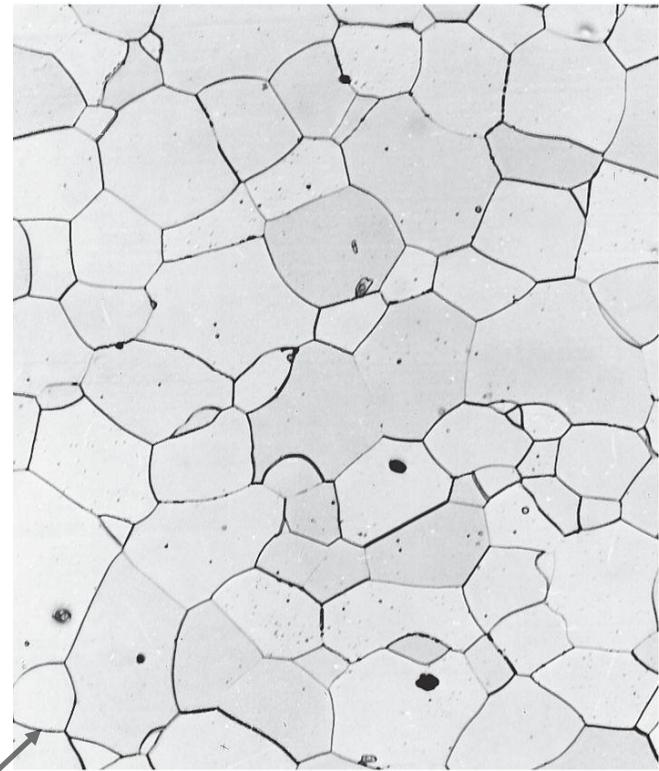
- 가시 광선 (광원) 사용; 조명 장치; 반사 방식
- 반사의 ‘명암’으로 미세 조직 판단
- **반사 명암은 반사도의 차이에 의해 나타난다.**
- Useful up to 2000X magnification.
- 세밀한 시편 준비가 필요 (metallography; 금상학):
 - Polishing (연마) removes surface features (e.g., scratches)
 - Etching (식각) changes reflectance, depending on crystal orientation.



광학현미경 관찰법



Etching은 결합이 완벽하지 않은 결정립계에
존재하는 원자들에게서 더 빠르게 진행

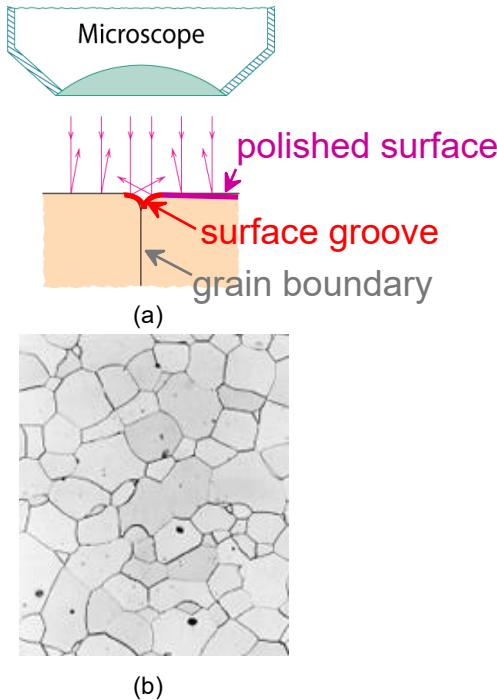


Photomicrograph courtesy of L. C. Smith and C. Brady, the National Bureau of Standards, Washington, DC (now the National Institute of Standards and Technology, Gaithersburg, MD.)



Optical Microscopy

광학현미경으로 결정립의 크기를 관찰 할 수 있다.
다결정 금속의 결정립의 정량화 시킬 수 있을까?



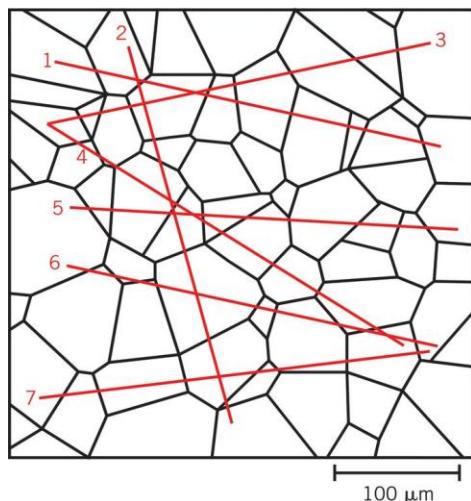
결정립의 ‘평균’ 크기를 사용한다. 결정립의 평균 크기를 측정하는 방법은...

1) 선 교차법

: 임의의 직선과 교차하는 결정립계의 수를 집계

2) 비교법

: 결정립 면적에 근거한 표준 차트의 경정립 구조와 비교.



$$\bar{l} = \frac{L_T}{PM}$$

L_T : 모든 선 길이 합

P : 교차의 총개수

M : 현미경 배율

\bar{l} : 결정립 평균 직경

Fig. 6.20(a) & (b), Callister & Rethwisch 9e.
[Fig. 6.20(b) is courtesy of L.C. Smith and C. Brady,
the National Bureau of Standards, Washington, DC (now the
National Institute of Standards and Technology,
Gaithersburg, MD).]



Microscopy

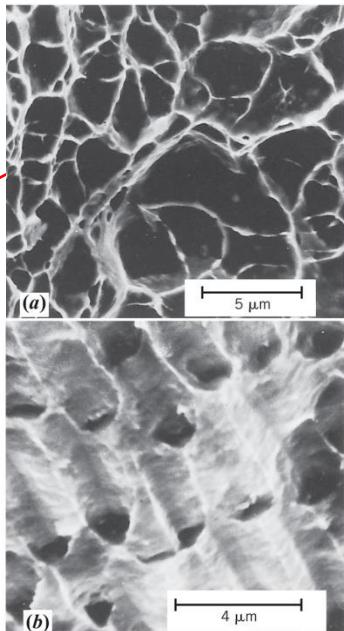
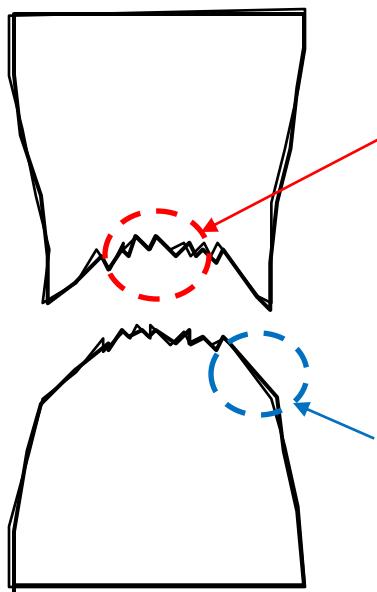
Optical resolution ca. 10^{-7} m = 0.1 μm = 100 nm

For higher resolution need higher frequency

- 광학현미경의 최대 배율은 2000배.
- 재료 내부의 구조적 요소들이 더욱 미세할 경우 관찰 불가능
- 전자 현미경?
 - 전자의 wavelengths: 3 pm (0.003 nm)
 - ❖ (Magnification - 1,000,000X)
 - Atomic resolution possible
 - Electron beam focused by magnetic lenses.
- 전자 현미경 방식
 - 투과 (transmission); Transmission Electron Microscope (TEM)
 - 주사 (scanning); Scanning Electron Microscope (SEM)
 - 주사 탐침 (scanning probe); Scanning Probe Microscope



SEM: application to fractography



섬유질상

Ductile fracture under 'uniaxial tension'

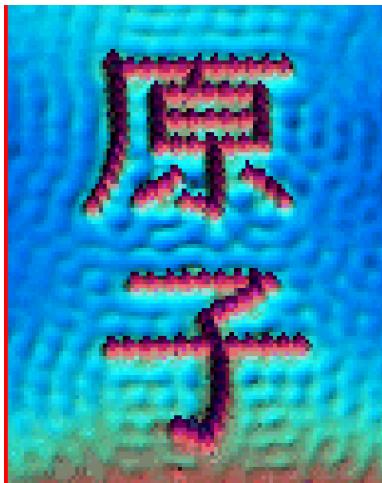
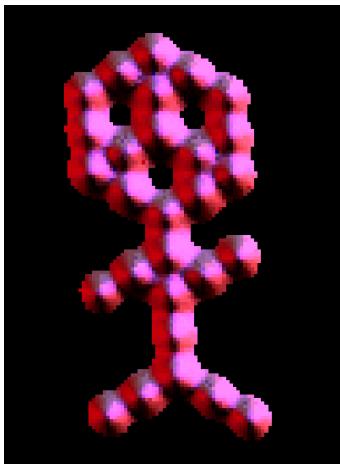
Ductile fracture under 'shear'

한쪽으로 늘어난 모양



Scanning Tunneling Microscopy (STM)

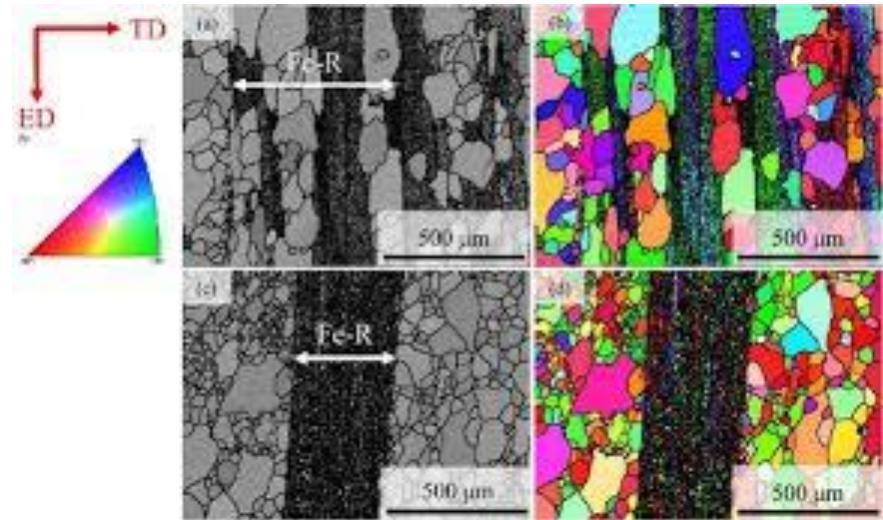
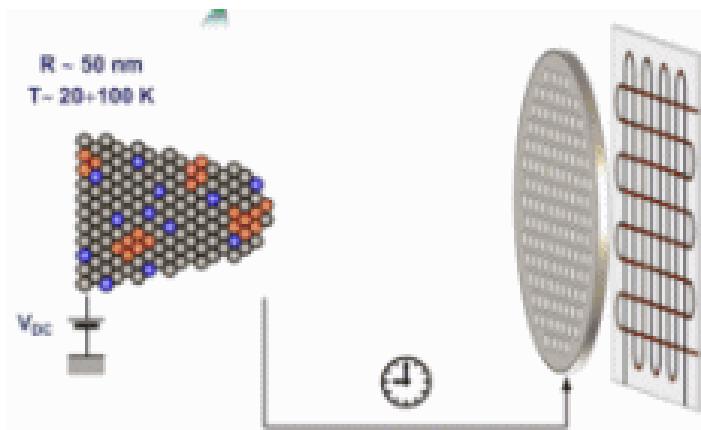
- Atoms can be arranged and imaged!



Photos produced from the work of C.P. Lutz, Zeppenfeld, and D.M. Eigler. Reprinted with permission from International Business Machines Corporation, copyright 1995.



최신 기법



Tomographic Atom Probe

Electron Back-Scattered
Diffraction

