3.044 Final Exam

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Guidelines:

- This is a 3-hour final examination, with a closed book portion (Part A) and an open book portion (Part B).
- You will receive *both* Part A *and* Part B of the exam at the beginning of the three hours. You may (and are *strongly* advised to) look over the entire exam to decide how to partition your time. You *must*, however, work on the closed book portion first, and turn in your answers before you open any reference materials for the open book portion.
- You may use a calculator and the attached equation sheet in part A.
- You may use any non-human resources in part B (in the medical sense, not the philosophical sense), except for classes of devices which can communicate (*i.e.* no cell phones, wireless-networked PDAs, or laptops—with or without wireless networking).
- You may answer the questions within each part of the exam in any order you like.
- Start answering each question on a fresh sheet of paper. Read the questions carefully before attempting to answer them. Please write legibly.
- Write your name on every answer booklet that you hand in.
- Graded exams and final grades will be available by May 23 (at the latest). If you wish to have these mailed to you, provide an address (U.S. Mail or interdepartmental) on the front cover of any of your exam answer booklets. If you do not specify otherwise, I will hold on to it and assume you will pick it up at some point. (Note: Interdepartmental mail is notoriously unreliable to FSILGs—three years ago, a student with questions about his December final didn't get it from interdepartmental mail until May!)
- I hope you do well. If everybody gets above a 90 average, I will give out a lot of As!

Sorry, no points for your names this time.

Part A: Closed Book

1. Process selection (45 pts)

   In each of the following cases, two alternative processing options are presented to accomplish a goal. Your task is to (1) present and explain one advantage of each option (about one sentence each), and then (2) make a recommendation—which might not be the best choice in all circumstances, but describe in one sentence why it is the best for a particular application.

   (a) As the final step in making an integrated circuit, nickel “bumps” are deposited which will later be used to solder connections to a package or board. Candidate processes for making the nickel bumps are:

   - Electroless nickel reduction from aqueous solution.
   - Electron beam evaporation/physical vapor deposition.

   (b) Oxide-conducting fuel cells made of yttria-stabilized zirconia (YSZ) can use any oxidizing fuel without reforming. Their anodes are made of a nickel-YSZ metal-ceramic composite (a “cermet”), at which fuel vapor reacts with oxide ions according to, for example:

   $$CH_4 + 4O^{2-} \longrightarrow CO_2 + 2H_2O.$$

   Note this reaction can only take place at the vapor-metal-ceramic triple boundary, and the nickel and YSZ phases must be continuous. Candidate processes for making the metal-ceramic composite are:

   - Sintering together powders of nickel and YSZ.
   - Vapor deposition (CVD or PVD) and patterning to form YSZ-nickel structures engineered for high mass transfer coefficient.

   (c) Titanium-magnesium alloys are not yet out of the laboratory, but might become high-performance aerospace structural materials. (On the magnesium-rich end, titanium adds corrosion resistance; on the titanium-rich end, magnesium lowers the density and offers opportunities for strengthening by internal oxidation, resulting in MgO particles.) Candidate processes for making sheets of these alloys are:

   - Cast a slab or plate, then hot/cold roll it into sheet.
   - Physical vapor deposition (from two separate sources).

   (Hint: consider their melting and boiling points.)

   (d) An aluminum automobile engine intake valve must meet tight tolerance and finish specifications. For higher performance, valves can be made “hollow”, meaning essentially with a hole drilled in the back. Candidate processes for making aluminum valves (which will later be machined for finish in critical areas) are:

   - Lost wax investment casting (injection mold wax, coat with ceramic and binder, melt out wax, fire/sinter ceramic, pour liquid aluminum in ceramic mold).
   - Die casting (inject aluminum into metal mold).

   (e) A fuel cell capable of propelling a vehicle is made up of stacks of individual reaction cells. Each cell is separated from the next by a layer which serves as the anode of one cell and the cathode of the next—the bi-polar plate. This layer must povide both structure and conductivity. Candidate materials/processes for the plate are:

   - Stamped sheet metal.
   - Carbon-filled polymer molding, a thermoplastic composite casting process.

2. Application of process maps (30 pts)

   Using process maps alone, rule out as many processes as possible for manufacturing the following parts (6 pts each):

   (a) Large iron boat anchor.
   (b) Small complex nylon valve casing.
   (c) Ceramic "microturbine" 5 mm across.
   (d) Complex steel piston rod for a large diesel engine.
   (e) Very thin polymer sheet for a laptop microprocessor chip package.

3. Tape casting aluminum nitride chip packages (25 pts)

   Aluminum nitride is a good conductor of heat which is used for large chip packages in desktop and server computers. The tape casting process for making the square package involves several steps, which happen in a continuous line:

   - Extrude thick slurry of AlN powder and binder onto a moving substrate to form the "tape".
   - Pyrolize (burn off) the binder at medium temperature.
   - Sinter the powder at high temperature

   (a) Sketch the velocity profile of the pseudoplastic slurry in a flat channel-shaped extrusion die, assuming power law behavior with $n = 0.7$. (5)
   (b) When the slurry leaves the die, it will have uniform velocity. Given the change in velocity profile, what defects might be introduced at the die exit, and how might they be prevented? (5)
   (c) Sketch the density of AlN (relative to its full density) vs. time as it goes through sintering, identifying the stages of sintering. (5)
   (d) Assume the dominant sintering mechanism in Stage I is bulk diffusion from the grain boundary to the neck. Does this mechanism result in densification? (4)
   (e) Larger powder size leads to larger grain size, and better thermal conductivity. On the other hand, if the powder size doubles, how much longer will it take to get to the same dimensionless neck radius x/r? (6)

Helpful equations for Part A

- Power law model of non-Newtonian flow behavior:

$$\tau_{yx} = \mu_0 \left(\frac{\partial u_x}{\partial y} \right)^n$$

- Velocity profile of a power-law non-Newtonian fluid in a channel:

$$u_x = -\frac{n}{n+1} \left(-\frac{\Delta P}{\mu_0 \cdot L} \right)^{\frac{1}{n}} \left[\left(-\frac{\delta}{2} \right)^{\frac{n+1}{n}} - y^{\frac{n+1}{n}} \right]$$

- Sintering neck radius x vs. time t and particle size r for bulk diffusion from grain boundary to neck:

$$\frac{x}{r} \propto r^{-3/5} t^{1/5}$$

Process Maps

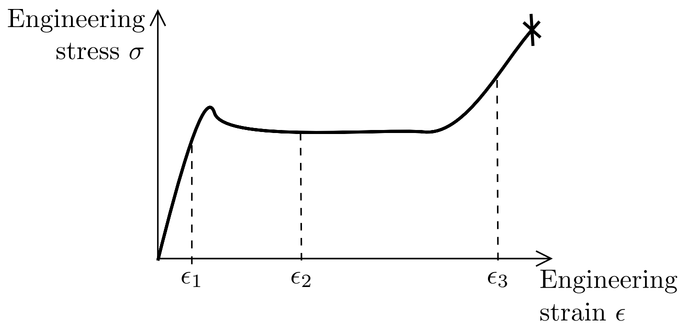
Figures removed for copyright reasons.

Source: Figure 12.3, 11.31, 11.32, and 11.33 in Ashby, M. F. *Materials Selection in Mechanical Design*. Boston, MA: Butterworth-Heinemann, 1999. ISBN: 0750643579.

Part B: Open Book

1. Forming a PETE package (32 pts)

   Poly(ethylene terephthalate) (PETE) is an easily-formed, somewhat viscoelastic, and relatively easy to recycle polymer used in many forms of packaging. A thick PETE sheet is expanded into a large mold using high pressure gas, and deforms to relatively high strain. You can consider the shape of the PETE sheet to be a hemisphere which expands throughout the process.

   The stress-strain curve for PETE is shown above. For each of the three different extents of strain indicated on the curve as ϵ_1, ϵ_2 and ϵ_3, answer the following three questions:

   (a) Is the expanding sheet stable (does it maintain roughly uniform thickness) at this strain? (2 pts each)

   (b) Sketch the shape and approximate thickness distribution you expect to see at this strain. (4 pts each)

   (c) Sketch and/or describe the polymer conformation(s) (that is, arrangement of molecules) you expect to see at this strain. (3 pts each)

   At the end of the process cycle, the sheet covers the inner surface of the mold, then the gas pressure is released, the mold opens and the package is ejected.

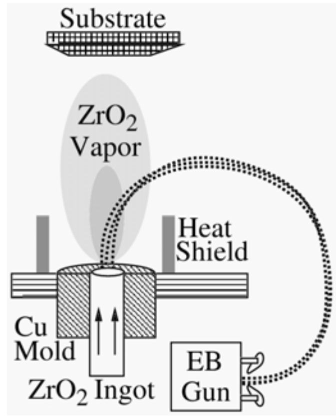
   (d) The final size of the PETE package is somewhat smaller than that of the inside of the mold. Why do you think this happened? (5)

2. Patterning in a chemical sensor (11 pts)

   A sensor for various chemicals has two main “parts”: a small and very detailed array of sensor molecules which self-assemble into trenches 10-20 nm across, and a larger set of wires connected to relatively simple logic circuits which do not need to be smaller than 1 micron across. The goal is to fabricate both of these “parts” on a single substrate to simplify manufacturing and packaging.

   Briefly describe the benefits and drawbacks of using only electron beam lithography, only ultraviolet lithography, and some combination of the two in order to accomplish this goal.

3. Electron beam physical vapor deposition of zirconia (32 pts)



Electron beam physical vapor deposition is commonly used to make yttria-stabilized zirconia thin films for applications from thermal barrier coatings to anti-reflective optical coatings.

Data:

- ZrO_2 melting point: 2720°C.
- ZrO_2 vapor pressure: 0.1 Pa at 2110° C, 1 Pa at 2450° C, and 10 Pa at 2690°C.

(a) Extrapolate the above data to estimate the vapor pressure at the (optically) measured pool surface temperature of 2750°C. How accurate do you think this extrapolation will be? (5)

(b) Estimate the ingot feed velocity required to keep the liquid pool top surface at a constant level while evaporating at 2750°C. (Develop your answer based on evaporation of pure ZrO_2.) (6)

(c) Using 6Å as the collision diameter and a source diameter of 5 cm, estimate the vapor plume cosine power n where condensation flux at substrates facing the source is given by $J \propto \cos^n \theta$. (5)

(d) The process starts with uniform ingot composition of 10 wt% Y_2O_3, then the Y_2O_3 concentration in the liquid decreases to 6 wt% at steady-state as yttria evaporates faster than ZrO_2. What is the approximate yttria fraction of films deposited at the original liquid composition of 10 wt% Y_2O_3? (5)

(e) The liquid pool is well-mixed but very shallow, only about 5 mm deep. Approximately how long does it take for the composition to change from the original composition to the steady-state? (6)

(f) For ceramic thermal barrier coatings on metal substrates, because the metal thermal expansion coefficient is larger than that of the coating, a porous structure with somewhat separate columnar grains is desired. (This is because a non-porous ceramic coating on a metal would crack during repeated heating and cooling; if the film already has somewhat separate columnar grains, they can separate slightly during heating instead of cracking.) Roughly what is the maximum substrate temperature at which such a structure can be produced? (5)

4. Essay: Stability and materials processing (25 pts)

Stability analysis is a rich subject with important implications for materials processes, which was discussed in a very qualitative way in 3.044. On the one hand, instability leads to the annoying roughness on metal plating or tearing during sheet forming; on the other, it forms the complex spherulitic or dendritic structures which lead to strengthening.

Select and write about a process in which stability plays a role. Explain why a phenomenon in the process is stable or unstable, if possible appealing to general principles such as limitation by diffusion ahead of or behind a moving interface. In particular, discuss how the need to either suppress or promote unstable behavior guides the design of the process.

(5 pts writing, 5 pts organization, 5 pts process description, 10 pts stability discussion.)