

2009 spring

*Microstructural Characterization
of
Materials*

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Introduction - 수업진행관련

- 실습 포함
- **Web lecture assistance :** <http://eng.snu.ac.kr/lecture/index.php>
 - 모든 강의 자료는 **web**을 통해서 받아 볼 수 있음.
- 설계학점 포함
- 평가
 - 중간고사 (25%), 기말고사 (30%),
Homework (10%), Quiz+참여도 (10%), 조별활동 (25%)
- **교재 – scanned materials in web**
 - 주교재:
 - 1. **ASM handbook Vol 10. Materials Characterization**, The materials information society, 1986
 - 보조교재:
 - 2. **Microstructural Characterization of Materials**, 2nd ed. By D. Brandon, W.D. Kaplan, Wiley
 - 3. **Principles of Instrumental Analysis**, 5th ed. D. A. Skoog, Saunders College Publishing, 1992
 - 4. **Elements of X-ray Diffraction**, 2nd ed. by B.D. Cullity, Addison-Wesley Publisher
 - 5. **Scanning Electron Microscopy and X-ray Microanalysis**, By Goldstein Plenum publisher

Introduction - 수업내용관련

- **Course Goals**
 - **To introduce fundamental concepts of microstructural characterization, including theory, interpretation, equipment, and limitations.**
 - **To help you identify appropriate characterization**
- **실습 (Demonstrations)**
 - **XRD, SEM, TEM, (XPS + AES)**
 - 실습시간이 아직 정해 지지 않은 관계로 실습은 1-2주 전 강의시간에 통보
 - 실습결과의 해석 – **Report**
- **설계 – Report**
 - 목표설정 (구조분석, 화학분석, 목표 원소,.....)
 - **Source**와 감지기를 조합하여 분석기기를 설계
 - 예상되는 분석 결과
 - 고안된 분석기기의 문제점 (한계, 극복해야 할 점 등)

Introduction - 실습내용관련

- ***X-ray diffraction***
 - Measure θ - 2θ data from a specimen and find the materials
- ***Sample preparation***
 - Demonstration of Focused Ion Beam
- ***Scanning electron Microscopy***
 - Imaging with secondary electrons
 - Analysis of Energy Dispersive Spectroscopy (X-ray spectrum)
- ***Transmission Electron Microscopy***
 - Demonstration of Image and e-diffraction acquirement
 - Identification of the material
- ***X-ray Photoelectron Spectroscopy and Auger Electron Spectroscopy***
 - Quantitative analysis of the spectrum

Policies and Procedures

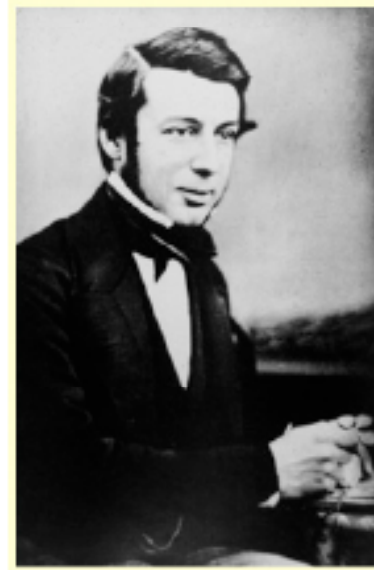
- ***All homework are due by the start of class on the stated deadline.***
 - Late assignments go to my office. If I'm not around, slide it under my door and leave me an email so that I know when you turned it in.
 - You lose 20% of the full assignment value per day late. Since homework are due on Wednesday, you can get 80% credit if you turn it in on Thursday, 50% on next Monday, nothing thereafter.
- ***If you wish, you may work together on homework assignments. BUT, you must hand in your own work, in your own words.***
- ***IMPORTANT: you MUST reference your sources appropriately, including texts, journals web sites, etc.***
 - Article authors, title, journal, volume, year, pages
 - Book authors, title, publisher, year, pages
 - Web address
 - etc.

Microstructure = internal structure

- Biology was revolutionized when **Leeuwenhoek** and others started to use microscopes to look at the internal structure of plants. They were able to relate many characteristics of plants to their cell structure, for example.
- Similarly, **Sorby† (portrait)** was one of the first to make cross-sections of materials such as iron and examine them in the microscope, so that he could relate properties to structure.

*(<http://www.ucmp.berkeley.edu/history/leeuwenhoek.html>)

† <http://www.shu.ac.uk/sorby/hcsorby.shtml>



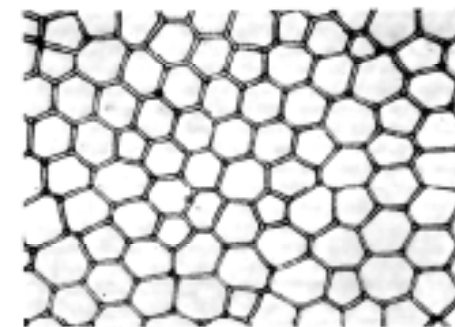
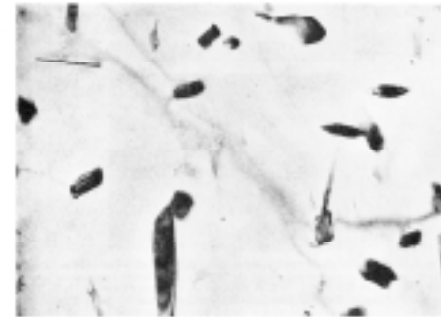
What is microstructure?

- Microstructure originally meant the structure inside a material that could be observed with the aid of a microscope.
- In contrast to the crystals that make up materials, which can be approximated as collections of atoms in specific packing arrangements (*crystal structure*), microstructure is the collection of defects in the material.
- What defects are we interested in? Interfaces (both grain boundaries and interphase boundaries), which are *planar defects*, dislocations (and other *line defects*), and *point defects* (such as interstitials and vacancies as well as solute atoms in solution).
- Since the invention of prefixes for units, the micrometer (1 μm) happens to correspond to the wavelength of light. Light, obviously is used to form images in a light/optical microscope. Thus *microstructure* has come to be accepted as those elements of structure with length scale of order 1 μm .
- Microstructure is vitally important down to the nanometer scale (nm), however, for which one must use electron microscopy (e.g. TEM) and surface microscopies (e.g. atomic force microscopy = AFM).

Types of Defect

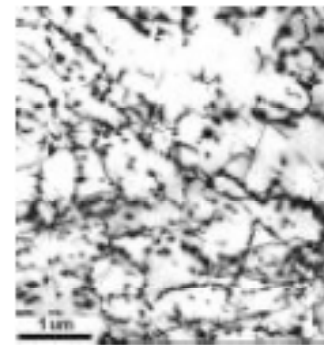
- Interfaces - phase boundaries
 - Micrograph shows theta-prime precipitates (Al_2Cu) in Al
= **planar defect**
- Interfaces - grain boundaries
 - Micrograph actually shows a soap froth but the image is representative of a grain boundary network in a polycrystal.
= **planar defect**
- Dislocations
 - This messy image illustrates the difficulty of counting dislocations: generally one avoids counting individual dislocations!
= **line defect**

Porter & Easterling



Reed-Hill

Fig. 15.20 Cellular structure in tin as viewed normal to the interface. 180 x.
(Hutter, J. W., ASM Seminar, *Liquid Metals and Solidification*, 1968, p. 342.)



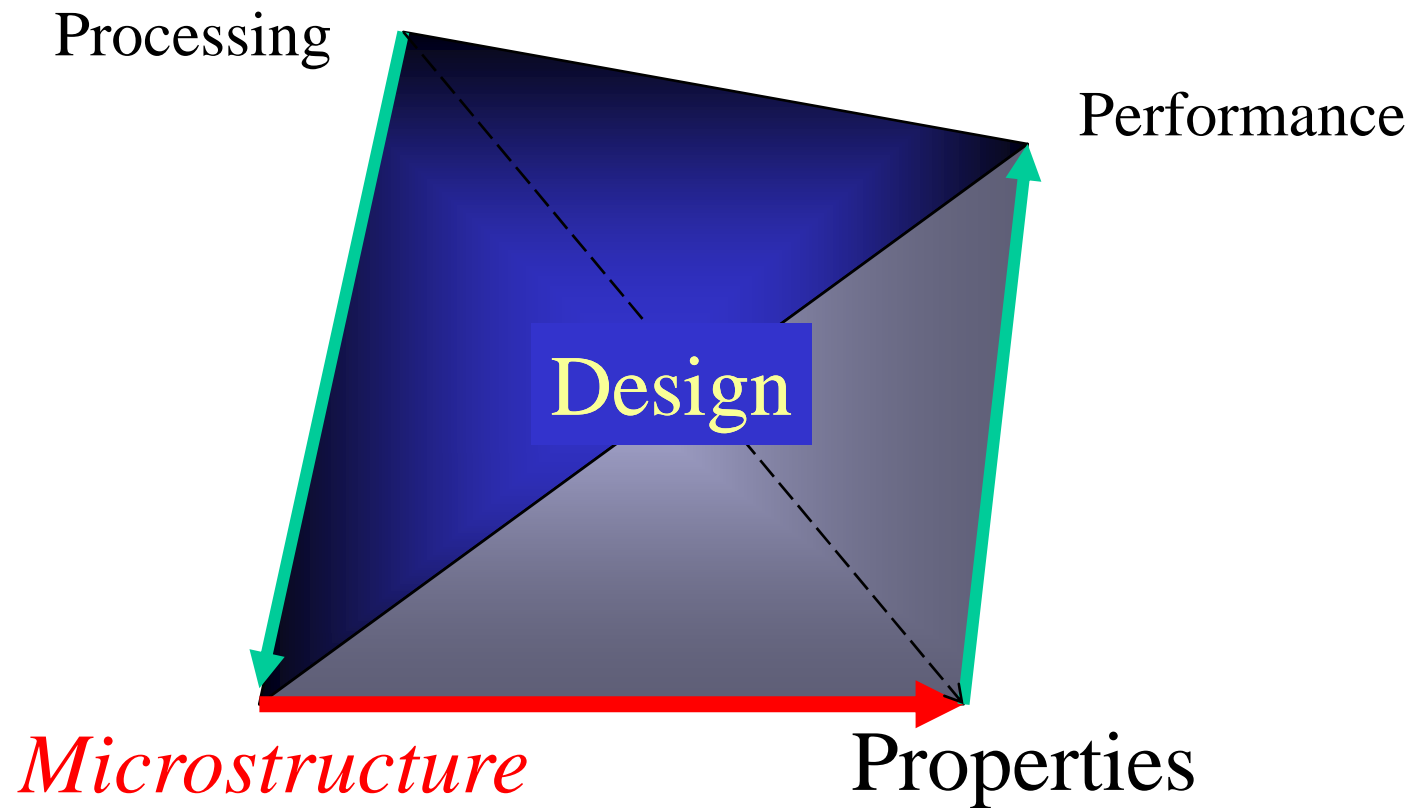
How to Observe Microstructure

- Observation of microstructure requires us to make images.
- In order of increasing effort, the standard methods are; 1) optical microscopy; 2) scanning electron microscopy (SEM); 3) transmission electron microscopy (TEM); 4) atomic force microscopy (AFM); 5) scanning probe microscopy (SPM).
- Most microscopy techniques rely on topographic contrast require specimen preparation in order to reveal the microstructure. Also useful is varying color (grayscale) in different grains/phases based on, e.g. oxide layer thickness.

Microstructural Parameters

- It is essential to quantify microstructure in order to be able to predict properties quantitatively.
- What you quantify depends on the property, i.e. what question you ask of the material.
- Examples of quantitative microstructural parameters:
 - Grain size
 - Void fraction
 - Second phase particle size
 - Aspect ratio of second phase particles or grains
 - Average distance between particles

Microstructure-Properties Relationships



Microstructural Parameters, Properties

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)

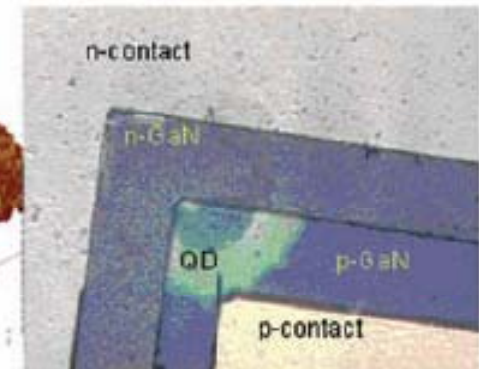
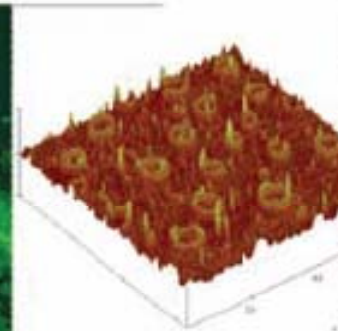
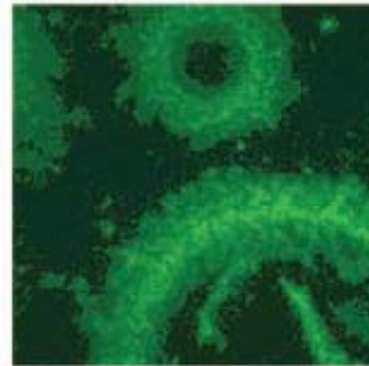
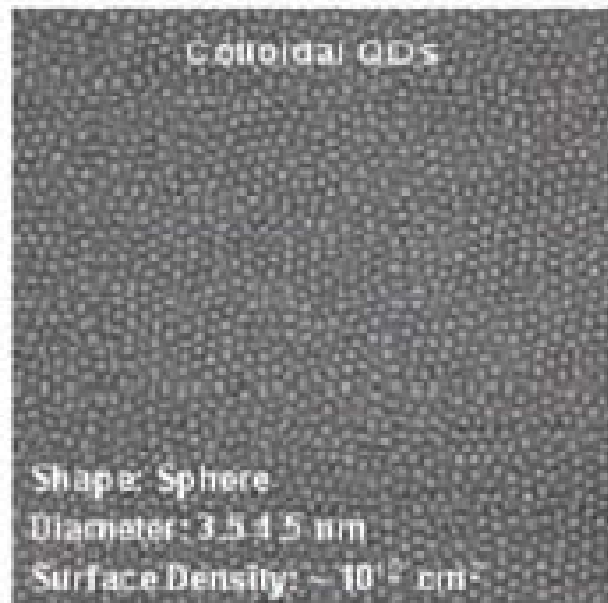
Microstructure-Property Relations

- *The optimization of material properties through control of microstructure is the central paradigm in the discipline of Materials Engineering.*
- *History: blacksmiths represent the original (empirical) practitioners of this approach. To this day, they vary the microstructure of steels by varying their heat treatment in order to affect their mechanical properties. Try searching on “Damascus steels” to find out more about the astonishing variety of microstructures used in sword blades, for example.*



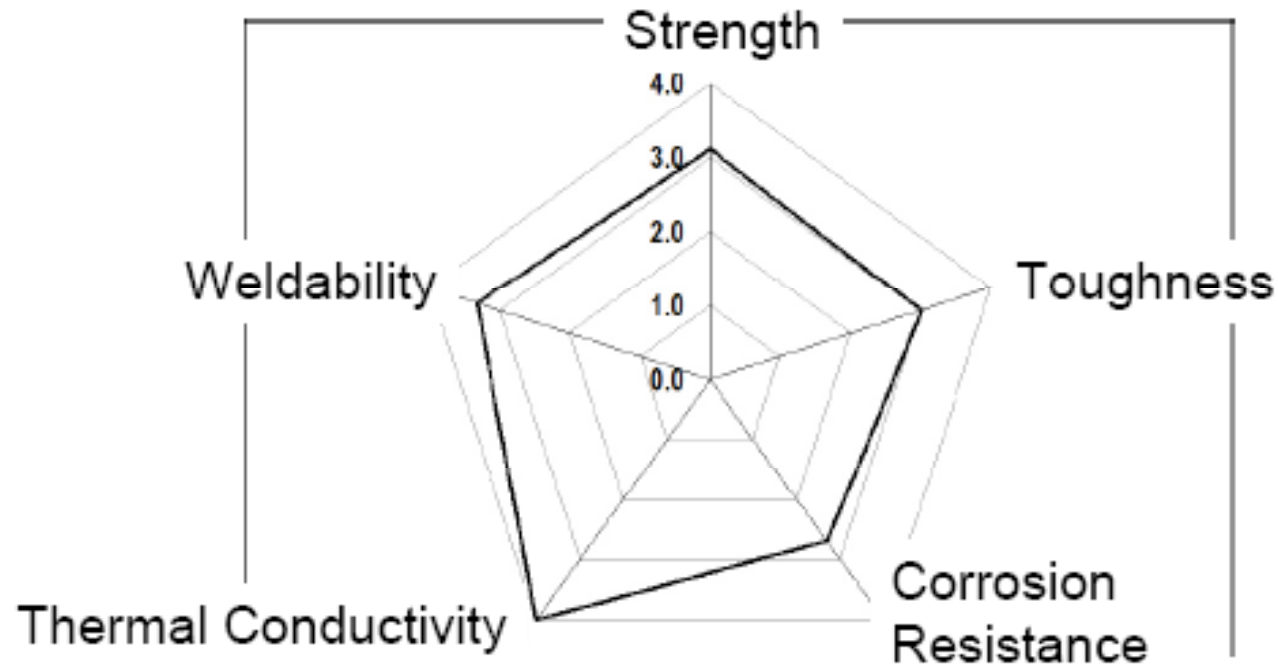
Microstructure-Property Relations

- **Cutting edge: controlled doping (=alloying) of semiconductors to make quantum dots. This is phase separation to make a two phase structure in which the size of the (approx.) spherical particles of one phase are small enough that quantum effects are important.**



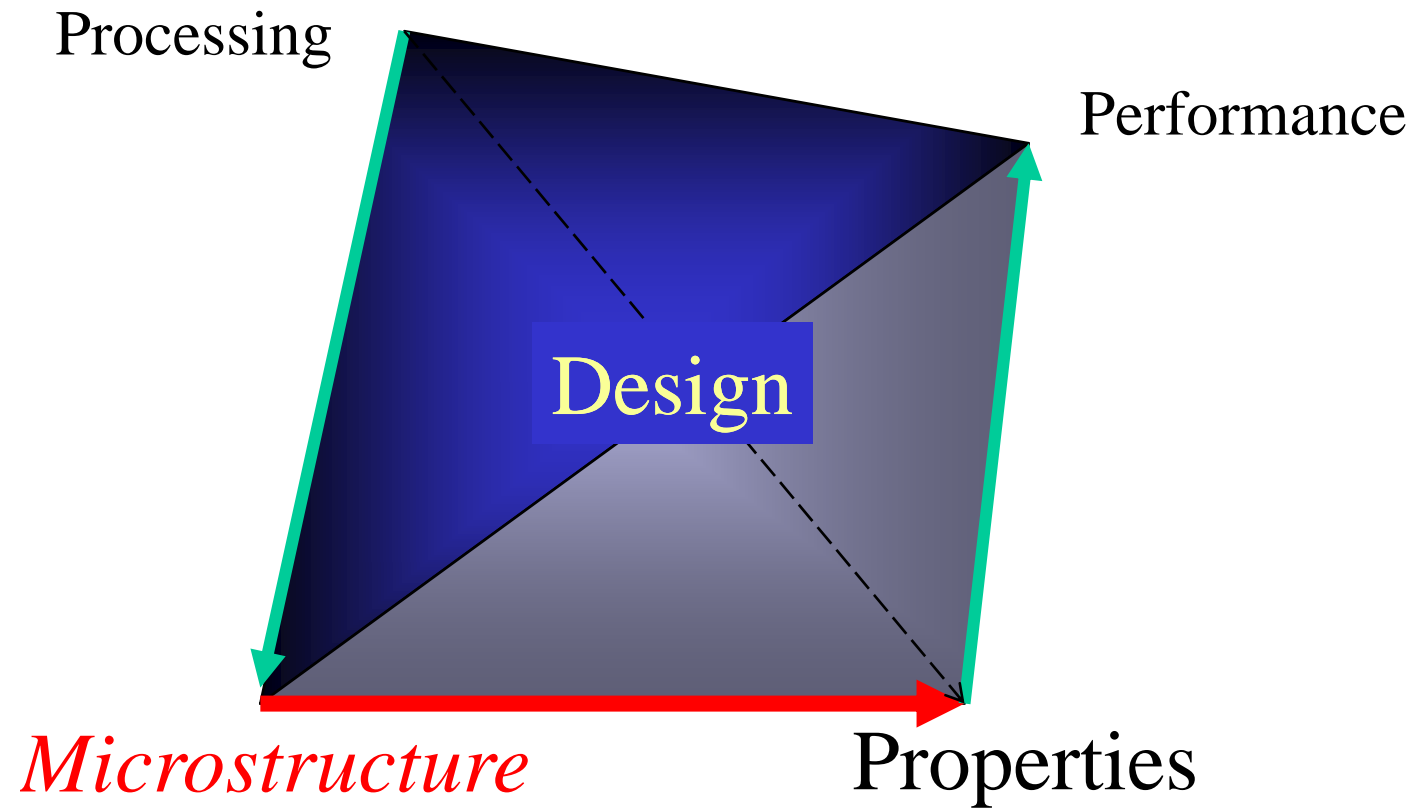
The Electrochemical Society *Interface* • Winter 2006

Optimization of Multiple Properties



- It is generally the case that any given material must meet minimum requirements for multiple properties.
- Therefore it is not feasible to optimize one property at the expense of all others.
- On the “spider diagram” example, one can visualize this requirement by seeing that the polygon must have vertices at some distance from the origin along every axis.

Microstructure-Properties Relationships



“Tailor-made Materials Design”