

TEACHER INSTRUCTIONS

Sintering: Grain Boundaries, Interfaces, and Porosity

Objective: To demonstrate how individual particles and powders can be processed and formed into large solid objects and investigate the material concept of porosity.

Background Information: All materials must be processed in some way to achieve either a particular material property or create a specific shape. There are many different material processing techniques, but one that is fundamental to ceramic materials is sintering. *Sintering* is a processing technique by which a solid mass of material is formed from the fusion of many smaller pieces, often a powder. This is done primarily through the application of heat but sometimes by adding pressure concurrently. It is unique in that sintering is a solid state process, unlike other common material shaping techniques that require melting the material to pour into a mold, followed by cooling. The typical temperatures used for sintering are around $2/3$ of a material's melting temperature, making it ideal for materials with extremely high melting points. Sintering has been used for centuries in the construction of pottery by ancient civilizations. However, recently, the advent of nanotechnology has used nanoparticles to create materials with novel and unique properties.

Sintering is effectively a process where *porosity*, i.e. open space, is removed from compacted powder particles to form a solid mass. This is a means of *densification* since you are decreasing the amount of volume that a given amount of mass takes up. Material moves to the contact points between particles and fills in the open space. This accumulation of material at pores causes contact points to extend and become *interfaces* between grains, commonly called *grain boundaries*. This process is outlined in Figure 1 with a point contact between particles, marked by a red dot before sintering, becoming a grain boundary after sintering (the red line). Material moves to pores by *diffusion* facilitated by high temperatures.

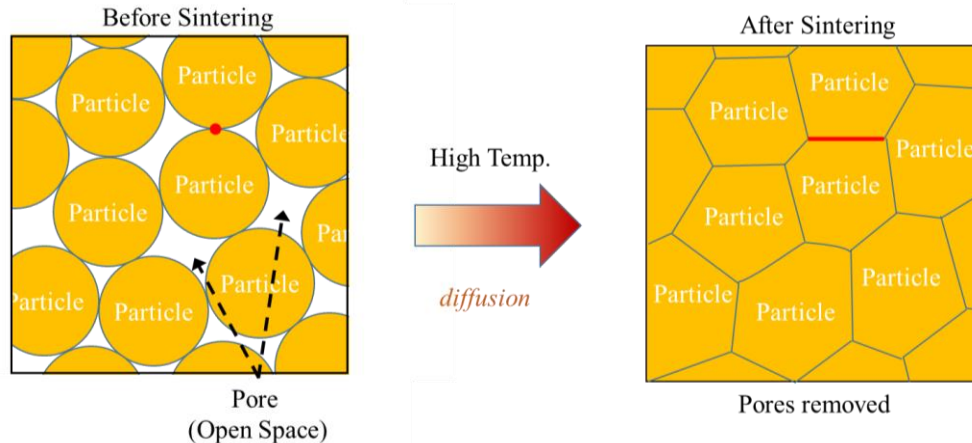


Figure 1: During sintering, high temperatures allow material to transport to the open space between powder particles. Eventually, the pores disappear resulting in a dense pore free object.

Why do the pores shrink in the first place? The pores shrink by material diffusing to the empty space creating grain boundary interfaces, which is driven by energy.

Interfaces can be described in terms of energies because work should be applied to create the new surface area. For example, when you blow through a ring with a soap film to make a soap bubble, the blowing is work done on the soap film, resulting in the flat soap film bending out, creating a bubble. As the bubble grows, it increases its surface area and eventually becomes a sphere, as shown in Figure 2. The Greek symbol γ (gamma) is often used to denote the energy per unit area for surfaces in J/cm^2 . If you have not met or exceeded the energy needed to make a spherical bubble (by blowing hard enough), the shape will go back to being flat when you stop adding work (stop blowing).

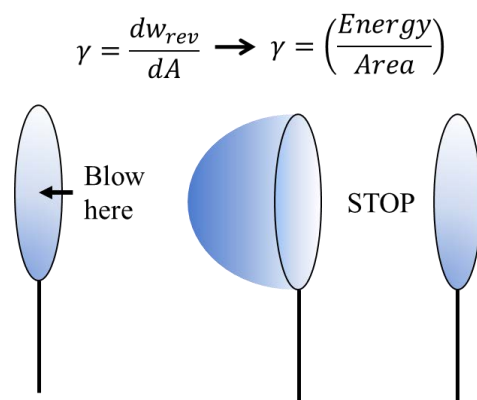


Figure 2: In order to create a surface, work must be done over that area, just like when blowing a bubble.

Title: How does the sintering process affect the porosity?

Lab Description: In this lab, polyester beads are used as an analogy to demonstrate the process of sintering. Each polyester bead represents an individual powder particle. As the particles bond together in hot water, a porous solid is created which can be cut or broken to investigate its porous structure.

Keywords:

Interface: a boundary defining a change in a material's chemistry, type, structure, etc.

Grain: small individual 'pieces' of a larger portion of material.

Grain Boundary: interface between two grains.

Porosity/Pores: the open space between grains.

Densification: a means of densifying a material; the volume that material takes up is decreased while the mass stays the same.

Microstructure: structure of a material as observed through microscopic examination.

Diffusion: movement of atoms from one location to another due to some driving force, such as high temperatures.

Materials List:

1 – 12 oz container of InstaMorph Moldable Plastic beads (10 grams/experiment)

Juice glasses, beakers, test tubes, or other clear containers – 1 per group to mix the hot water and InstaMorph beads.

Pot holder/glove – a glove to protect hands from the hot water.

Pen, Pencil, Fork/tongs, etc. – 1 utensil per group for retrieving beads from water.

Paper towels or rags – for placing beads onto and absorbing spilled water.

Scissors, kitchen shears, or tin snips – for cutting the sintered body in half to examine the pore structure.

Water – for sintering the submerged beads together.

Hot plate, stove, Bunsen burner, etc. – Something to heat water to the desired temperature.

Thermometer – for measuring the temperature of the water as it is heated.

Rulers or micrometers – for measuring pore sizes and other physical dimensions.

Safety Precautions: It is recommended that a glove or pot holder be worn by students when retrieving the beads from the hot water to prevent any possible burns. Lab glasses should be worn by anyone nearby the shears cutting the sintered mass to protect eyes from plastic pieces. Depending on the age of the students, the teacher could do the cutting for the students. Furthermore, standard lab rules and procedures (e.g. using the items as described in the handout, not for any other purposes) should be followed.

Optional Demonstration (to perform before experiment):

An unglazed (not shiny) ceramic pot -gardening pots are excellent for this- can be used to demonstrate the concept of porosity in a real world ceramic. The day before you plan to do the experiment, fill the unglazed pot with water and have the students record the height of the water line. Let the pot sit overnight with the top covered by plastic food wrap or the like, and have the students record the height the next day that they come to class (it will be less because some of the water is absorbed into the pores in the pot). Discuss with the students why they think the water line went down, and where the water could have gone.

Instructions:

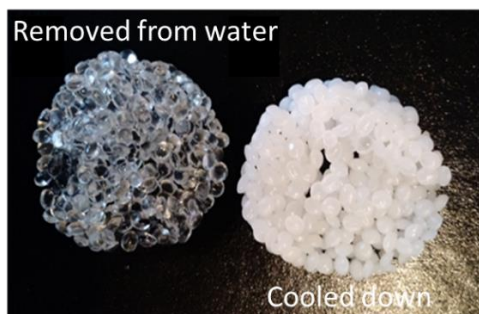
1. Discuss with the students the basic equations discussed in the supplementary information.
2. The students should weigh 10 grams (g) of InstaMorph beads. If the beads are unable to be weighed, the amount of beads needed for 10g can be calculated (refer to supplementary information) [estimated time to complete: 10-15 minutes].
3. Heat enough water (1/4 cup for each group) to *at least 150°F (65.5°C)* for students to submerge their beads [time estimate: ~5 min. – can be done before class starts].
 - Water can be heated in whatever way is most convenient (stove, microwave, hot plate, electric water kettle, etc.).
 - To keep InstaMorph moldable for longer and to speed up the bonding/sintering process, the water can be heated above 150°F. CAUTION: This will increase the

temperature of the beads and increase the chance for burns or other heat related injuries.

- Either the teacher can heat the water and add it to student's beakers, OR if the teacher deems it appropriate, each group can heat the water themselves if there are enough heat sources, heating vessels, thermometers, etc.
4. Students put the 10g of InstaMorph beads into a clear glass container, and either the students or the teacher pour enough hot water to cover them.
- The container should not be wider than 1.5 inches so there are multiple layers of beads on top of one another. Larger diameter containers will require larger amounts of beads.
 - i. Consequences of a thin layer of beads are that the fracture surface for the end of the lab will not have sufficient enough area to measure or clearly see the pores.
 - The water cools quickly below the activation temperature of the beads, keeping them from bonding, so do not delay the transfer from the heat source to the beads.
5. Wait for the beads to change from opaque to clear as shown below [time estimate: < 10 min. – depends on temperature of water]:
- Have students record their observations.



6. Being careful not to disturb the bead mass too much, remove the now “sintered” beads from the water using the pen or fork and place them on a paper towel to cool and drain [time estimate: < 5 min.]. Once cooled, the beads will return to an opaque color, as shown below.
 - Cooling can be accelerated by holding it under a cold running faucet or placing it in a cool water bath.

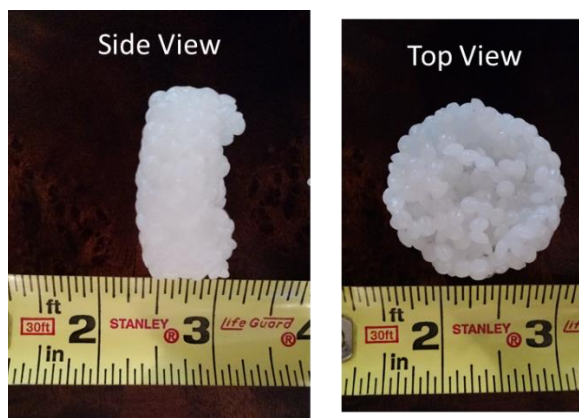


7. Once cool enough to touch, task the students with examining the sintered mass and recording their observations of its structure, strength, rigidity, etc.
8. Have the students calculate the percent porosity and/or percent theoretical density using the procedure outlined in the Student Handout.

9. Then use the scissors or shears to cut the porous body in half.
 - These beads are remarkably strong when densely bonded together, in which case regular, small scissors may not be sufficient to cut them. If the mass is thin or did not bond well, it can be quite brittle and easier to cut through. This will vary experiment to experiment.
10. Have students examine the microstructure of the cut face, looking for pores.

Lab Delivery Hints:

1. This lab is best done in groups of 2-3 students. Together the group can weigh the beads or work on estimating how many beads are needed if unable to weigh them.
2. There is more than one brand of this type of polymer bead. InstaMorph, shown in this document was purchased from Amazon.com but there are also colored InstaMorph beads as well, if desired.
3. The container should not be wider than 1.5 inches so there are multiple layers of beads on top of one another. Larger containers will require larger amounts of beads. Using a 1.5 inch diameter container will result in a 10 gram mass of approximate dimensions shown in the image below:



4. Use glass containers – the beads can bond to plastic containers making clean up more difficult.
5. Remind students to be careful not to smash/mush the beads when removing them from the water so pores don't collapse.

Troubleshooting: The time it takes for the beads to become clear and bond together is dependent on the temperature of the water, which can cool rapidly when heat is turned off, so water may need to be reheated multiple times if students are waiting, or heated above the target temperature in anticipation of it cooling. 170-175°F makes the beads bond quickly and allows time to transfer water from the heater to the vessel containing the beads.

If it is taking too long to bond the beads and become clear, increase the temperature of the water to around 170-180°F, but remember the considerations above. This should cause the beads to bond and become clear in under 1 minute and allow more time to get the water into student's vessels.

*****Wear appropriate safety equipment to prevent any burning from the hot water*****

Cleanup/Replacement parts: The beads are not reusable experiment to experiment. However, they can be heated and remolded if the students want to take them home. Otherwise, they should be thrown away. Ensure the water has been dried from the tools before storage.

TEACHER DISCUSSION QUESTIONS

Sintering: Grain Boundaries, Interfaces, and Porosity

Discussion Questions to Ask Before the Lab

1. Ask students what type of things they can think of that may be created using sintering or powder processing.

Discussion: Remind them that it is bonding of a powder by applying heat and sometimes pressure. During winter, making a snowball is a direct application of sintering that most people have experience with. Pressing snow between your gloves to make a snowball applies pressure as well as heats the snow. The contact points between the small snow particles melt and refreeze and the snowball bonds together.

It is common that people say pottery or coffee cups but you should emphasize that it isn't just those things that are made via sintering. Most people think of those examples as ceramics, but many other functional materials use powder processing techniques. 3D printing of powdered metals is becoming a rapidly utilized process due to the ability to create complicated shapes. There is a 6 minute video showing such uses on YouTube: <https://www.youtube.com/watch?v=cRE-PzI6uZA>.

Here are a few other examples of objects that are a direct result of sintering that students may know a few of: Teflon non-stick pan coatings, protective body armor and bulletproof windows, jet engine turbine blades and other parts that operate in high temperature environments, solar cell (photovoltaic) thin films, radioactive material containment, fuel cell components, kitchen cooktops, and bioactive scaffold implants for tissue and bone growth.

2. Ask students what they think the benefits of the “near net shape” forming ability of sintering might be (ie: the ability to make the object before sintering close to the shape you want after sintering).

Discussion: Provide the example of a clay pot; it looks like a pot when the clay is wet and molded, which is before sintering. After sintering, the shape still looks like the same pot, just slightly shrunken due to densification and the removal of pores. This is near net shape processing.

One benefit is the initial production shape of the part before sintering is very close to the final (net) shape, so the need for final finishing or shaping steps is reduced. Removing final steps such as grinding or cutting can eliminate production costs and reduce the time needed to make the object. In many cases, ceramics are very difficult, or impossible to cut after sintering (due to their high hardness), so near-net shaped processing can be the only way to produce complex shapes.

3. Are there any difficulties associated with the near net shaping of objects?

Discussion: Remind the students that you start with a porous object and through heating, the object shrinks and becomes denser. What would happen if the object was very complicated and had areas of varying thickness? One common difficulty is the less uniform a shape is, differences in how the object shrinks can occur. The pieces that are thin may shrink more than the thicker, larger pieces of the same object making the object distorted.

Discussion Questions to Ask After the Lab

1. Have students think of different ways things can be heated. What methods would they think would work better for reducing porosity of a part and why? Remember that ceramics and metals that are processed with sintering have very high melting temperatures.

Discussion: There are many different methods to heat an object: ovens, fire, microwaves, friction, etc. Some of the methods such as ovens are better at getting the powder uniformly hot for even sintering and porosity reduction. Others, such as fire, heat locally, in a small area. That area may sinter, but the rest would not.

Students can relate to microwaves, which are exactly like the ones in their kitchens. They should think about whether or not it would work for ceramics. Do coffee mugs get hot? Usually not, but the liquid inside does. Microwaves emit high frequency radio waves, at just the right frequency that they preferentially interact with water, fat, and sugar molecules within organic matter (food) to cause bond vibrations, which cause the increase in temperature. This is why some plastics, also made of organic material, are not “microwave safe.” It is more difficult to heat up ceramics and glasses because the atoms are more restricted and bonded tightly to one another in these materials.

2. Ask students how the performance of a ceramic material would change if it wasn't sintered to full density (i.e. if there were pores left). Would it be stronger or weaker? Are there any advantages to having a structure that purposely has pores in it?

Discussion: When ceramics are used for structural (buildings, parking garages, etc.) or high impact (body armor, turbine blades, etc.), incomplete sintering (pores left in the material) would cause a decrease in performance – they would be weaker. The pores would act as weak spots in the material where cracks could easily propagate. Sometimes ceramics are made to be purposely porous (contain many pores, often in a uniform arrangement). The pores can act as a pathway between the material for things like water (ceramic filters-like those found in your filtered water bottles), ions (ceramic solid electrolytes in batteries), etc. Porous ceramics are also used to mimic the material in your bone.

SUPPLEMENTARY INFORMATION

Equations:

$$A_{circle} = \frac{\pi}{4} d^2$$

Equation 1: Area of a circle in terms of diameter.

$$2r = d$$

Equation 2: The relation of a circle's radius (r) to its diameter (d).

$$V_{bead} = \frac{1}{6} \pi d^3$$

Equation 3: Volume (V) of a single bead of diameter (d) by combining Equation 1 and Equation 2.

$$V_{total} = V_{bead} \times n$$

Equation 4: The total volume (V_{total}) is equal to the 'n' number of beads times the volume of an individual bead, V_{bead}

$$n = \frac{6}{\pi d^3} * \left(\frac{m}{\rho_{bead}} \right)$$

Equation 5: The number of beads (n) of density (ρ) and diameter (d) needed to generate a desired mass (m) can be calculated. This assumes spherical particles.

$$\rho_{bead} = 1.37 \text{ g/cm}^3$$

Density of a polyester thermoplastic bead¹.

$$A_{total_pores} = A_{avg_pore} \times n_{total_pores}$$

Equation 6: The estimated total area of pores is the average area of a pore (A_{avg_pore}) multiplied by the total number of pores on the face (n_{pores}).

$$\% \text{ Porosity} = \frac{A_{total_pores}}{A_{bulk}} \times 100$$

Equation 7: The estimated percent of the face that is pores by using the total average porous area (A_{total_pores}) to the area of the bulk solid (A_{bulk}).

$$\% \text{ Density} = 1 - \% \text{ Porosity}$$

Equation 8: The estimated density of the solid based on how much open space (pores) exists.

The Equation 5 can be used to calculate the number of beads needed for 10 grams if the student cannot weigh it.

¹ British Plastics Federation, Plastipedia: *Polyesters (Thermoplastic) PETP, PBT, PET*
<http://www.bpf.co.uk/plastipedia/polymers/Polyesters.aspx>

STUDENT LAB HANDOUT

Sintering: Grain Boundaries, Interfaces, and Porosity

Background Information: All materials must be processed in some way to achieve either a particular material property or create a specific shape. There are many different material processing techniques, but one that is fundamental to ceramic materials is sintering. *Sintering* is a processing technique by which a solid mass of material is formed from the *fusion* of many smaller pieces, often a powder. This is done primarily through the application of heat but sometimes by adding pressure concurrently. It is unique in that sintering is a solid state process, unlike other common material shaping techniques that require melting the material to pour into a mold, followed by cooling. The typical temperatures used for sintering are around 2/3 of a material's melting temperature, making it ideal for materials with extremely high melting points. Sintering has been used for centuries in the construction of pottery by ancient civilizations. However, recently, the advent of nanotechnology has used nanoparticles to create materials with novel and unique properties.

Sintering is effectively a process where *porosity*, i.e. open space, is removed from compacted powder particles to form a solid mass. This is a means of *densification* since you are decreasing the amount of volume that a given amount of mass takes up. Material moves to the contact points between particles and fills in the open space. This accumulation of material at pores causes contact points to extend and become *interfaces* between grains, commonly called *grain boundaries*. This process is outlined in Figure 1 with a point contact between particles, marked by a red dot before sintering, becoming a grain boundary after sintering (the red line). Material moves to pores by *diffusion* facilitated by high temperatures.

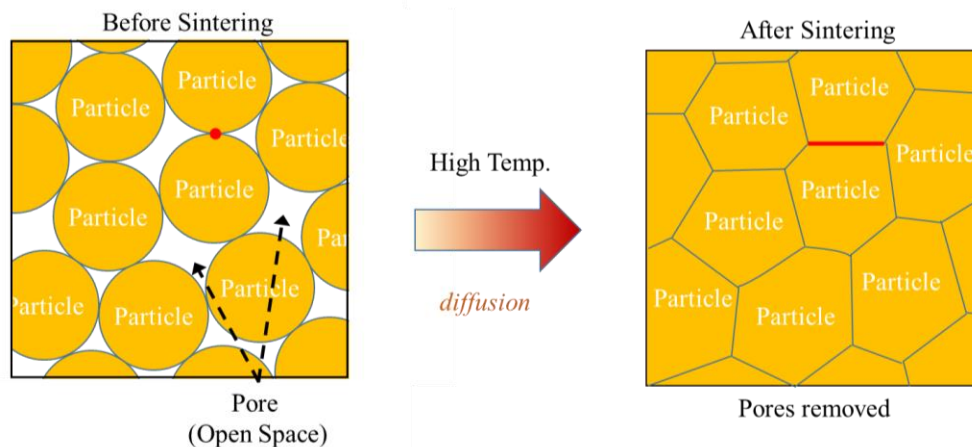


Figure 3: During sintering, high temperatures allow material to transport to the open space between powder particles. Eventually, the pores disappear resulting in a dense pore free object.

Why do the pores shrink in the first place? The pores shrink by material diffusing to the empty space creating grain boundary interfaces, which is driven by energy.

Interfaces can be described in terms of energies because work should be applied to create the new surface area. For example, when you blow through a ring with a soap film to make a soap bubble, the blowing is work done on the soap film, resulting in the flat soap film bending out, creating a bubble. As the bubble grows, it increases its surface area and eventually becomes a sphere, as shown in Figure 2. The Greek symbol γ (gamma) is often used to denote the energy per unit area for surfaces in J/cm^2 . If you have not met or exceeded the energy needed to make a spherical bubble (by blowing hard enough), the shape will go back to being flat when you stop adding work (stop blowing).

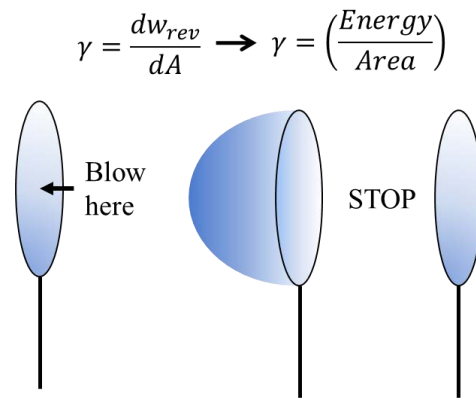


Figure 4: In order to create a surface, work must be done over that area, just like when blowing a bubble.

Title: How does the sintering process affect the porosity?

Lab Description: In this lab, you will explore the material processing technique *sintering* using plastic beads and analyze the resultant porous microstructure.

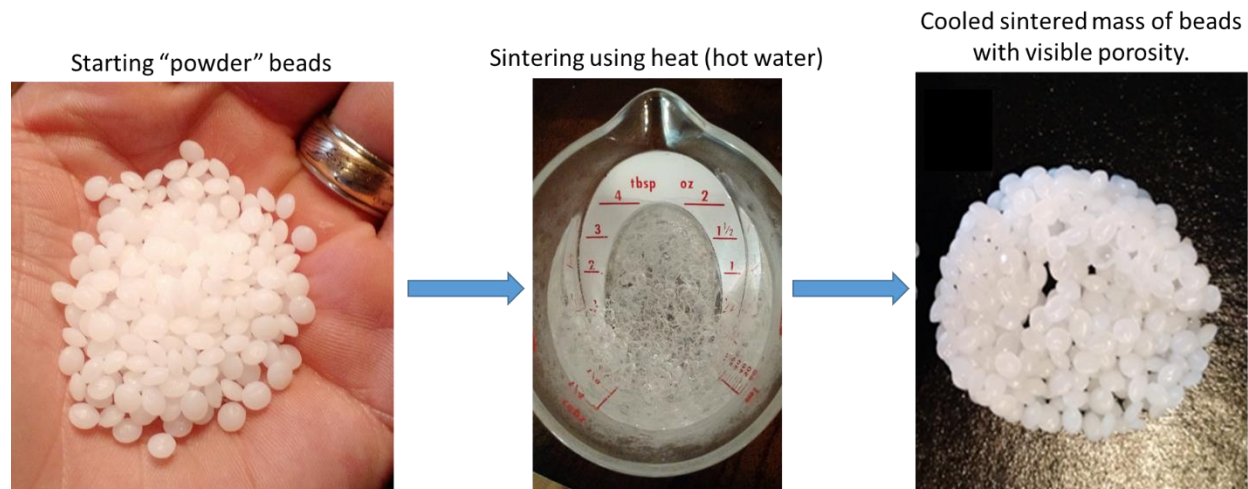


Figure 3: This lab begins with loose particles that, through the application of heat in the form of hot water, are sintered and bonded together. The resultant microstructure will be analyzed.

Keywords: Grain, Grain Boundary, Interface, Surface Energy, Diffusion, Porosity, Microstructure

Materials List:

10 grams InstaMorph beads
Enough hot water (~150°F) to just cover the beads
Beaker or container for the beads and water
Utensil to retrieve the beads from the water
Insulating gloves (hot-mitt)
Paper towels for cleaning water
Thermometer to monitor water temperature (*optional*)
Hot plate to heat water to 150°F (*optional*)

Safety Precautions: When retrieving the mass from the hot water, be sure to wear a thermally insulating glove to protect your hand from burns or other heat related injuries. Standard lab rules and procedures (only using the equipment as indicated in the instructions) should be followed.

Instructions:

1. Ensure you have all required materials to perform the lab.
2. Review the questions in the student handout and data sheet to get an idea of what you need to pay attention to and record.
3. Weigh 10 grams of InstaMorph beads and put them into a clear glass container.
4. Using a thermometer to monitor water temperature, heat up the water to 150°F or the instructed temperature determined by the teacher.
 - a. Be sure to put on heat insulating gloves to protect your hands from any potential burns or heat related injuries while handling the hot water.
5. Make sure the experimental setup is ready and quickly transfer the heated water to the beaker containing the polymer beads. Pour enough water into the beaker so that the beads are completely covered.
6. Record the time needed for beads to turn clear and any observations during the sintering process.
7. When the beads are completely clear, carefully remove the sintered mass from the water. Being careful not to splash any hot water, using a pencil, fork or similar tool, place the mass on a paper towel to drain.
 - a. *CAREFUL*: don't squish the beads too much when retrieving them from the water.
8. Examine the sintered body and estimate what you think the percent of the material is open space (pores) vs. material.
9. Once it is cool enough to handle (beads turn back to being opaque), record any observations on the material's apparent structure, strength and rigidity properties as compared to the mound of beads you weighed out previously.
10. Approximately measure the pore diameters, number of pores, and the height and diameter of the surface of the sintered mass (should be close to the diameter of your container); then record the values onto the data sheet and calculate the percent porosity.

- a. First measure the diameter of the surface and calculate the area of the bulk material. Use Equation 1, below.

$$A_{bulk} = \frac{\pi}{4} d^2$$

Equation 1: The area of the surface including pores by measuring the diameter, approximating a circle.

- b. Count how many pores and record (n), and then measure the diameter of as many of the pores as you can, based on your judgment.
- c. Calculate the areas of pores considering a circular shape and average the values to get a representative area.
- d. Multiply that average pore area by the total number of pores you see (n) on the surface to get a total pore area using equation 2.

$$A_{total_pores} = A_{avg_pore} \times n_{total_pores}$$

Equation 2: The estimated total area of pores is the average area of a pore (A_{avg_pore}) multiplied by the total number of pores on the face (n_{pores}).

- e. Calculate the porosity percentage, which will be the ratio of porous area (part d) to the bulk area (part a) described by Equation 3.

$$\% \text{ Porosity} = \frac{A_{total_pores}}{A_{bulk}} \times 100$$

Equation 3: The estimated percent of the face that is pores by using the total average porous area (A_{total_pores}) to the area of the bulk material (A_{bulk}).

- f. It may be easier to think of it terms of density, which is described by Equation .

$$\% \text{ Density} = 1 - \% \text{ Porosity}$$

Equation 4: The estimated density of the material based on how much open space (pores) exists.

11. Cut the sintered bead mass in half, examine the cut surface, and record any observations on the internal microstructure.
12. Complete the questions on the Student Question Handout.

EXTRA CREDIT

Can you think of another, quicker way that could be used to find the % porosity of the sintered mass? If so, find it. Hint: you'll need the density of a single instamorph polyester bead (1.37 g/cm³).

Clean Up: Wipe up any excess or spilled water with paper towels. Dry the utensils and beakers. The sintered beads can be reheated and molded so you can take pieces of the material home!

Data Sheet for a Beads and Porosity

Weight of beads in grams		Time for beads to turn clear	
Estimated % of Pores		Estimated % of Dense Material	
Observations during sintering while beads were submerged in water:			
What are the properties of the cooled, sintered mass:			
What observations of the microstructure of the sintered body and porous surface, after cutting, can be made?			
Diameter of the surface		Area of the surface of the bulk material, A_{bulk}	Height of sintered mass
Measured pore diameters		Calculated pore areas	
1)	6)	1)	6)
2)	7)	2)	7)
3)	8)	3)	8)
4)	9)	4)	9)
5)	10)	5)	10)
Number of pores counted on surface (n)	Calculation of average pore area, A_{avg_pore}	Calculation of total pore area, A_{total_pores}	
Calculations for % Porosity and % Density:			

STUDENT QUESTION HANDOUT

Sintering: Grain Boundaries, Interfaces, and Porosity

1. What percent of the material did you estimate to be pores (open space) vs. material? How different were the estimated values compared with the calculated % of pores and % of dense material? When you cut the material to look at the inside surface, would your estimates of porosity have been more accurate?
2. Why did the porosity decrease when exposed to hot water? If the beads were left in hot water over night, would you expect higher porosity % or lower?
3. What relationship exists between the porosity and density? If porosity is increased, do you think strength will increase or decrease?
4. If the mass was put back into the hot water and you added pressure by pressing the beads together using a fork, what would happen to the remaining porosity?

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