2009 spring

Microstructural Characterization of Materials

03.04.2009

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Contents for previous class

Microstructure: structure inside a material

that could be observed with the aid of a microscope

Observation of Microstructure: to make image

from the collection of defects in the materials

→ **OM, SEM, TEM, AFM, SPM**

Properties

- Strength
- Toughness
- Formability
- Conductivity
- Corrosion Resistance
- Piezoelectric strain
- Dielectric constant
- Magnetic Permeability

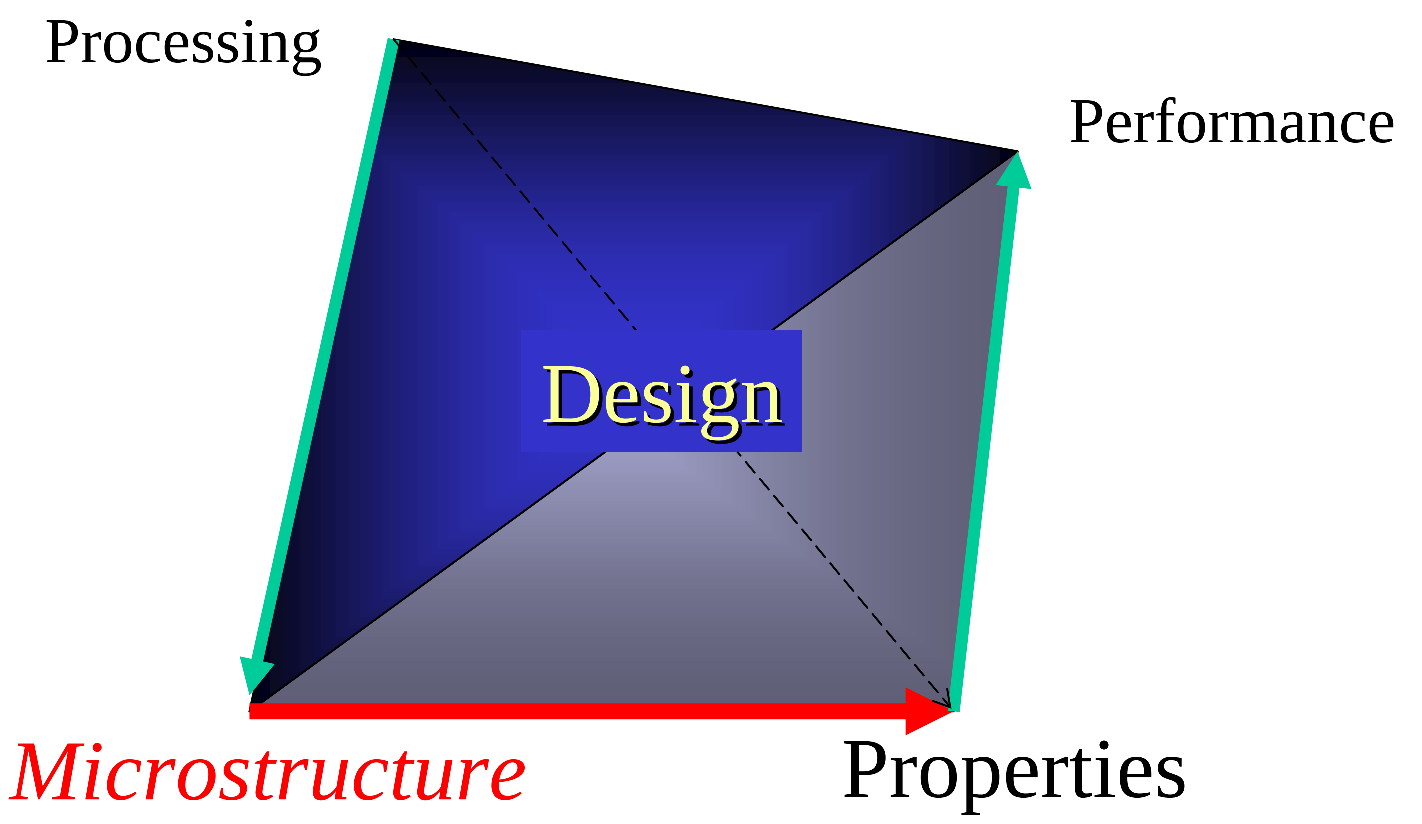
↔

Microstructural Parameters

- Grain size
- Grain shape
- Phase structure
- Composite structure
- Chemical composition (alloying)
- Crystal structure
- Defect structure (e.g. porosity)

Contents for previous class

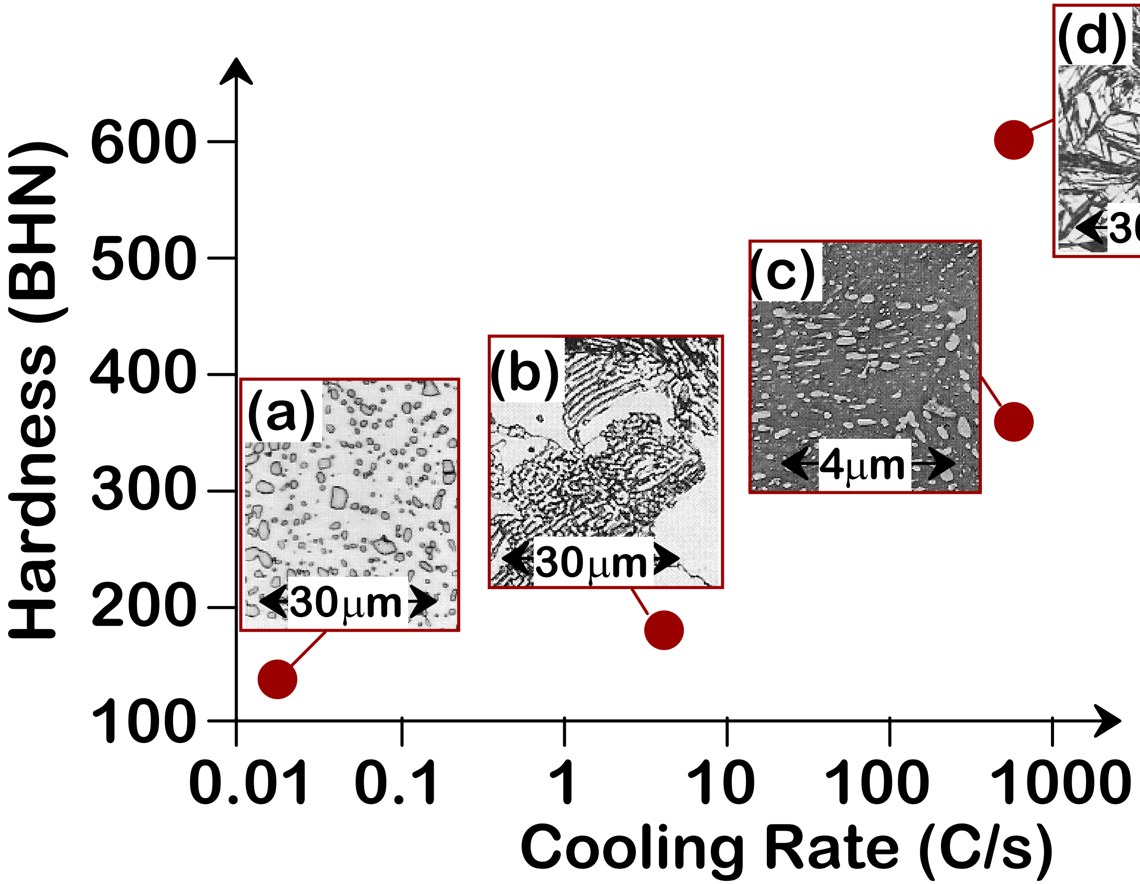
“Tailor-made Materials Design”



optimization of material properties through control of microstructure

Structure, Processing, & Properties

- Properties depend on structure
  ex: hardness vs structure of steel



Data obtained from Figs. 10.21(a) and 10.23 with 4wt%C composition, and from Fig. 11.13 and associated discussion, *Callister 6e*. Micrographs adapted from (a) Fig. 10.10; (b) Fig. 9.27;(c) Fig. 10.24; and (d) Fig. 10.12, *Callister 6e*.

- Processing can change structure
  ex: structure vs cooling rate of steel

Contents for today’s class

- *Length Scale of Microstructure*

1) Effect of atomistic length scale on material’s properties

including brief introduction for microstructural observation methods

-

2) Effect of defect structure on material’s properties

including brief explanation of polycrystals and crystal orientation

Length Scale of Microstructure

Structure

Nanostructure 0.1-100 nm

Sub-atomic Electronic	Atomic	Microscopic	Bulk
<0.1 nm	0.1-10 nm	1-1000 μm	>1 mm
1 nm = 4x10^{-8} in	HRTEM, STEM AFM	Optical Microscope SEM,TEM, XRD	Naked Eye

Length Scales of Microstructure

- **Many important intrinsic material properties are determined at the *atomistic length scale*.**

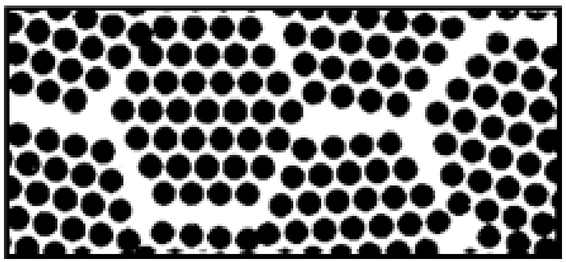
- **The Properties of materials are, how, often strongly affected by the *defect structure*. For example, polycrystals have different properties than single crystals just because of the variation of crystal orientation, combined with the anisotropy of the property. This immediately introduces the idea that the behavior of a material can vary from on location to another.**

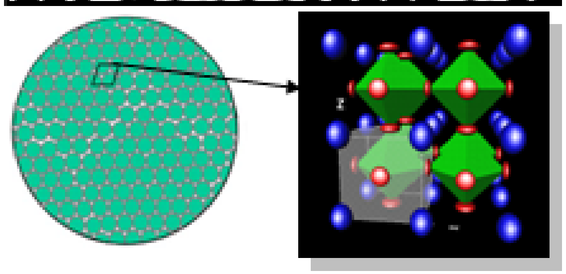
- **Amorphous – from the Greek for “without form”**

  not to materials that have no shape,

  but rather to materials with no particular structure

Crystals

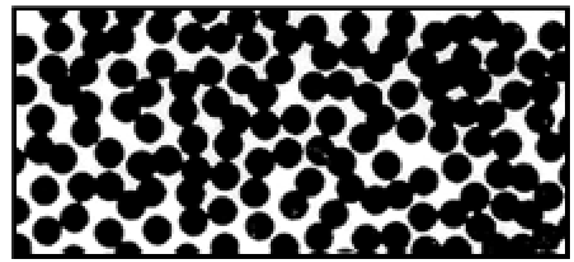


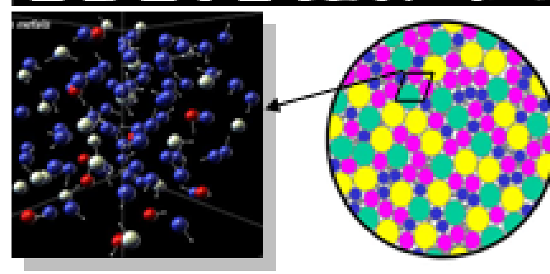


Building block: arranged in orderly,
3-dimensional,
periodic array

- **grain boundaries**

Liquids, glasses

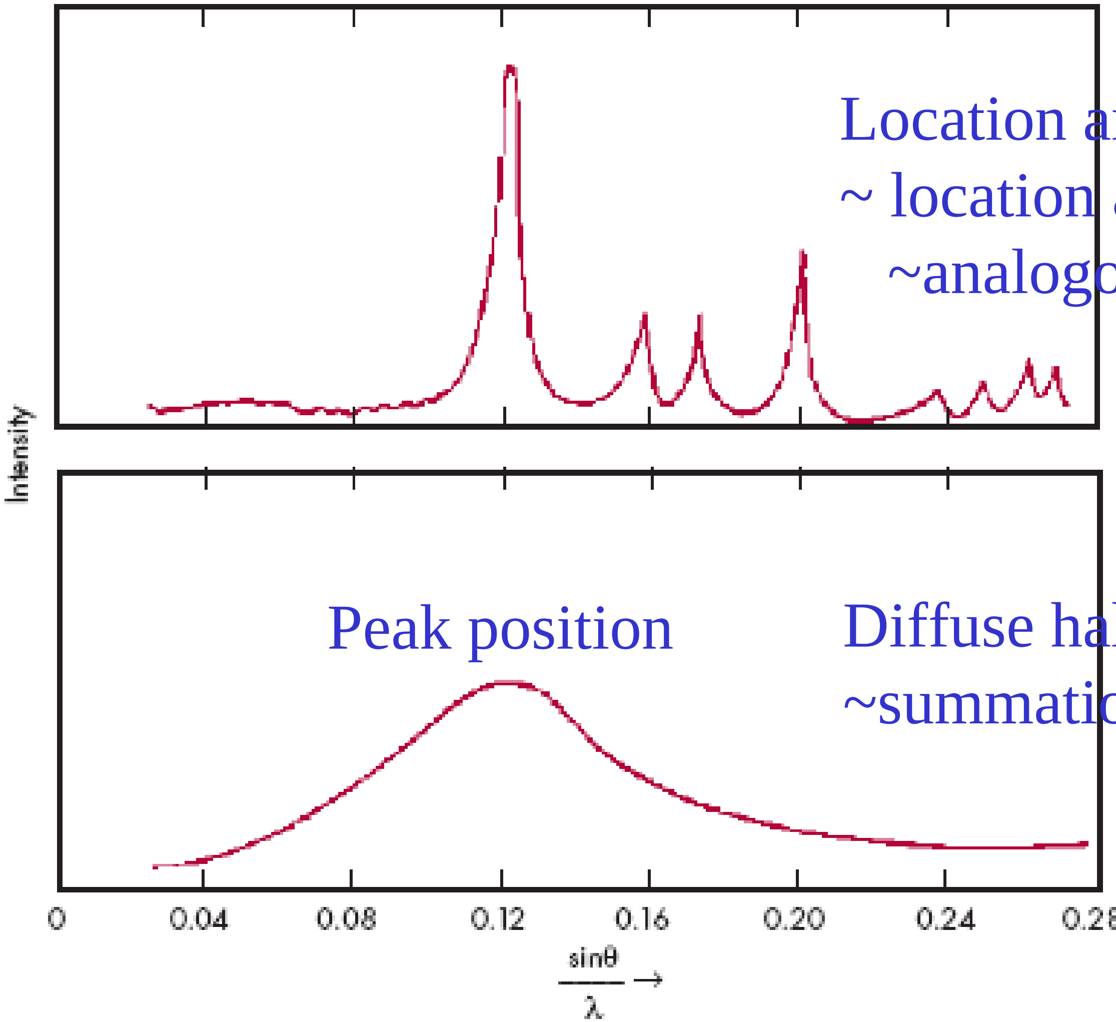




nearly random = non-periodic

- **no grain boundaries**

X-ray diffraction results



Location and intensity of peaks
~ location and arrangement of atoms
~analogous to a fingerprint

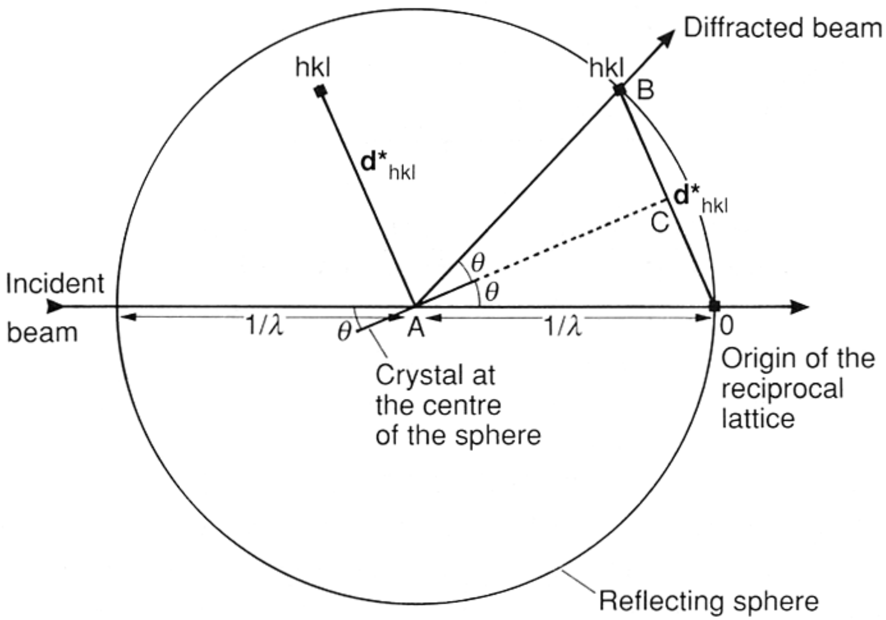
Peak position

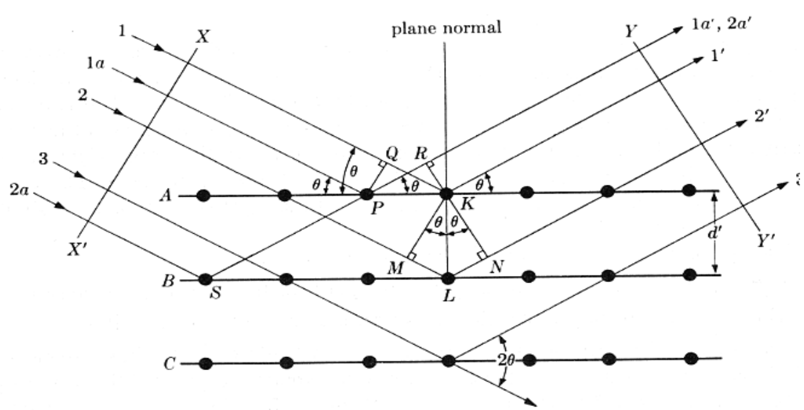
Diffuse halo pattern
~summation of interference

Figure 3. Characteristic Diffraction Patterns from Crystalline Material (Top) and Amorphous Material (Bottom). [3]

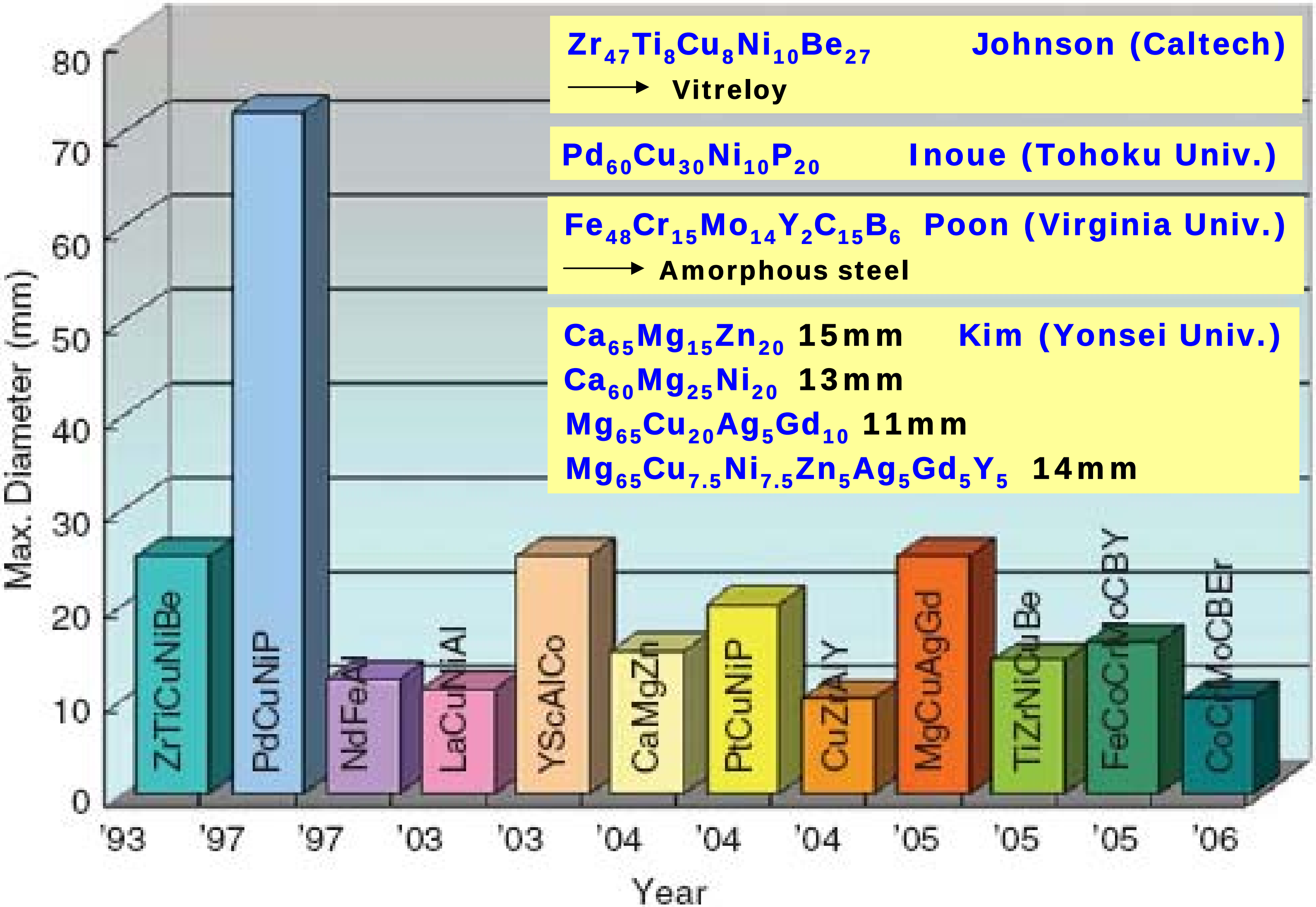
XRD (X-ray Diffraction)

X선을 결정에 부딪히게 하면 그 중 일부는 회절을 일으키고 그 회절각과 강도는 물질구조상 고유한 것으로서 이 회절 X선을 이용하여 시료에 함유된 결정성 물질의 종류와 양에 관계되는 정보를 알 수 있다.



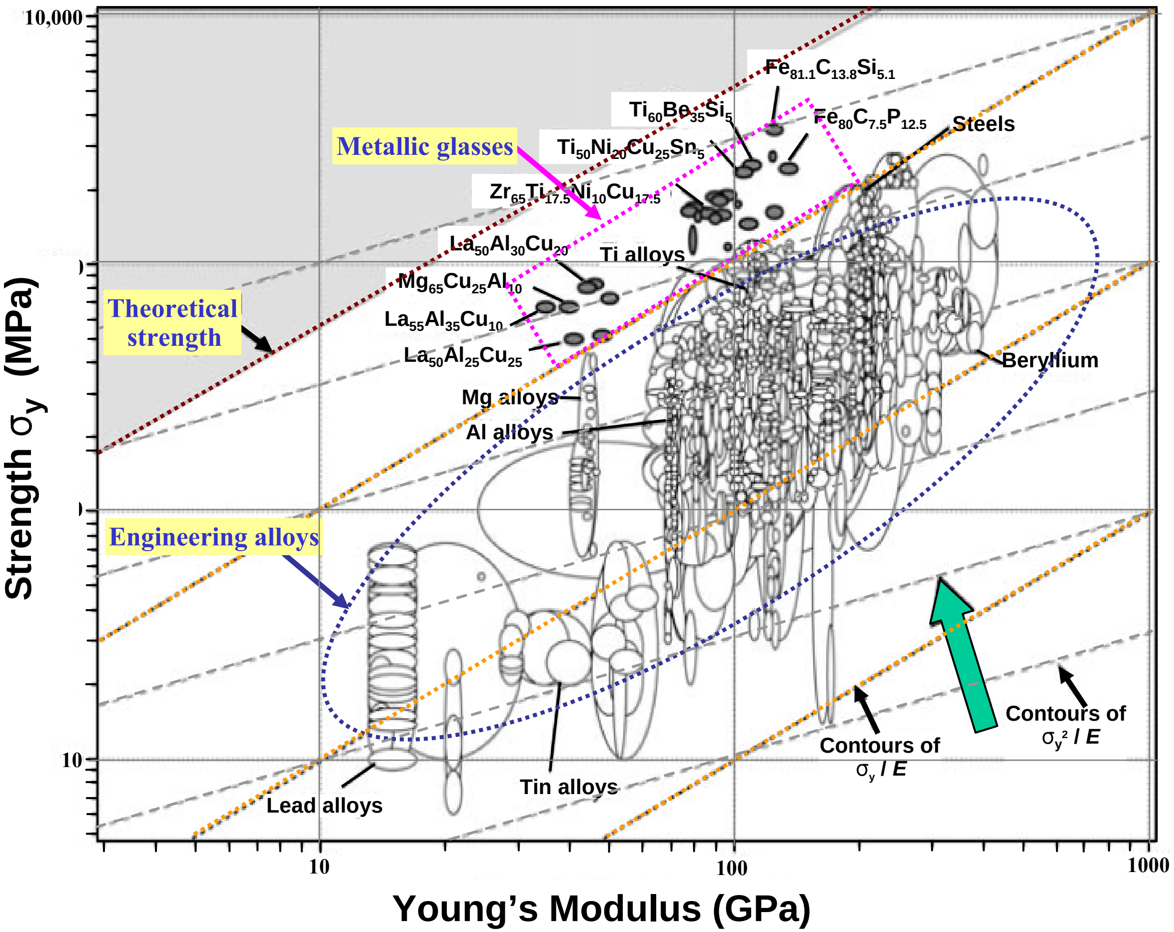


Recent BMGs with critical size ≥ 10 mm



A.L. Greer, E. Ma, MRS Bulletin, 2007; 32: 612.

High strength of BMGs



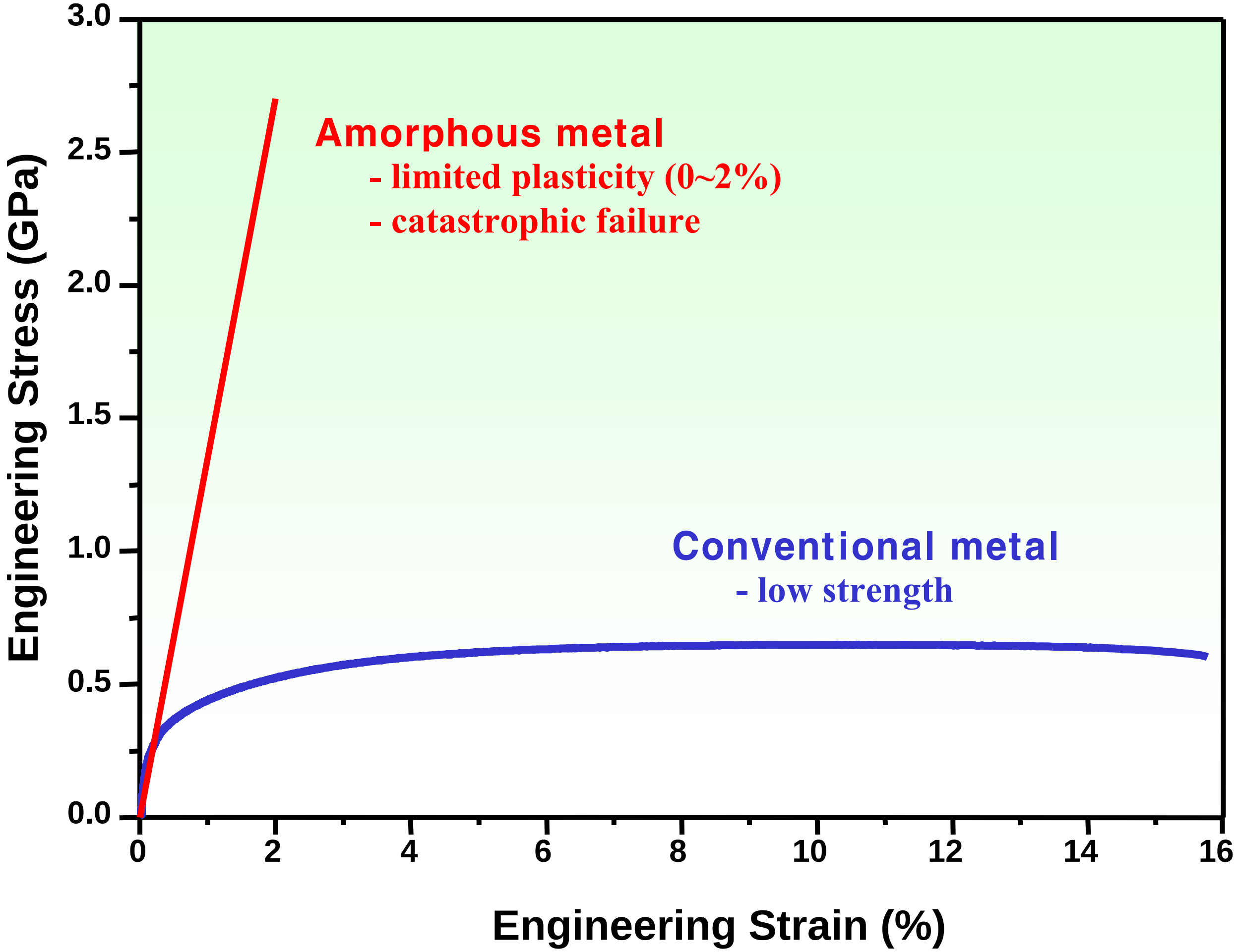
High fracture strength over 5 GPa in Fe-based BMGs

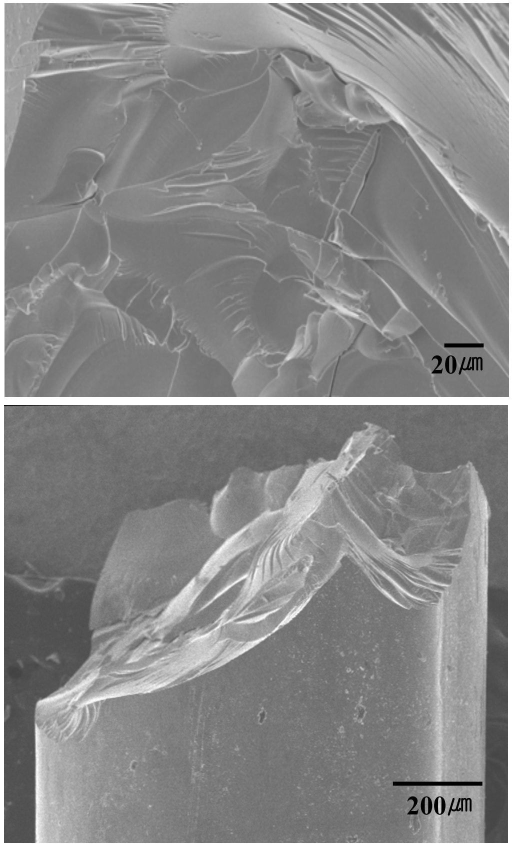
A.L. Greer, E. Ma, MRS Bulletin, 2007; 32: 612.

Limited Plasticity by shear softening and shear band

- Microscopically brittle fracture

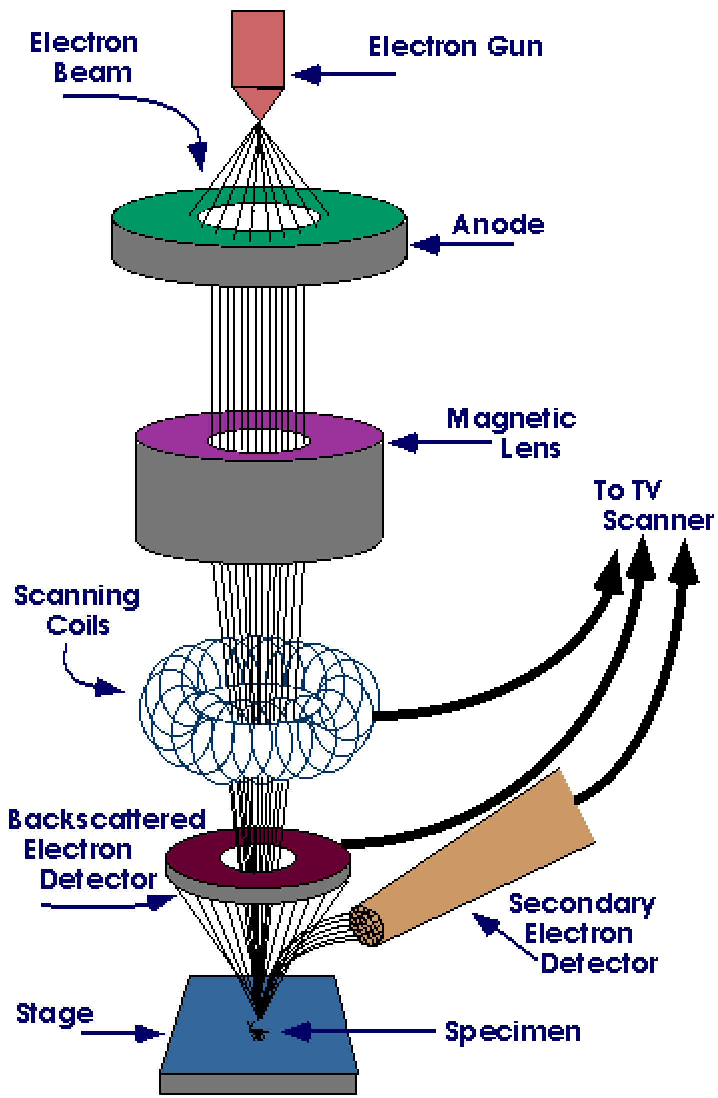
→ Death of a material for structural applications

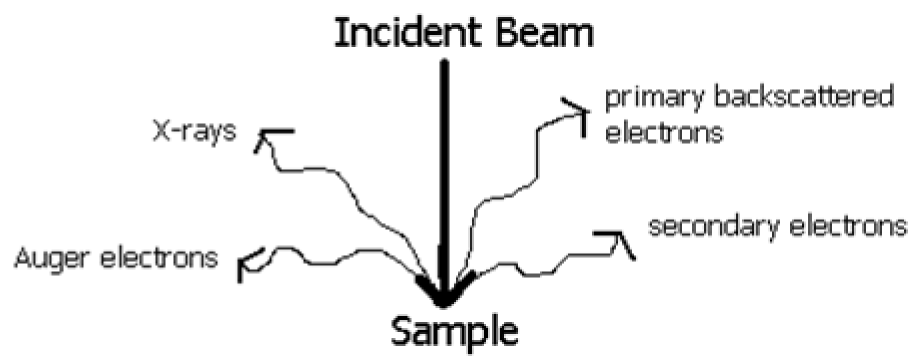




SEM (Scanning Electron Microscopy)

SEM은 Electron beam이 Sample의 표면에 주사하면서 Sample과의 상호작용에 의해 발생된 Secondary Electron를 이용해서 Sample의 표면을 관찰하는 장비이다.





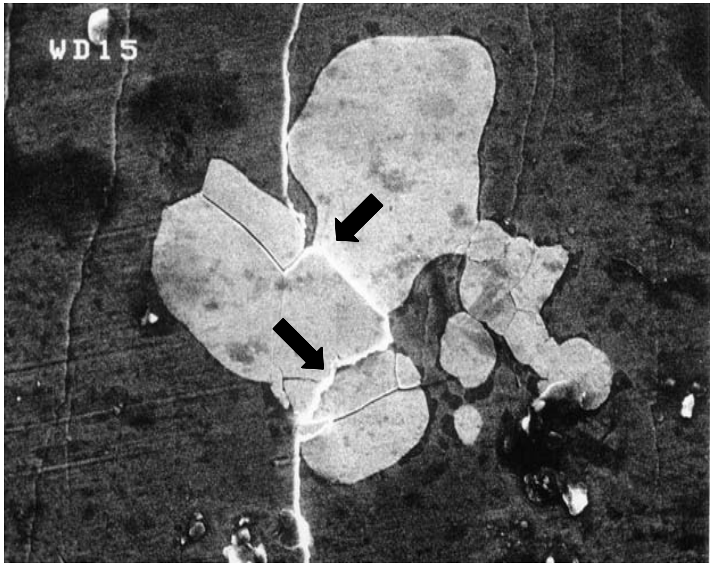
Plastic deformation in metallic glasses

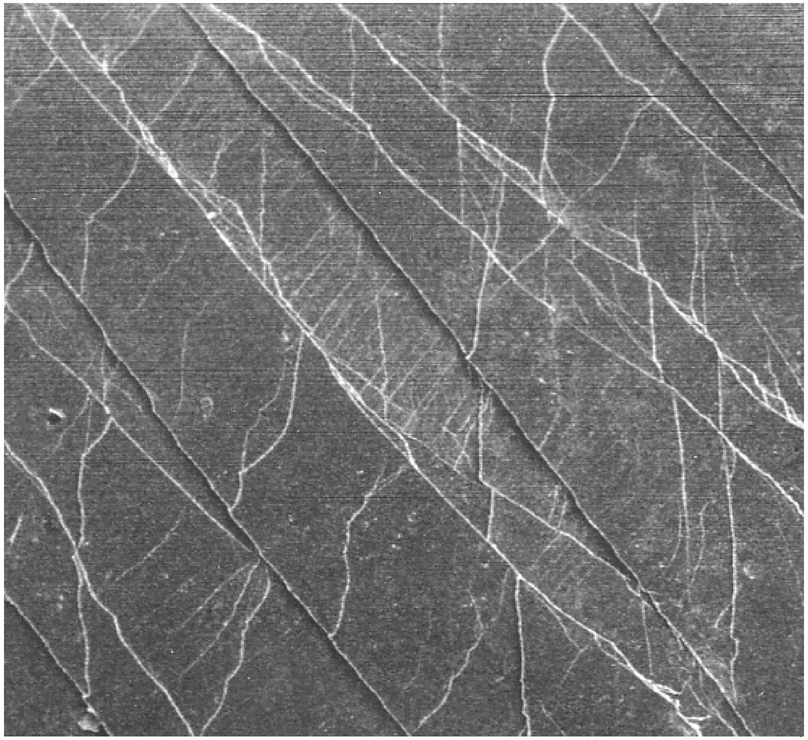
Plastic deformation in metallic glass

- *No* dislocation / *No* slip plane
- Inhomogeneously localized plastic flow in the shear band

interrupt the localization of stress and deformation

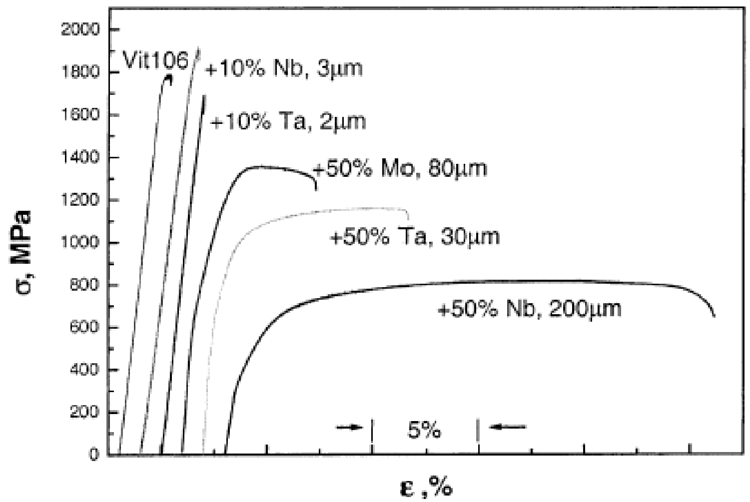
- Prevent propagation of single shear band ⇨ BMG matrix composites
- Multiple shear band formation

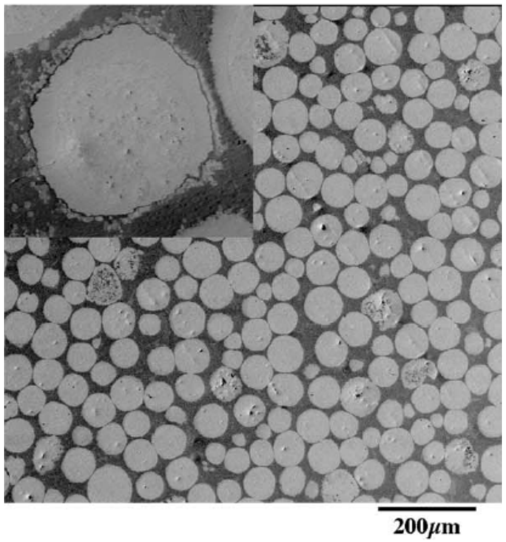




Ex-situ BMG matrix composites

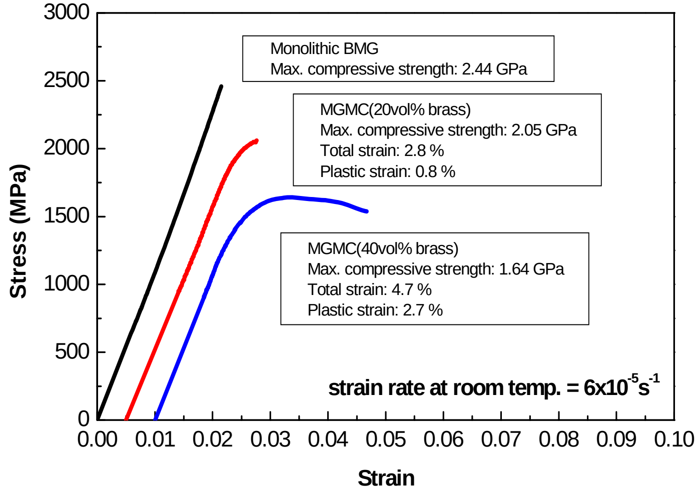
1) Casting : hard/ductile particle

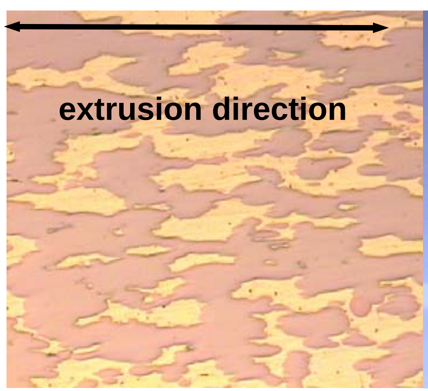


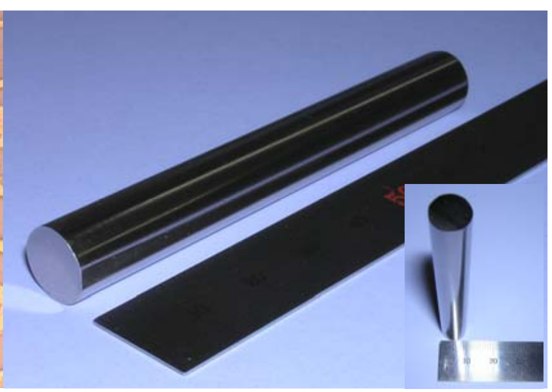


(Johnson et al., Acta Mater.,1999)

2) Extrusion : ductile powder



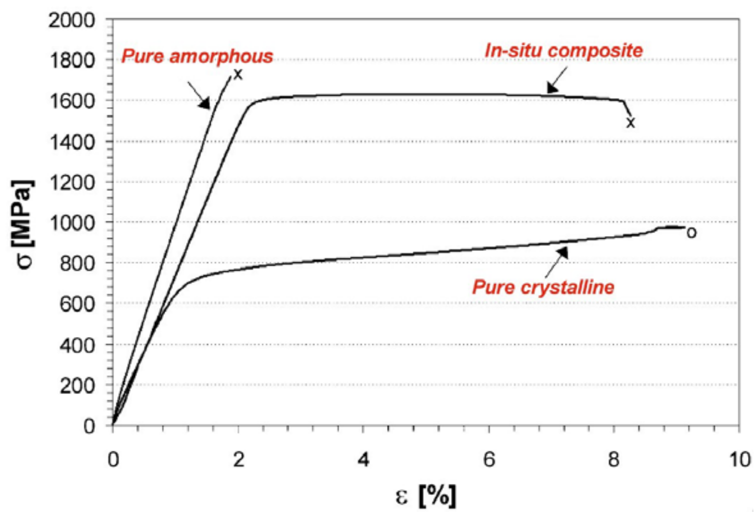


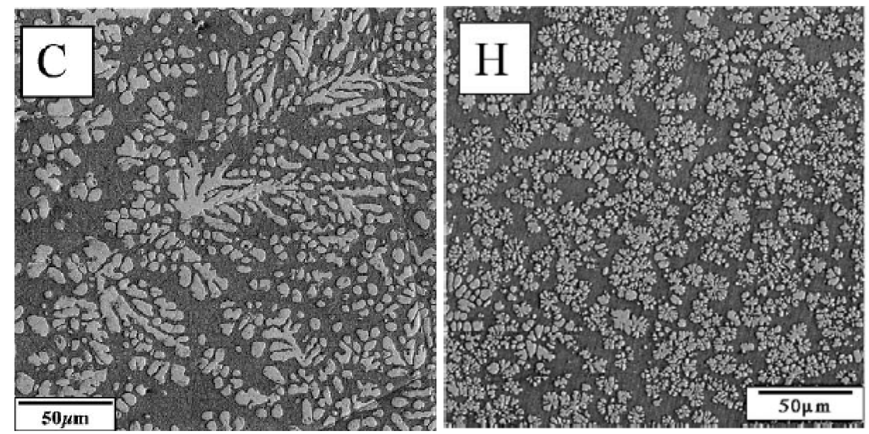


(Kim et al., J. Non-cryst. Solids, 2002)

In-situ BMG matrix composites

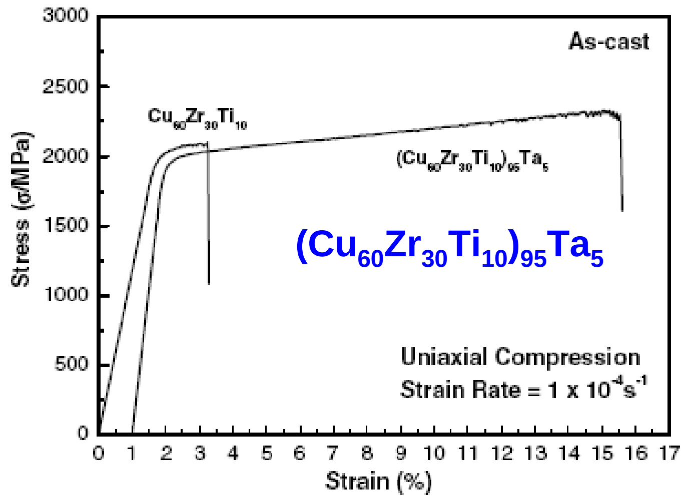
1) Solidification : formation of primary ductile phase

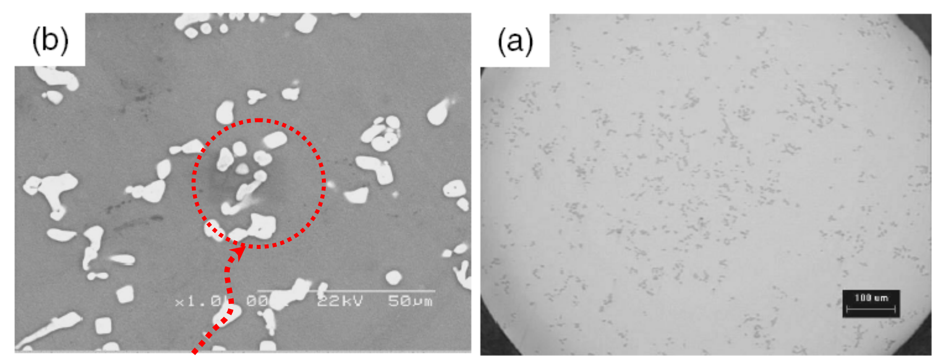




(Johnson et al., Acta Mater., 2001)

2) Solidification : precipitation of ductile phase



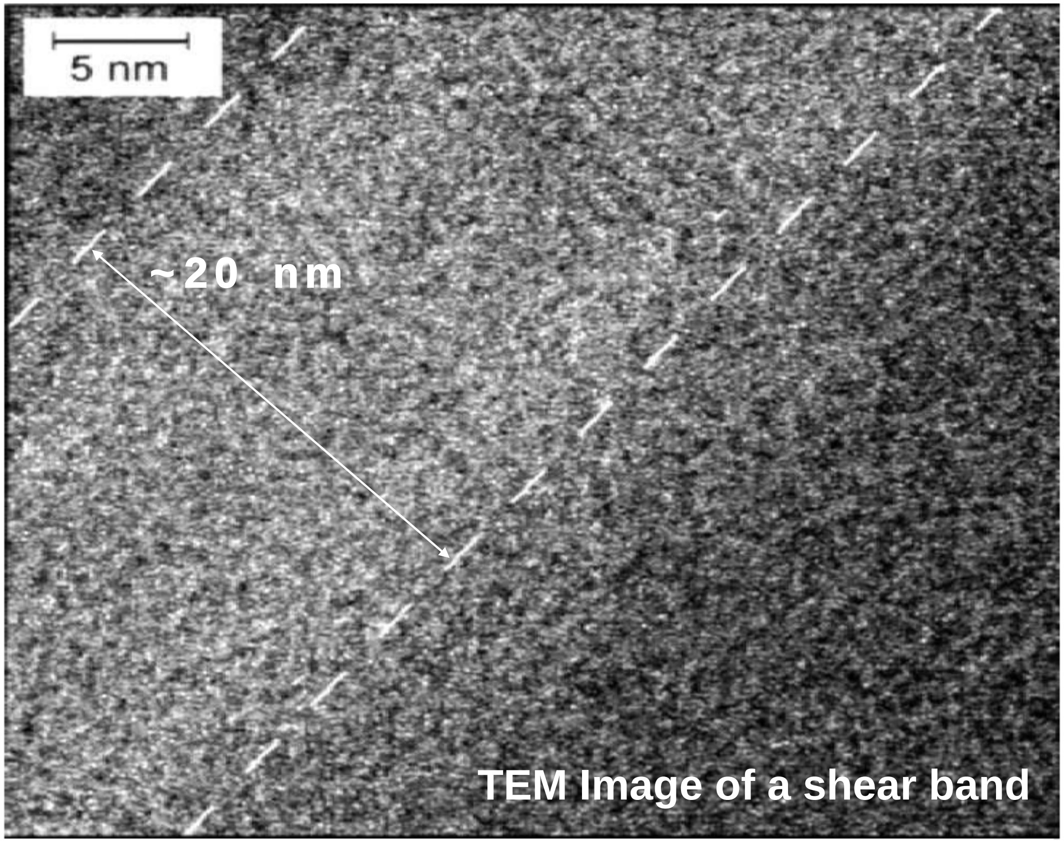


Ta rich particle

(Johnson et al., Acta Mater., 2001)

Size of heterogeneity

Shear bands are ~20 nm in width

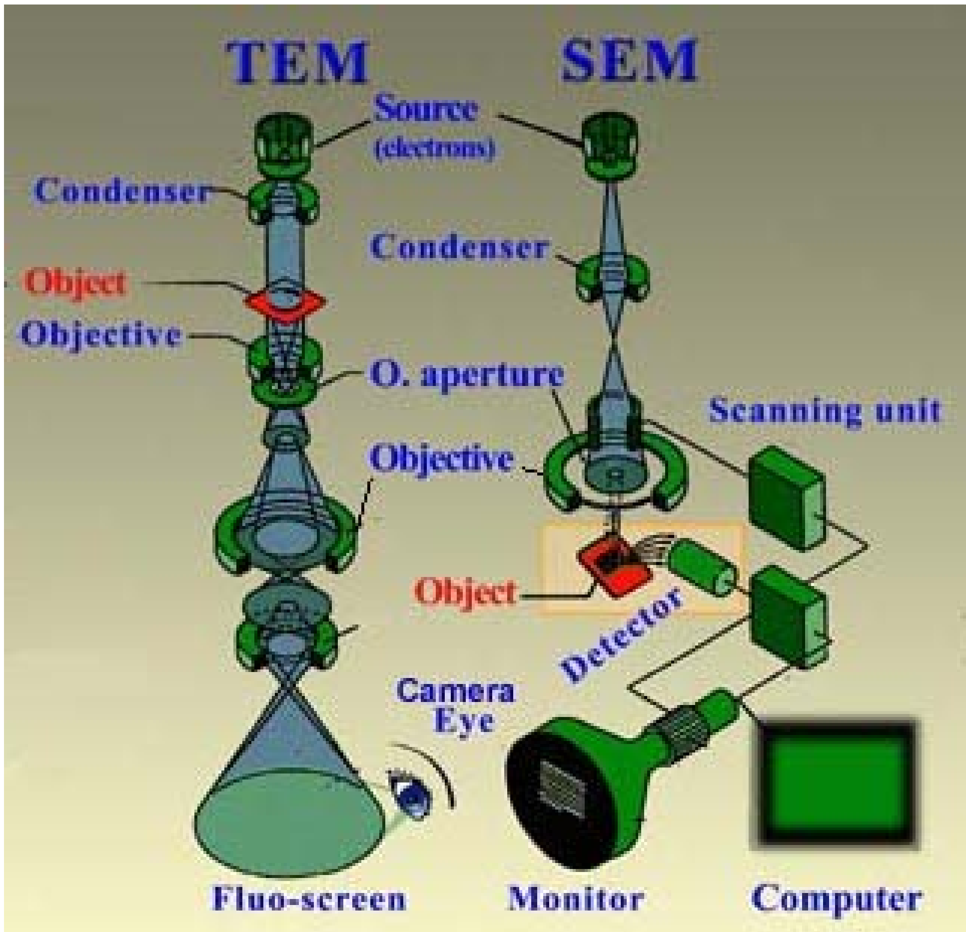


- **Prevent propagation of single shear band**

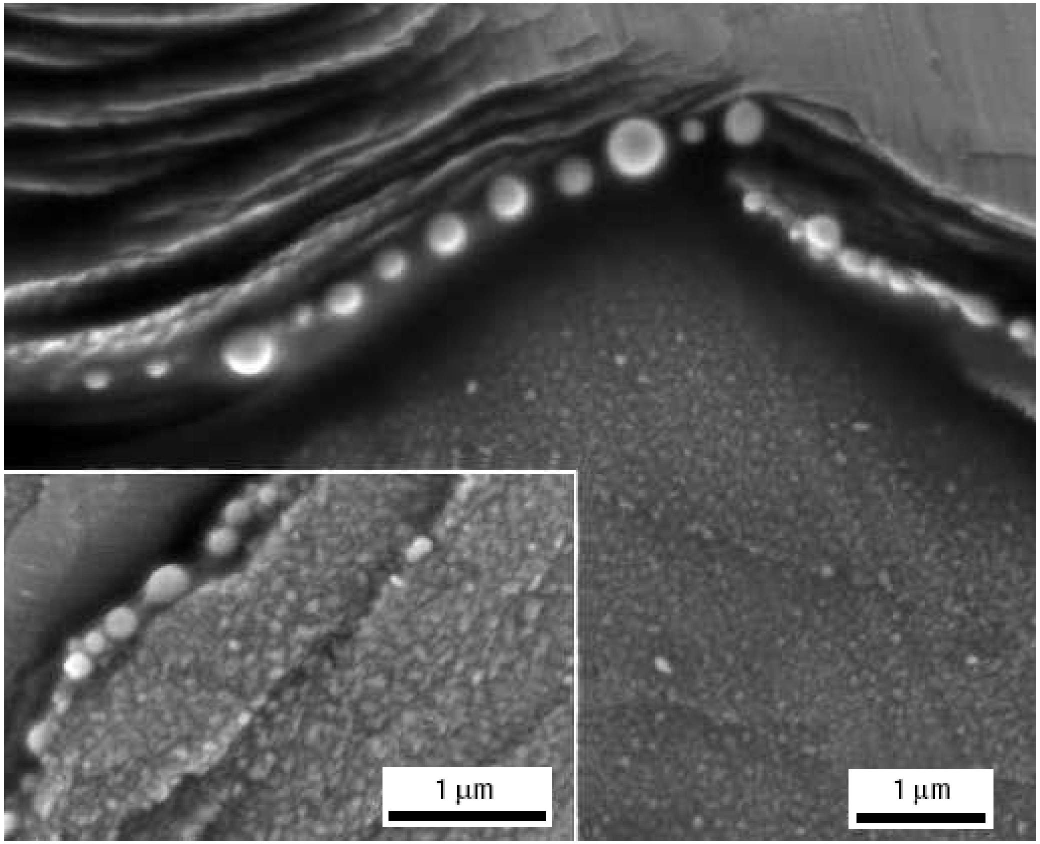
➡ **Micro- or nanometer scale heterogeneity**

TEM (Transmission Electron Microscopy)

TEM은 electron beam이 통과할 수 있도록 ultrathin sections을 만들어 관찰할 수 있도록 하는 기능적 장치로 여러 가지 각각의 시스템으로 구성되어 있다.



Observation of SBs after three point beam bend test



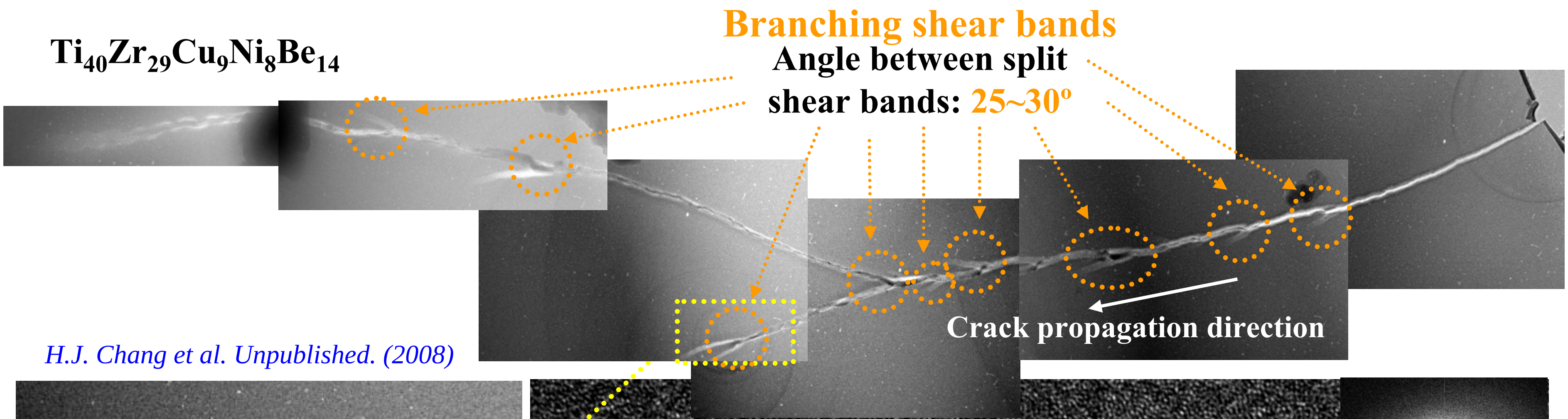
J. J. Lewandowski et al., Nature Mater. 5 (2006) 15

Melting of Sn coating on the surface of Vit. 1 on the compression side

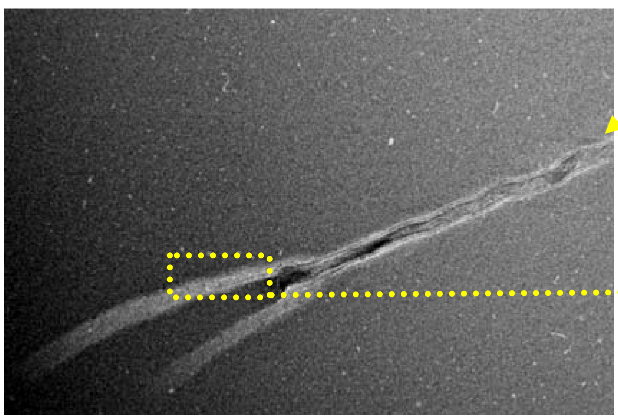
→ evidence for temperature rise ($T_{m, Sn}$ = 232℃)

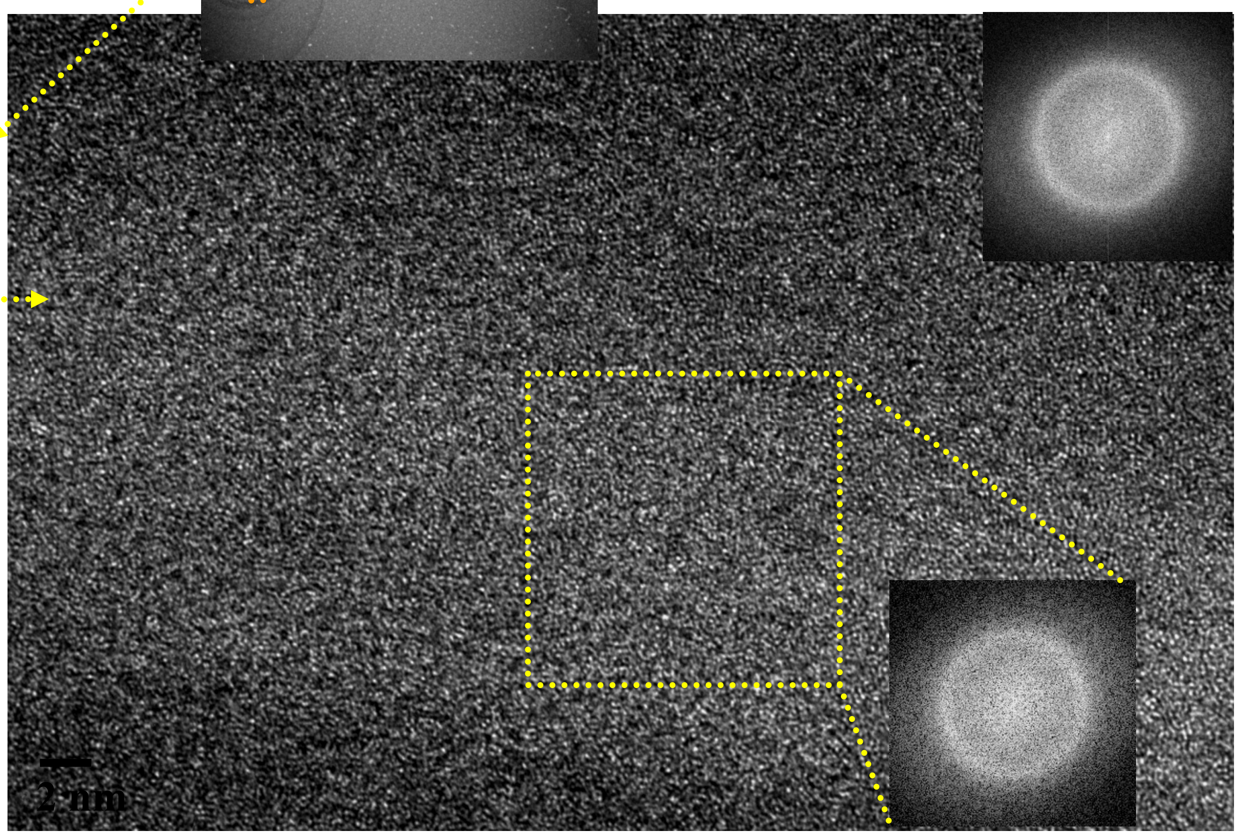
Observation of shear bands after tensile test

$Ti_{40}Zr_{29}Cu_9Ni_8Be_{14}$



H.J. Chang et al. Unpublished. (2008)

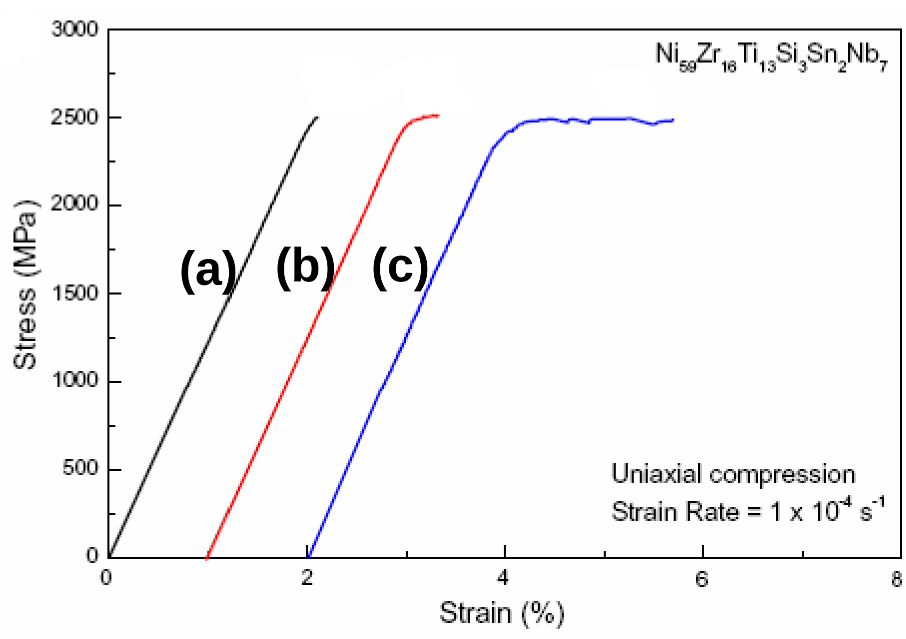


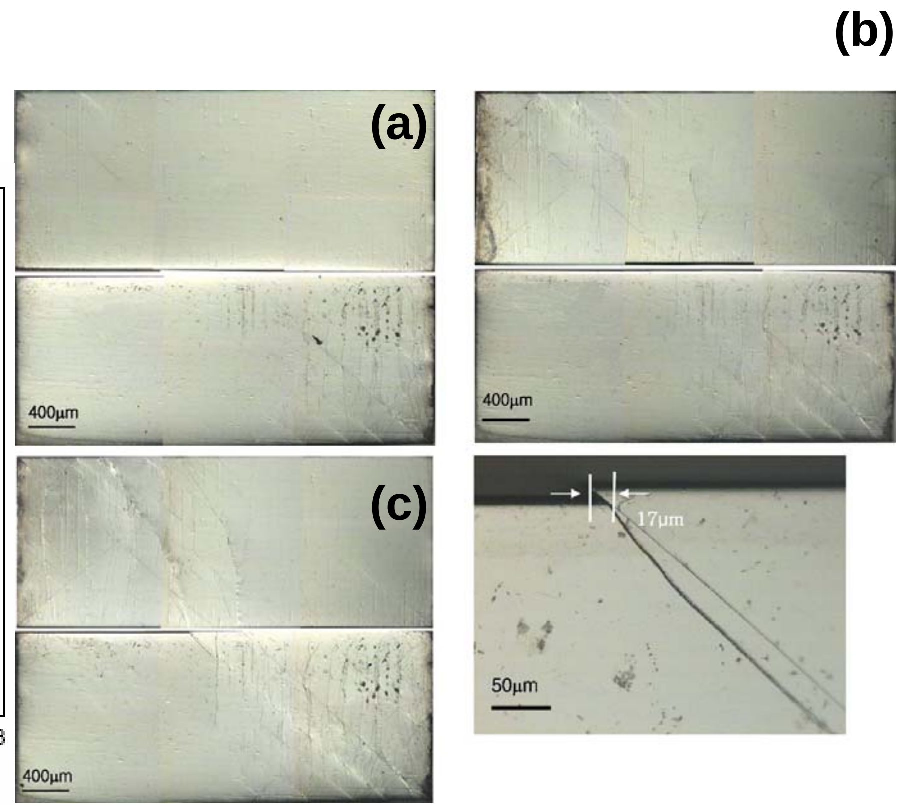


- **Width of shear band : 10 nm**
- **No crystallization in the shear band**

OM images for interruptions during compression

- **Stress–strain curves and related OM images for each interruptions during compression test**

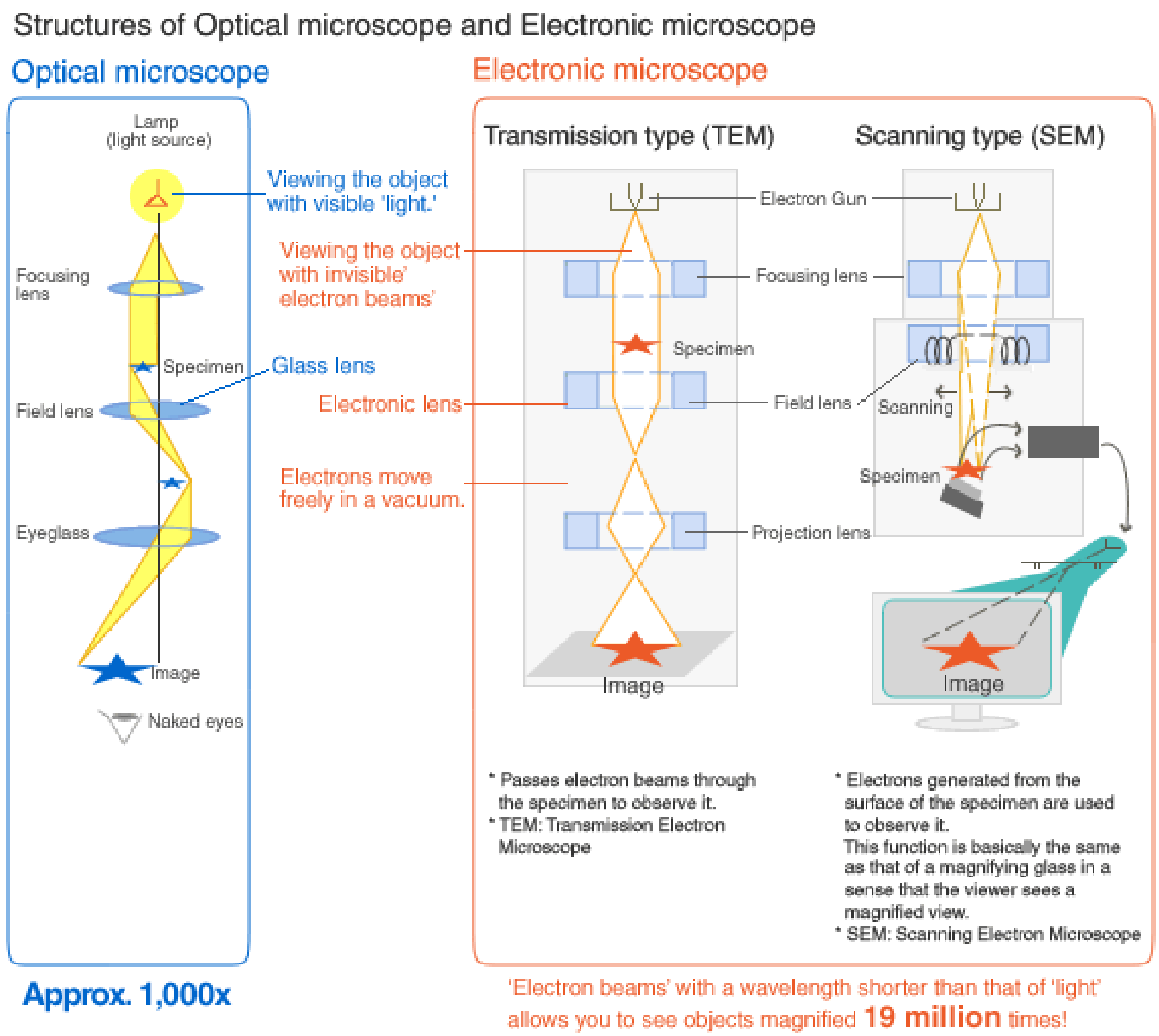




J. Y. Lee et al. Acta Mater. 54 (2006) 5271-5279

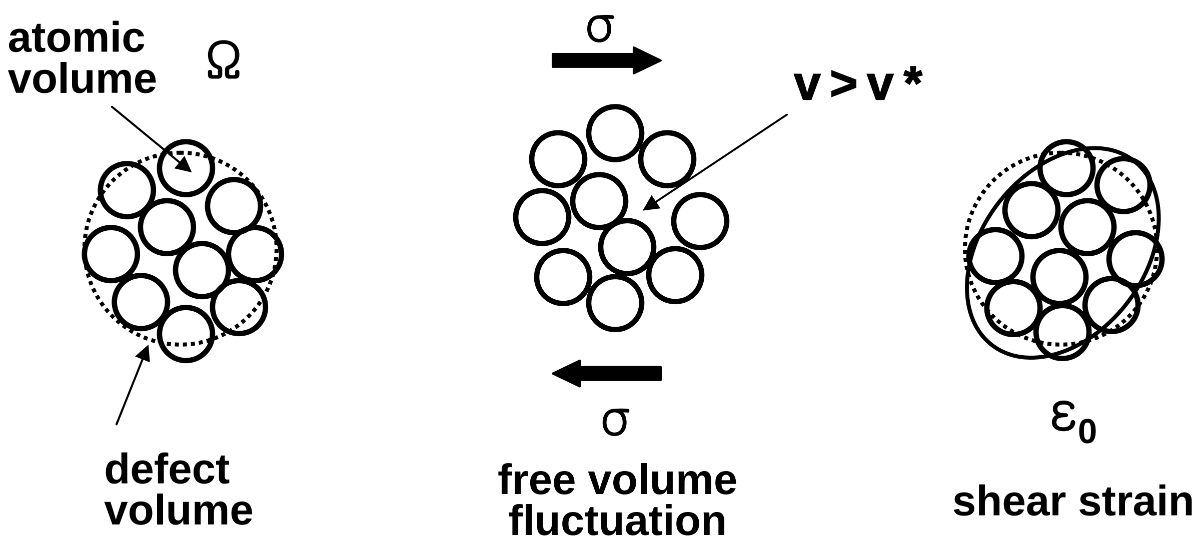
Optical Microscope

OM과 TEM은 기본적인 구성 즉 렌즈의 배열은 같으나 렌즈를 무엇을 사용하느냐 하는 차이 이다. OM은 유리(glass)를 EM은 magnetic lens를 사용한다. 광원은 OM이 시광을 EM이 전자(빔)를 사용하므로 전자현미경은 칼라 상을 볼 수 없는 것이다.



Size of heterogeneity

- Elementary flow event in an metallic glasses

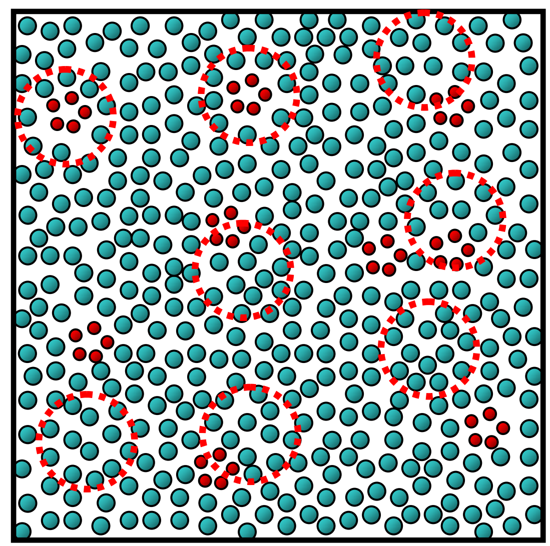


Probability $\exp(A\frac{v^*}{v_f})$

average free volume

Flow governed by localized defect (~10 atoms) and creates defects

atomic scale heterogeneity



$$\eta = \eta_0 \exp\left\{A\frac{V_0}{V_f}\right\}$$

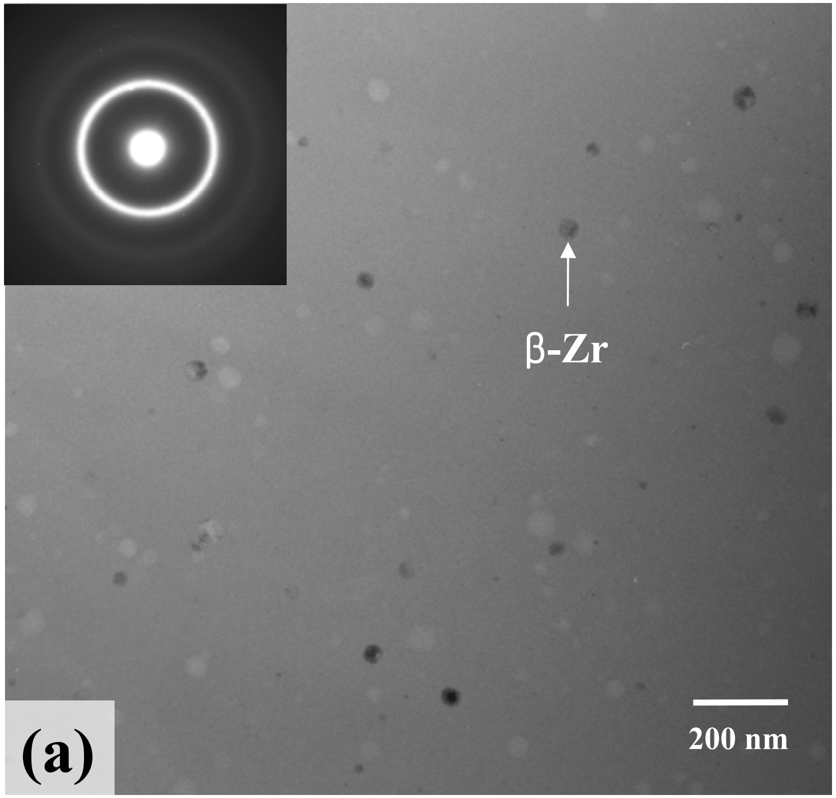
Enhancement plasticity in BMGs with atomic scale heterogeneity

Effect of quenched in quasicrystal nuclei

Effect of quenched-in quasicrystal nuclei

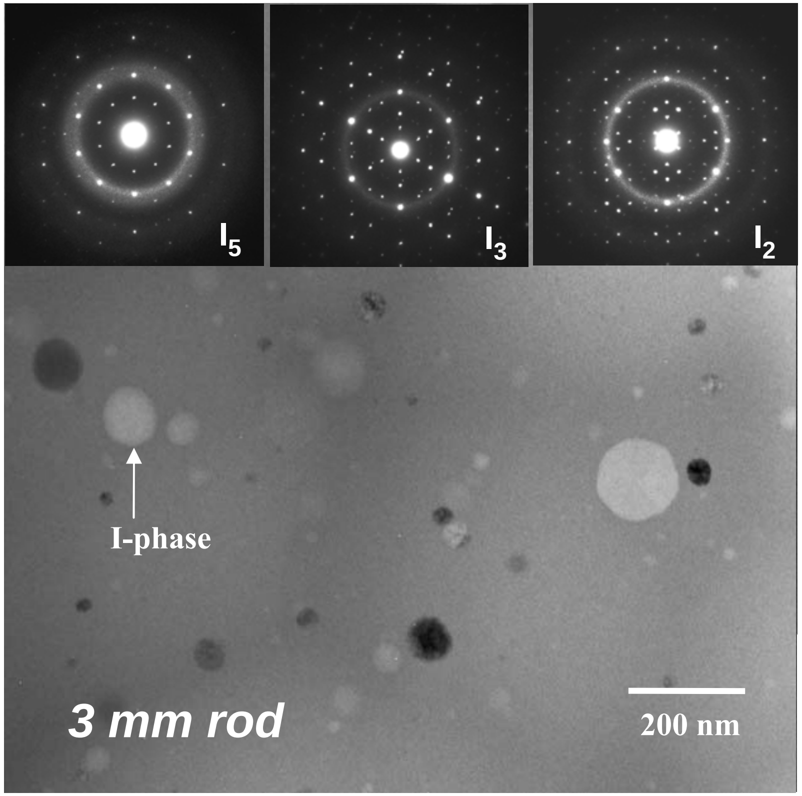
2 mm rod

(a) $Zr_{63}Ti_5Nb_2Cu_{15.8}Ni_{6.3}Al_{7.9}$



β-Zr particle(~70 nm) in amorphous matrix

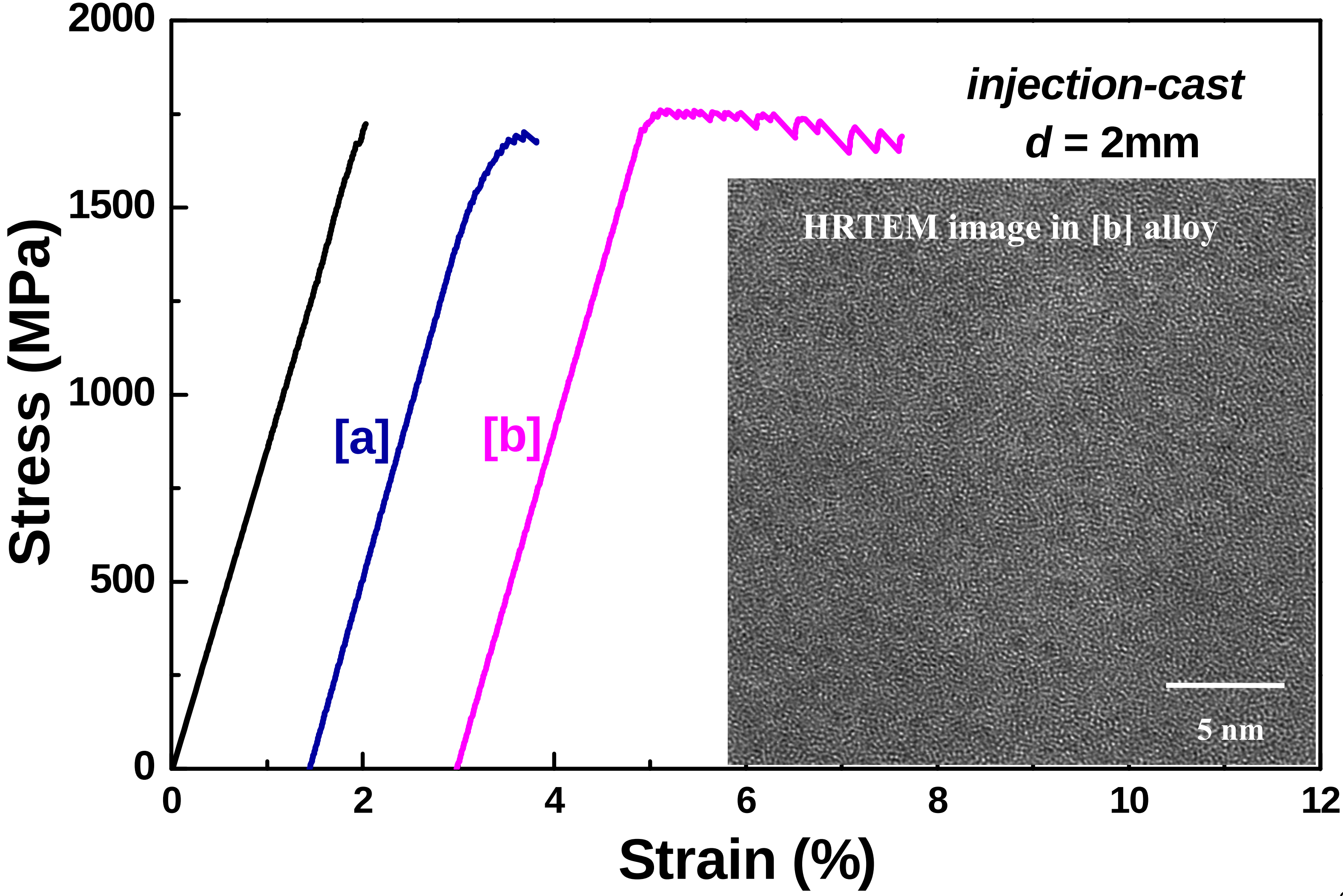
(b) $Zr_{57}Ti_8Nb_{2.5}Cu_{13.9}Ni_{11.1}Al_{7.5}$



Fully amorphous structure

Effect of quenched-in quasicrystal nuclei

- Compression test

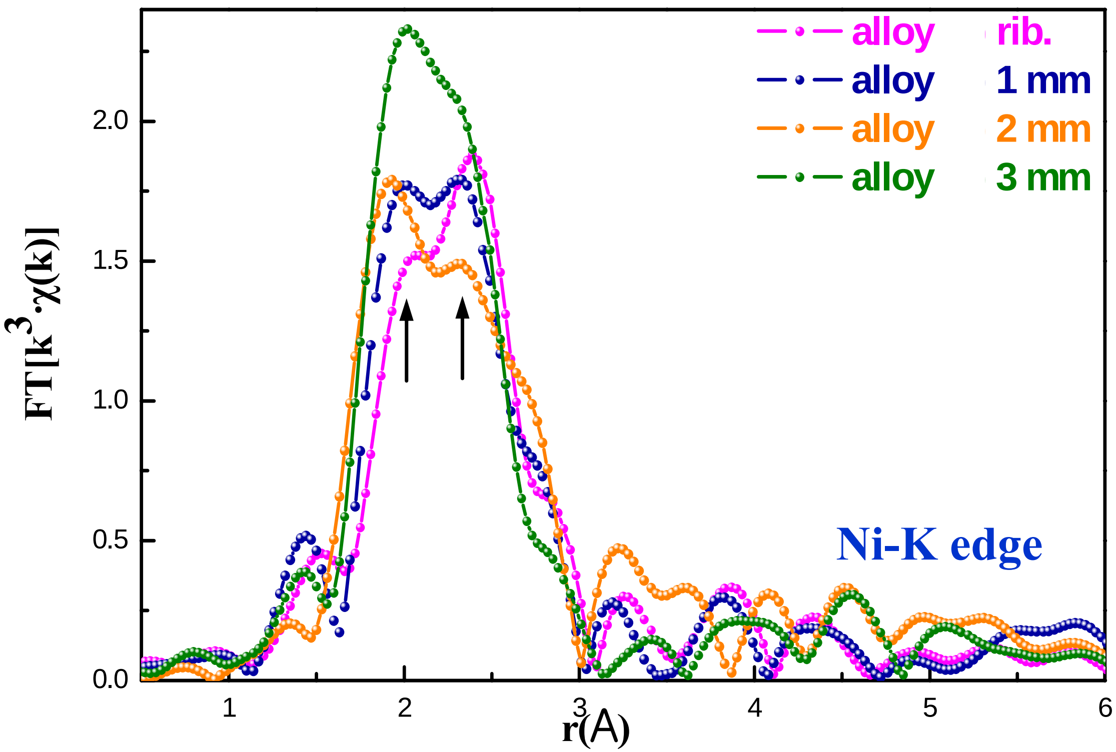


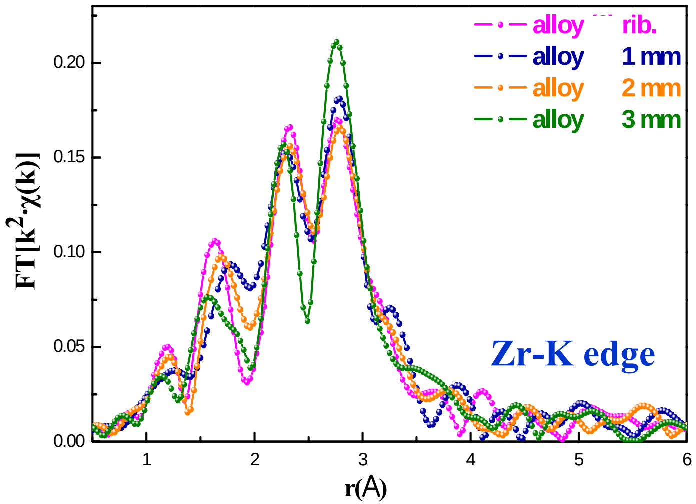
Effect of quenched-in quasicrystal nuclei

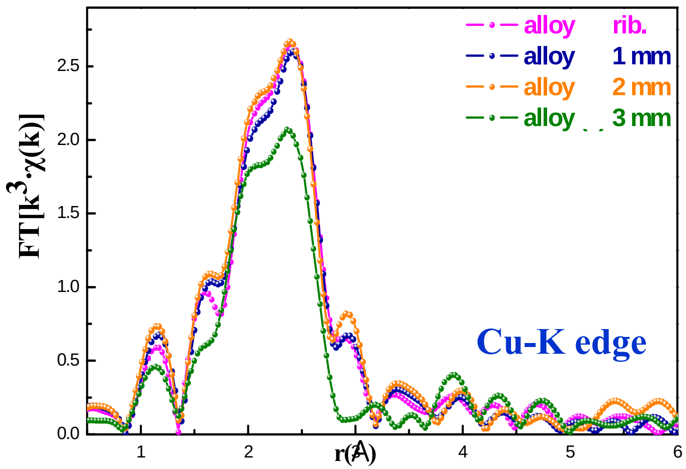
- **EXAFS analysis**

Extended X-ray Absorption Fine Structure

(b) $Zr_{57}Ti_8Nb_{2.5}Cu_{13.9}Ni_{11.1}Al_{7.5}$





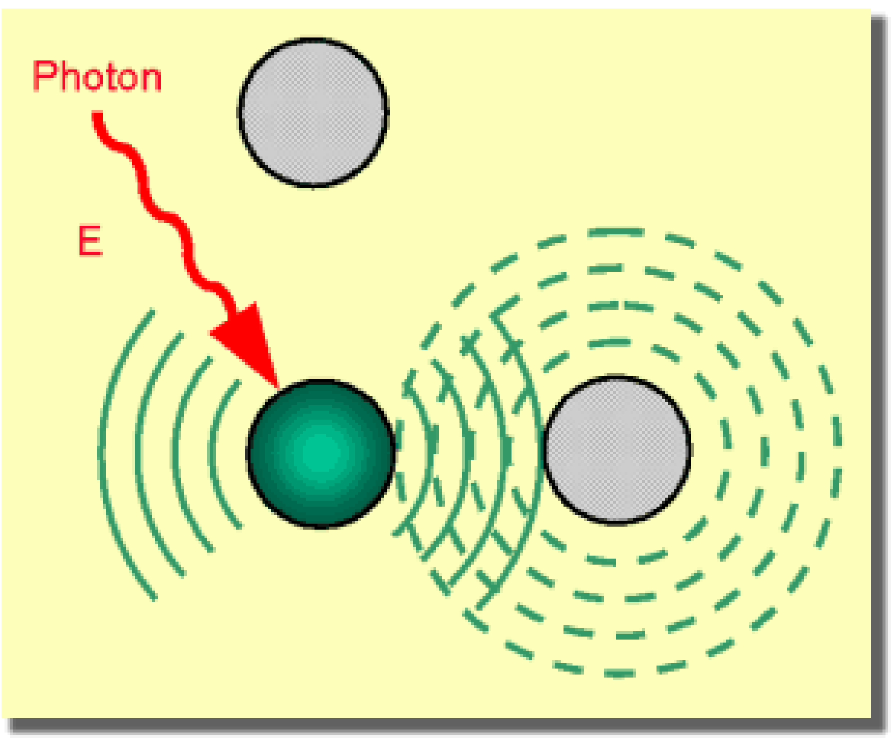


Distinctive structural change around Ni atom

Intensity change due to microstructural change

EXAFS Analysis

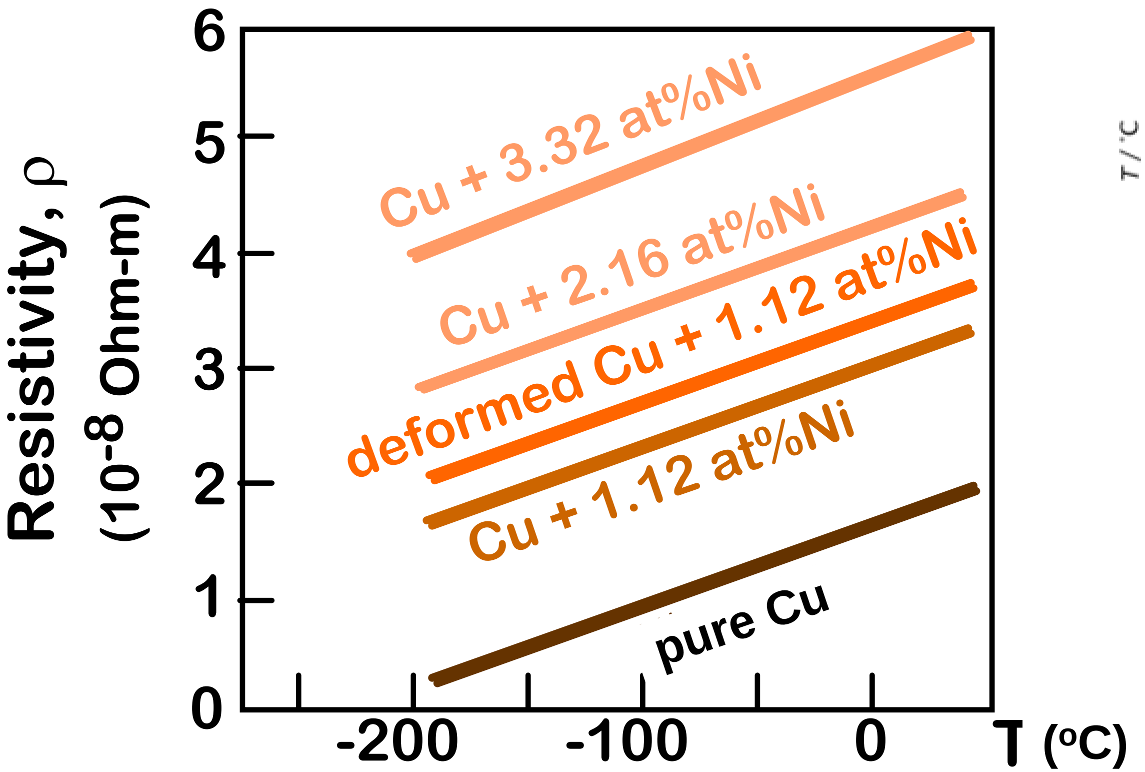
Extended X-ray Absorption Fine Structure (EXAFS)는 고체를 통과한 X-선의 흡수 spectrum을 분석하여 원자단위의 구조를 분석하는 방법으로 그 동안 전이금속을 포함한 분자, 촉매, 비정질 재료, 초전도재료 등 다양한 재료들에 있는 특정 원소 주위의 local structure 분석하기 위하여 많이 사용되어 왔다.

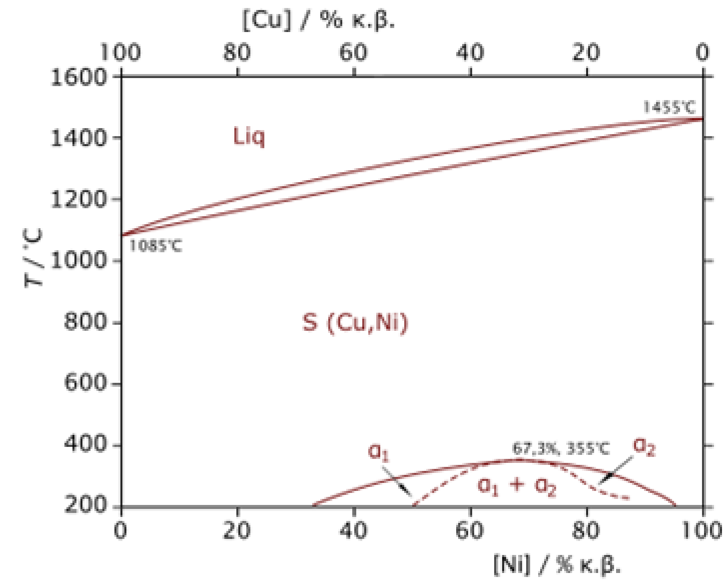


포항방사광 가속기

ELECTRICAL Properties

- Electrical Resistivity of Copper:





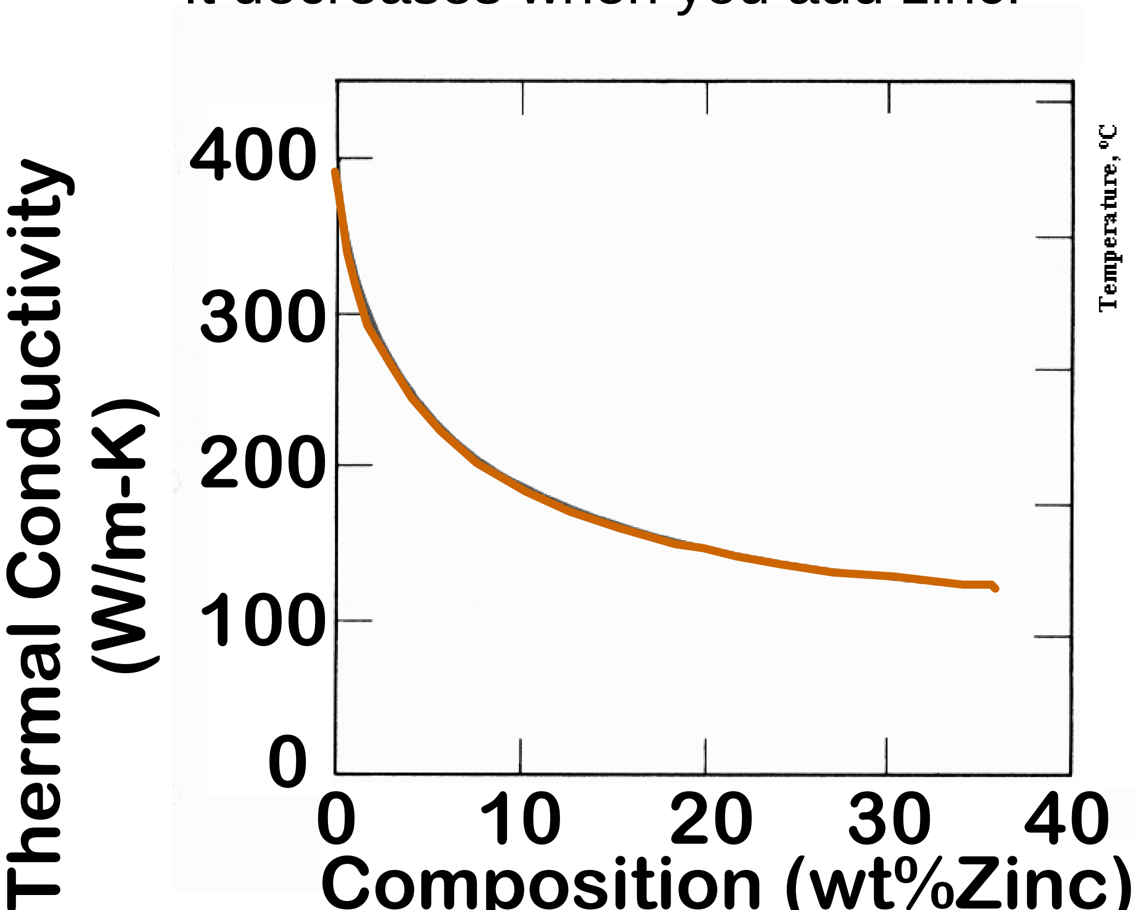
Adapted from Fig. 18.8, *Callister 6e.* (Fig. 18.8 adapted from: J.O. Linde, *Ann Physik* **5**, 219 (1932); and C.A. Wert and R.M. Thomson, *Physics of Solids*, 2nd edition, McGraw-Hill Company, New York, 1970.)

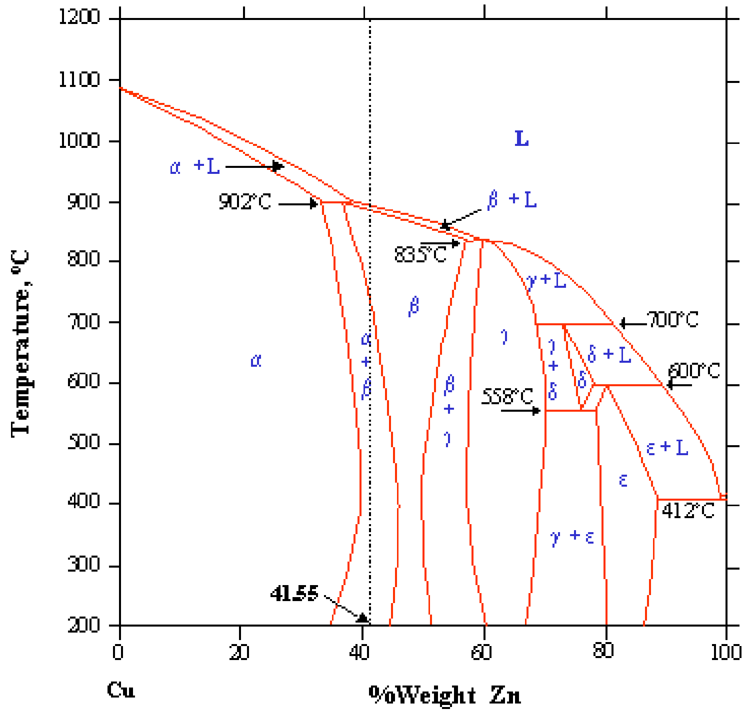
- Adding "impurity" atoms to Cu increases resistivity.
- Deforming Cu increases resistivity.

THERMAL Properties

- Thermal Conductivity of Copper:

  --It decreases when you add zinc!



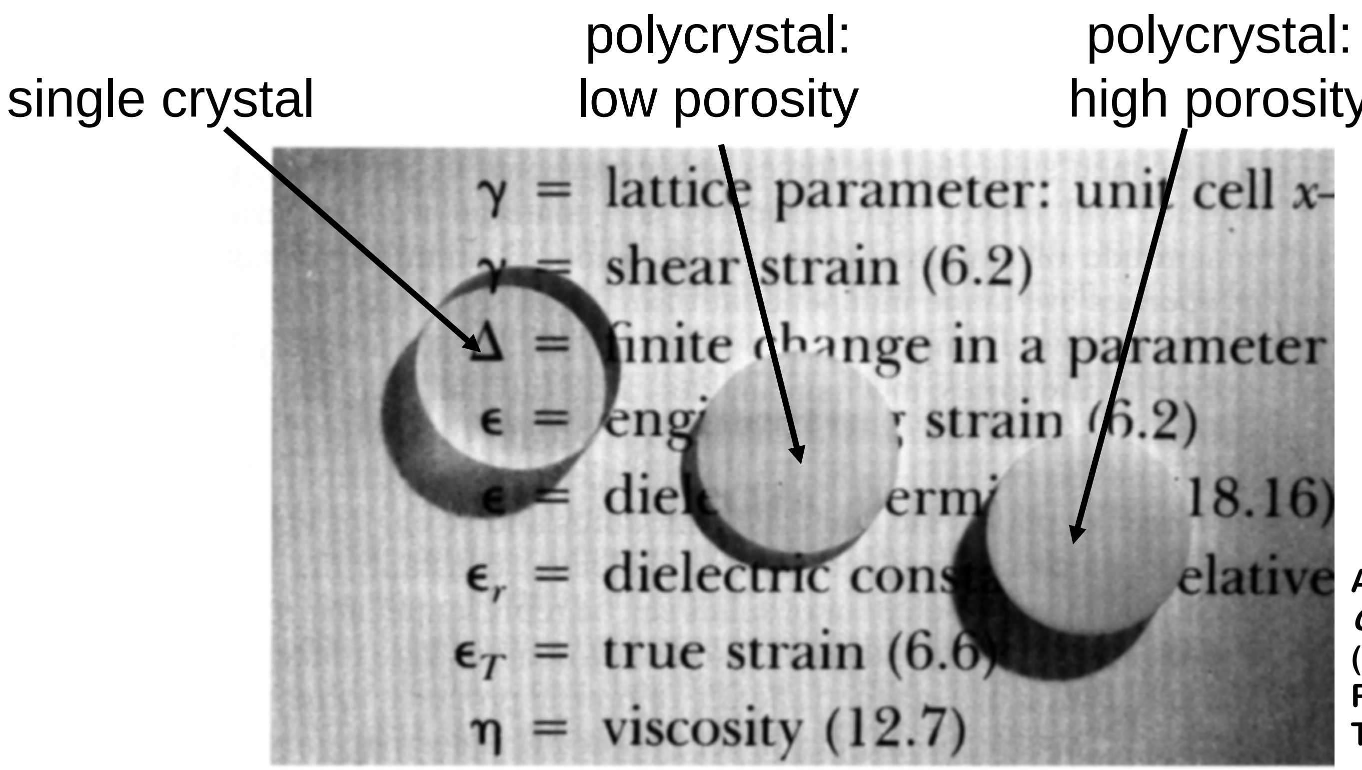


Adapted from Fig. 19.4, Callister 6e. (Fig. 19.4 is adapted from Metals Handbook: Properties and Selection: Nonferrous alloys and Pure Metals, Vol. 2, 9th ed., H. Baker, (Managing Editor), American Society for Metals, 1979, p. 315.)

OPTICAL Property

- Transmittance:

  --Aluminum oxide may be transparent, translucent, or opaque depending on the material structure.

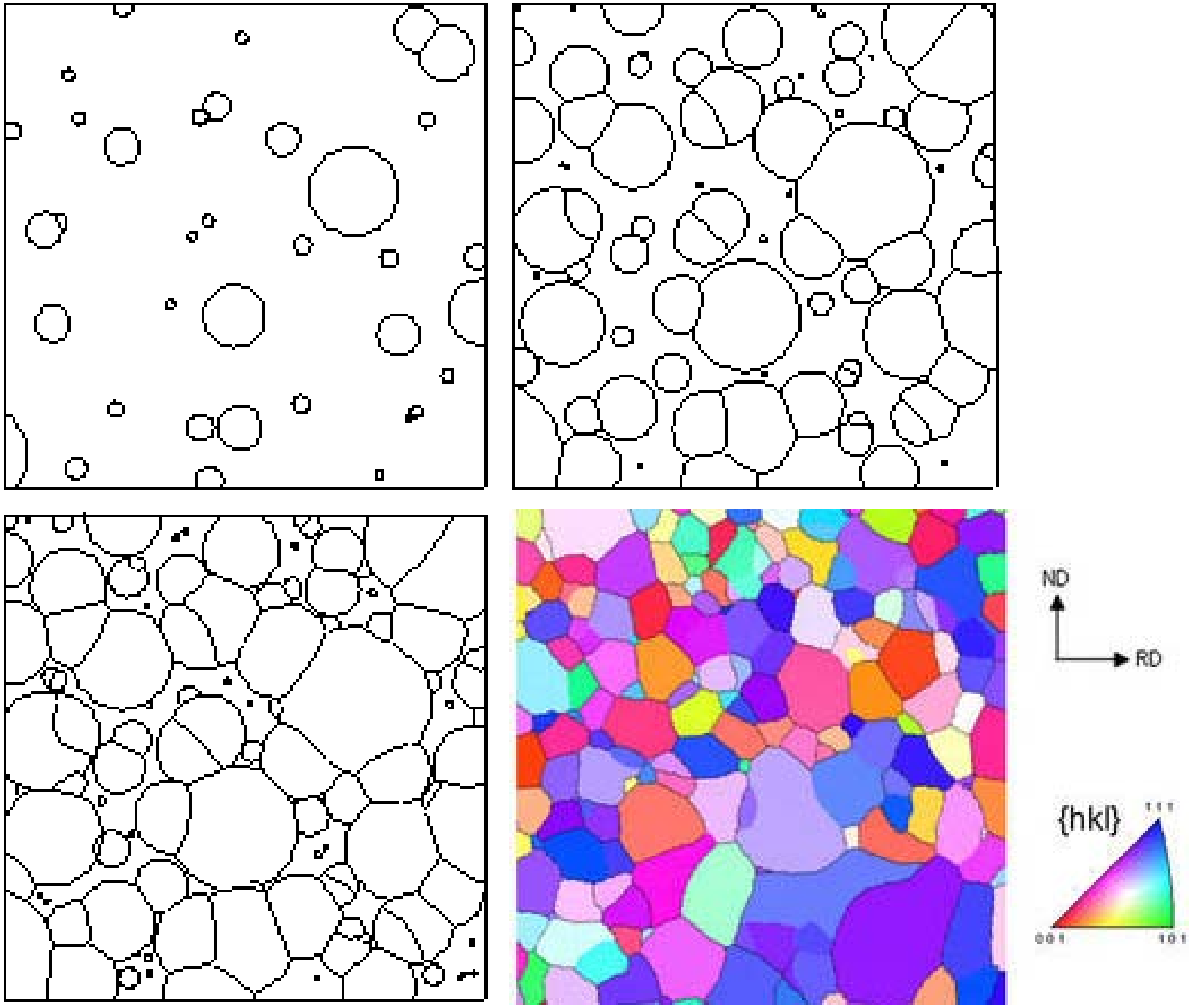


Adapted from Fig. 1.2, *Callister 6e.* (Specimen preparation, P.A. Lessing; photo by J. Telford.)

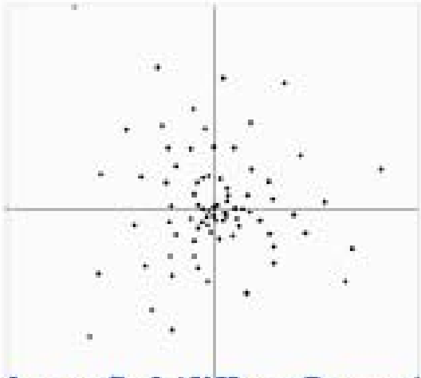
Polycrystals

- Most materials are polycrystalline. This means that there are distinct crystals (grains) present, each of which has a different orientation from its neighbors. Each boundary between a pair of phases is termed a *grain boundary* or *homophase interface* and is, obviously, a *planar defect*.
- Why can grain boundaries be observed? The different orientations of the crystal directions in the grains on either side of a boundary mean that there is a discontinuity across the boundary. In metals and ceramics, the discontinuity is very narrow (one or two atoms). The boundary can be observed because it etches preferentially (topographic) or produces strain (observable in electron microscopy), for example.
- How can we quantify the idea of crystal directions varying from one grain to another? The (crystallographic) *orientation* of a grain requires three angles to be measured (from mathematics: one must specify a rotation in 3D). The average orientation of grains in a material is known as its *texture* (also known as *fabric* in rocks and minerals).

Grain growth

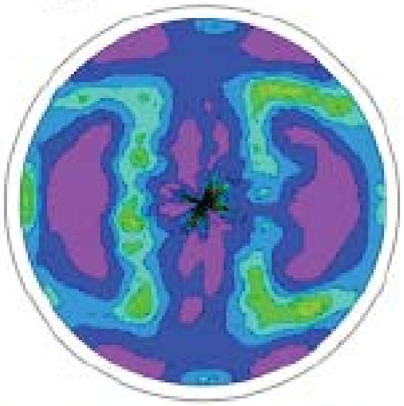


Crystal Orientation: texture



escher.epfl.ch/diffractOgram/

100



- The best method of quantifying texture is to measure the crystallographic orientation of a statistically representative set of grains in the material.
- Historically, this was a tedious exercise with Laue X-ray diffractograms that was only possible on single crystals or very coarse polycrystals.
- The standard characterization method for *texture* (crystallographic preferred orientation) in an average sense is to measure *x-ray pole figures*.
- A more modern technique is that of Orientation Imaging Microscopy (OIM), which is readily available in the SEM (and much used here at CMU). This technique produces a map of orientation measured on a regular grid of points (pixels).