**Introduction to High-Throughput Materials Development**

**Introduction**

1. Nanoscale and microscale design differ fundamentally in the following way:

Nanoscale design is typically more heavily directed towards devices

Hierarchy of material structure is more pronounced at the nanoscale

Microscale simulations are directed at lower length and time scales

One can be supported using first principles and atomistic modeling

1. Which is more closely related to high-throughput computational screening?

Combinatorial search for candidate Li ion battery electrode materials

Exploring process-structure-property relations for field service conditions in composite materials

Search for steels with high formability for automotive fenders

Search for Al alloys with high fracture toughness in aircraft panels

1. Which of these is least important in computational high-throughput screening of materials?

Benchmarking with known reference materials

Data mining and correlations

Human intuition

Predictive materials modeling and simulation

1. Which of these aspects has received greatest attention to date in engineering practice?

Materials design

High-throughput materials development

Materials selection

1. Material structure hierarchy refers to

Atomic configuration

The tendency of materials to behave differently as a function of time

Various quantized electron orbitals

The various levels of structure from atomic through mesoscale to the component/application level

1. Concurrent materials and product design

Requires that the materials be developed first, and then integrated into the product

Combines bottom up and top down information

Is only top down

Is only bottom up

1. Robustness is desirable to

Ensure that the material performance has limited sensitivity to variations of chemistry and process route

Ensure multifunctionality

Provide increases in material properties/performance

Minimum design iterations.

1. One of the primary driving forces for accelerated discovery and development of materials is:

Increased automation

High cost of raw materials

The novelty of new materials is exciting to investors

Time to introduce new materials is much longer than design and prototyping of products

1. Historical materials discovery and development was largely:

Pursued by large, distributed development teams

Performed in a few months or few years

Persistently empirical or “Edisonian”

Based on predictive modeling and simulation

1. The U.S. Materials Genome Initiative seeks to accelerate materials development by \_\_\_\_\_\_\_\_\_\_ phases of development, certification, manufacturing and deployment.

Parallelizing

Serializing

1. The Materials Innovation Ecosystem

Refers only to traditional materials science of process-structure-property performance relations

Is confined only to universities

Refers to the overall ecosystem of materials science, design, modeling and simulation, data sciences and informatics, manufacturing, and training/education

Mainly concerns innovation in start-up companies

1. The complexity of materials discovery is due to

Complex property dependence on structure and composition

Large number of compositions

Multiple processing conditions

Variety of structures

1. How can HTMD accelerate development?

Efficient exploration large numbers of compositions

Intelligent planning and selection of experiments

Incorporation of accurate instrumentation

Integration with advanced computation and machine learning

1. Which of the following is an early 20th century example of HTMD?

Mittasch, Haber and Bosch screening of over 4000 catalysts.

Kennedy published the first alloy phase diagram.

Haber discovery of ammonia synthesis.

Hanak reported multiple sample concept.

1. In his 'Multiple sample concept' paper, how did Hanak cover a large composition range in single experiment?

Composition gradient

Structure gradient

High-through processing

1. The company Symyx was formed around the time of the publication of which HTMD landmark paper?

J. Hanak, “Multiple sample concept” in 1970.

Xiang et al., “A combinatorial approach to materials discovery" in 1995.

Fodor et al., “Combinatorial” peptide libraries with 1024 members” in 1991.

Danielson et al., combinatorial approach for 'Rare-earth phosphors' in 1997.

1. How was the library investigating polymer wetting built in the work reported by Meredith, Smith, Karim, Amis in Macromolecules 2000 33, 9747?

Temperature gradients

Structure gradients

Thickness gradients

Composition gradients

1. Which important material was obtained by Kohn in 2004 through HTMD polymeric materials?

1st degradable, radio-opaque stent

1. Two general goals of HTMD are materials discovery and knowledge discovery.

True

1. Which of the following libraries are used in HTMD?

Large data library

Gradient library

Discrete library

Experimental library

# Library Preparation

1. Co-deposition of thin films can be accomplished by using:

Sputtering

Evaporation

Pulsed Laser Deposition

Sputtering and Evaporation only

All of the above

1. Combinatorial physical vapor deposited thin film libraries prepared on DIFFERENT substrates will have identical microstructures and properties.

False

1. Producing thin film combinatorial material libraries of varying composition is easier with physical vapor deposition methods than with chemical vapor deposition methods.

True

1. How does the flux from a physical vapor deposition process vary with distance (r) from a point source?

1/(r^2)

1. When comparing sputtering to evaporation:

Evaporated species have more energy when they reach the substrate surface resulting in slower surface diffusion kinetics.

Sputtered species have more energy when they reach the substrate surface resulting in slower surface diffusion kinetics.

Sputtered species have more energy when they reach the substrate surface resulting in faster surface diffusion kinetics.

Evaporated species have more energy when they reach the substrate surface resulting in faster surface diffusion kinetics.

1. Which of the following physical vapor deposition methods produces a discrete combinatorial library of material compositions?

Masked Deposition

1. What challenge is presented when a compositional combinatorial library is prepared by vapor depositing thin films through a series of shadow masks?

It is impossible to form discrete compositional libraries.

Large quantities of material must be used.

The process cannot be automated.

Deposited films may not be compositionally homogeneous.

1. Which of the following is NOT true about the physical vapor deposition of thin films:

In a PVD process, the type of deposition source has no influence over the final film microstructure.

PVD is a line-of-sight deposition technique.

Thermal evaporation and pulsed laser deposition are examples of PVD techniques.

The adsorption, surface diffusion, and desorption rates of vapor deposited atoms all play a role in the kinetics of a PVD thin film deposition process.

1. Upset forging relies on varying the quench rate in order to create microstructure gradients.

False

1. Which of these processes are controlled for the solution treatment and aging process?

Solution temperature

Quench rate

Aging temperature

Aging time

1. Rapid Alloy Prototyping (RAP) typically takes more than one day to complete.

True

1. Which of these statements about RAP are true?

Studying the effects of various alloy compositions and thermomechanical treatments in a short duration

Modular concept, allowing for easy addition of new processing techniques and thermomechanical treatments

Mass producing new alloys for industrial use at a rapid pace.

1. If you were designing a Rapid Alloy Prototyping (RAP) study to explore the effects of varying Mo in the alloy Ni-35Co-20Cr-xMo with x = 10,12,14, or 16 wt. %, followed by 80% cold work, then aged at either 500°C, 550°C, or 600°C for 0, 1, 4, or 24 hours, how many combination of process conditions would you need to prepare?

40

1. Metal alloy libraries can be fabricated using

Diffusion multiples

Additive manufacturing

Thermal gradients

Investment casting

Directed vapor deposition

Deformation gradients

1. Alloy properties that depend on microstructure include

Strength

Density

Melting temperature

Fracture toughness

Formability

1. Diffusion multiples:

Can be used to establish equilibrium phase diagrams.

Can be used to establish diffusion coefficients.

Are fabricated by physical vapor deposition.

Undergo hot isostatic press (HIP) before being exposed to test temperature.

1. The most effective additive manufacturing methods to generate materials libraries include:

Electron Beam Additive Manufacturing (EBAM) process with multiple wire feeds

Vat photopolymerization

Powder bed methods

Material jetting using multiple nozzles

Direct metal deposition

1. A material library is a collection of samples with variations in attributes, such as:

Composition

Chemical structure

Material thickness

1. What is varied in the the library of atom transfer radical polymerization published by Zhang et al., *Macromol. Rapid Comm.* 2003 24, 81?

Catalysts

Initiators

Reaction Temperature

Reaction Time

1. What is varied in the polymer melt processing library described by Potyrailo et al., Macro. Rap. Comm. 2003 24, 123, using a microextruder system?

Gradient

Structure

Composition

Temperature

# Characterization of Composition and Structure

1. Which of the following concepts are related to control of materials structure?

Molecular bonding

Nano/microstructure

Segregation on a surface

Spatial organization of components

1. Which of the following spectroscopic methods was used to determine the PCL/PDLA compositions in the library discussed in this module?

UV/Visible/NIR

Raman spectroscopy

Fourier-Transform Infrared spectroscopy

X-ray fluorescence

1. What is typically included in the polymer chemical structure?

Monomers arranged spatially

Chemical bonds

Molecular size

1. What is the standard tool to determine molecule size of polymer?

MALDI-TOF Mass spectrometry

Gel permeation chromatography

Raman spectroscopy

Fourier-Transform Infrared spectroscopy

1. In studying the crystallinity in thin polymer films, what parameters are used to create the library?

Composition

Time

Thickness

Temperature

1. Which of these methods can possibly be used to identity a phase transformation?

XRD

Monitoring color change

Nano-calorimetry

Measuring change in reflectivity

Four-point resistance measurements

EBSD

1. Which of these methods can be used to determine the composition of inorganic material libraries?

Four-point resistance probes

Nano-calorimetry

XRF

EPMA

EBSD

1. Which of these methods can be used to determine the crystal structure of inorganic material libraries?

EPMA

EBSD

Four-point resistance probes

XRF

XRD

1. Which of these methods is based on measuring the thermal response to identify a phase transformation?

Parallel nano-scanning calorimetry.

Electron Probe Micro-Analysis

Laser deflection detection of micromachined cantilevers.

Multiple four-point resistance probing.

1. For identifying the crystal structure of inorganic materials, XRD has \_\_\_\_\_\_\_\_\_ than EBSD.

Longer sample preparation times

Better accuracy (i.e., can determine lattice parameter)

Higher spatial resolution

Faster acquisition time per each sampling volume

# High-Throughput Property Measurements

1. Which of the following are defined as material optical properties that are of interest for applications of HTMD?

Light absorption

Modulus

Conductivity

Capacitance

Emission

1. What type of materials for displays have been explored with HTMD?

Metals

Phosphors

OLEDS

Cathodes

1. What is the type of library used for the discovery of the superconducting materials in the paper of Xiang et al., *Science* 1995 *268*, 1738?

Structure library

Electronic library

Temperature library

Composition library

1. What does the HTMD indenter testing directly measure?

Pressure versus temperature

Force versus depth

Force versus modulus

Pressure versus abrasion

1. Which of the following comments are true for catalysis of chemical reactions?

Catalysis can increase the rate of reaction.

Catalysis can be used for selection of desired reaction.

Empirical processes can’t increase catalyst activity.

Catalysis only involve metal oxides and metals.

1. Which of the follow definitions are true?

Diffusion is referred to the rate of thermal motion of molecules to gas, liquid or solid.

Sorption is defined as molecules attaching to a surface

Protein adsorption of Al1-xTix and Al1-xNbx is affected by their structure gradient.

Adsorption is defined as molecules attaching to a surface

1. Which techniques can be used for high-throughput characterization of protein adsorption?

Wavelength dispersive spectroscopy

Gel permeation chromatography

Fluorescence spectroscopy

Spectroscopic ellipsometry

1. Biological properties in HTMD typically involve:

Cell separation

Material binding to a biological molecule

Biological molecules decomposition with heating

Cell adhesion to material

1. What libraries were created for the example study of osteoblasts cell adhesion on blend polymers of poly(DL-lactide) and poly(caprolactone)?

Film thickness gradient library

Temperature gradient library

Polymer structure gradient library

Polymer composition gradient library

1. Which of these methods is best suited for high-throughput yield strength measurements?

Compression test

Atomic force microscopy

Spherical indentation test

Tensile test

4-point bend test

Sharp indentation test

1. The elastic modulus is proportional to :

The square root of hardness

Density

The square of the longitudinal wave velocity

Yield strength

1. For high-throughput property measurements, which of these attributes is the least critical?

Possibility of automation

High accuracy

Rapid speed of measurement

Fine spatial resolution

1. Which one of these is not accomplished by the zero point correction discussed in Kalidindi and Pathak, Acta Materialia 2008 56, 3523?

Corrects for any inconsistencies caused by surface roughness of the sample.

Corrects for any inconsistencies caused by the imperfections in indenter shape.

Corrects the measurement of the yield strength such that it matches the tensile test data.

Makes the measurements in the initial elastic loading segment consistent with the predictions of Hertz’s theory.

1. Identify the true statements regarding high-throughput fracture toughness tests using indentation.

If material has high toughness, greater than 15 MPa {\sqrt{m}}*m*​, then it will not be possible to determine the local variations in fracture toughness.

Spherical indenters are used.

Uses a minimal among of material to establish a fracture toughness value compared to a convenient fracture toughness test.

It is possible to investigate how fracture toughness varies spatially across a sample.

Can only obtain quantifiable fracture toughness values when the material is relatively brittle.

1. The size of the indenter, as long as the tip geometry remains the same, does not affect the property measurement.

False

# Polymers for Proton Exchange Membranes in Fuel Cells

1. What is the function of the proton exchange membrane in fuel cells?

Fuel resistant, chemically inert

Proton conduction

Both A and B

1. What is varied in the libraries designed for proton exchange membranes?

Composition library of membrane components

Structure library of each membrane components

Temperature gradient library of drying membranes

Blending rates of membrane components

1. Which of the following comments on AC impedance spectroscopy is false?

AC impedance spectroscopy is used to test conductivity of films.

Impedance depends on film thickness

AC impedance spectroscopy need alternating current.

Impedance can’t be converted to resistance or capacitance.

1. HTC screening studies of PVDF/SEM-HEMA membranes indicates the strong influences of Kynar type. How correlations in structure-property relationship is discovered?

Lower viscosity of inert polymer showed higher conductivity.

Higher crystallinity of inert polymer exhibited lower conductivity.

Higher crystallinity of inert polymer exhibited higher conductivity.

Lower viscosity of inert polymer showed lower conductivity.

# Structural Alloys for Energy and Transport

1. What strengthening mechanism is generally most effective in producing alloys capable of withstanding high temperatures?

Work Hardening

Transformation strengthening

Grain boundary strengthening

Particle strengthening / Precipitation hardening

1. Precipitate nucleation and growth is more likely if free energy of compound formation is less than free energy of mixing.

**True**

1. Which of these influence whether an intermetallic phase will be formed?

ΔH*f*AxBy​

ΔHmix​

ΔSconf​

Atomic Radius

1. Which of the statements about the assessment of alloys is incorrect?

Evaluations are conducted in a multiple stages.

Accuracy of the evaluations increase with each stage.

The goal is to rapidly characterize as many system of elements with potential to be developed into alloys.

In this process, it is more detrimental to discard systems with development potential than to retain systems with no potential.

1. After you have identified the palette of elements and have screened using high-throughput computational calculations, what is the next step in the HTMD strategy for alloy development?

Generate alloy libraries with microstructure gradients, characterize, and screen.

Conduct more detailed computationally expensive calculations to further refine screening.

Generate alloy libraries with composition gradients, characterize, and screen.

Targeted detailed study on the most promising compositions and process routes.

1. Which stage of the high-throughput evaluation process involves calculation of phase equilibria for any given system of elements?

Step 1: Identify palette of elements.

Step 2: Screen using high-throughput computational calculations.

Step 3: Generate alloy libraries with composition gradients, characterize, and screen.

Step 4: Generate alloy libraries with microstructure gradients, characterize, and screen.

Step 5: Targeted detailed study on most promising compositions and process routes.

1. In "Step 1: Identify palette of elements,", what conditions are criteria for selecting the

Phases at the desired operating temperature must be the same as that at room temperature.

Alloy must be a single phase above the operating temperature in order to allow for precipitation hardening at <{\text{T}\_{\text{use}}}Tuse​

More than two phases may exist.

Elastic modulus and density, determined by rule of mixtures, must agree with the values for the alloy being considered.

1. Near the melting point, configurational entropy primarily depends on the gas constant and what?

Atom fraction of each element

Atomic number of each element

Size of each element

Mass fraction of each element b

1. The likely best step to evaluate the oxidation resistance in the HTMD strategy for high temperature alloy development is:

Step 1: Identify palette of elements.

Step 2: Screen using high-throughput computational calculations.

Step 3: Generate alloy libraries with composition gradients, characterize, and screen.

Step 4: Generate alloy libraries with microstructure gradients, characterize, and screen.

1. Examples of high-throughput computational calculations used for screening include:

Calculation of the lowest temperature that gives a single phase solid solution

Calculation of Gibb's free energy for compound formation

Calculation of the crystal structure

Calculation of elastic modulus

Calculation of density

Calculation of the number of possible combinations of elements

# Exploration of PSP Linkages in Dual Phase Steel

1. Which statement is true?

Different processing paths result in different microstructures and final properties because processing, structure, and properties are interconnected.

Different processing paths do not always change the microstructure but can result in different properties.

1. In the HT indentation testing, spherical indenter was used instead of a sharp indentation because (select all correct answers):

An indentation stress-strain curve can be obtained that includes elastic modulus, yield strength, and hardening rate.

The dual phase steel is brittle and use of sharp indenters would have caused cracking.

An indentation yield strength can be obtained which tends to be more sensitive to the heat treatment process.

1. Which are true statements regarding indentation measurement on the dual phase steel considered in this case study?

Bake hardening improves strength of dual phase steel.

The higher amount of martensite as a hard phase improves strength of dual phase steel.

Strength is the only property of dual phase steel that can be measured from indentation.

It is a sensitive test that can capture changes in properties of the steel with different amounts of martensite and cold work.

1. Choose the statements that are true for high-throughput prototyping protocols:

Accelerating development of a material system requires more effort to achieve the answer.

Material libraries that have a gradient of processing conditions can be easily assayed using indentation testing since the probed material in this technique is very small and multiple tests can be carried out along the gradient.

High-throughput prototyping goals are minimizing the effort and material in both mechanical testing and microstructure characterization.

1. In this case study, the first two PCs, PC1 and PC2, correlated well to (select all correct answers):

intercritical annealing temperature

amount of plastic deformation (cold work)

temperature of bake hardening

time of bake hardening