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**TEMPERED GLASS
AND
THERMAL SHOCK OF CERAMIC MATERIALS**

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TEMPERED GLASS

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KEY WORDS: glass, tempering, annealing

PREREQUISITE KNOWLEDGE: This material could be taught as a demonstration to a typical student of materials science, at the high school level or above. More depth can be provided by having students actually temper and anneal the glass, but this requires more equipment and time. This writeup contains a simplified method for making tempered glass which uses minimal equipment.

EQUIPMENT AND SUPPLIES: (Demonstration version) Automobile side windows, obtained from an auto wrecking yard; spring-loaded center punch; polyethylene bag large enough to hold a window; two polarizing filters; overhead projector. For the laboratory version, use all of the above plus the following: Pyrex** brand borosilicate glass rod, about 6 mm diameter; torch burning oxygen-acetylene or oxygen-natural gas or propane; water-filled bucket of 20-l capacity; side-cutter pliers.

PROCEDURE: First, introduce the concept that glass is a very strong material, state that tensile strengths greater than 6.9 GPa have been measured on fresh surfaces free from all damage. Surface damage, including that resulting from the corrosive action of water, creates tiny defects that reduce the useful strength to a tiny fraction (0.2%) of this value. By using a special property of glass--the dependence of its density on its cooling rate--glass can be toughened to make it more durable and its fracture may be controlled so that dangerous shards are not produced. This process is called tempering.

Glass, like all other ceramic materials, is brittle, meaning that it will fracture at a very low strain, usually 0.01 to 0.1 percent. Like all other ceramics, failure usually starts at the surface, and this surface is very easy to damage by contact with other hard materials or even by atmospheric attack. Since the surface is easy to damage and failure begins at the surface, a process is required which makes the surface less sensitive to incidental damage. This is done by placing that surface in compression. A thermal process for accomplishing this is the subject of this experiment.

As shown in Figure 1, the density of a typical glass is dependent on its cooling rate. At high cooling rates, the glass structure becomes rigid at a density corresponding to that usually seen at high temperature. At lower cooling rates, there is more time for the glass structure to adjust so that the final density is higher. If a piece of glass is heated to a temperature above

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** Made by Corning Glass Works.

point A on the curve shown and suddenly quenched, the surfaces will cool first and become rigid at the low density corresponding to that temperature. The inner glass will cool more slowly and will reach a greater density, which means that it will pull inward on the outer glass, placing it in compression. The inner glass is placed in tension to balance the compression, but it is shielded from incidental damage by the outer glass. Since the surface glass is in compression, it will withstand quite a lot of bending strain before it is placed in tension, as is required to break it. The incidental impacts in everyday use are resisted far better by such glass, which is called tempered glass. The mechanism for tempering glass is much different than that used for tempering steel, and it may be productive to discuss the difference to avoid confusion.

In the manufacture of tempered glass for the side and rear windows of automobiles and for sliding glass doors, the glass is heated above point A, then cooled rapidly by an array of air jets blowing on both of its faces. Demonstrate this using a piece of car side window glass obtained from a wrecking yard for about \$10. With a strong light behind the piece of tempered glass, place a polarizing filter on both sides of the glass. By rotating one of the filters, the pattern left by the air jets can be seen. Some students may recall seeing similar patterns through polarizing sun glasses in light reflected from back windows of cars. Polarized light is rotated differently passing through stressed glass than it is through unstressed glass. Demonstrate the toughness of the glass by supporting it between two short pieces of wood, then standing on it. It will support your weight easily, though you may want to resist the temptation to jump on it to make a point. **Note: Just in case the glass should break, place it inside the plastic bag so that the many fragments will be captured. This is important both to promote safety and to avoid cleanup problems.**

Ask the students why, if tempered glass is so strong, it is not used in windshields. The answer is that when it breaks, it fractures into very small, roughly equiaxed pieces. A rock flipped up by the car ahead could leave you trying to drive using an opaque windshield! This fracture characteristic is an important safety feature, in that tempered glass is relatively safe when broken. It does not fracture into long, knifelike shards such as seen in ordinary glass.

Someone may ask how tempered glass may be broken, say in an escape situation if needed. Emergency personnel--policemen, ambulance drivers, etc.--routinely carry spring-loaded center punches for just such emergencies. This kind of punch generates large shock forces, enough to make indentations in steel, with enough force to overcome the compressive stress in our tempered glass window. Once the outer glass is damaged by a large enough force, the force equilibrium is disrupted and the entire piece of glass destroys itself. Demonstrate this by first placing the car window in a plastic bag, then using the center punch to damage the glass through a small hole in the bag. The window will fail with a loud noise, and the pieces can be seen to be small and not very dangerous.

In a class permitting more depth in this subject, students may temper glass for themselves. By heating a Pyrex rod in a suitably hot gas flame, droplets can be formed and allowed to fall into a bucket of water to quench them. Such a quenching method is poorly controlled, so the size of the droplets is critical; small droplets will experience less tempering than desired and large droplets may fracture when quenched. **Use caution during this operation, both with molten glass and with less obviously hot glass that has been set aside.** Tempering can be demonstrated by first grasping the droplet in the palm of the hand, then cutting the protruding "tail" off, using a pair of

side-cutting pliers. Use safety glasses or close eyes during this operation. The droplet will detonate, and will be easily felt by the student. Upon opening the hand, the glass will be found as tiny grains.

These droplets may be annealed by heating them in a furnace to 500 C, then letting them furnace cool. If the tail is now cut off the droplet, there will be no dramatic fracture as before; the stresses have been relieved by the annealing process.

SAMPLE DATA SHEETS: Not Applicable.

INSTRUCTOR NOTES: Students should be closely supervised during the tempering and testing of the glass droplets.

REFERENCE: Kingery, W. D., H. K. Bowen, D. R. Uhlmann, Introduction to Ceramics, 2nd Edition, John Wiley & Sons, 1976, Ch. 16.

SOURCES OF SUPPLIES: Pyrex rod and polarized plastic sheets can be obtained from any scientific supply house, and a suitable torch from any supplier of welding supplies. Alternately, school shop facilities could be borrowed.

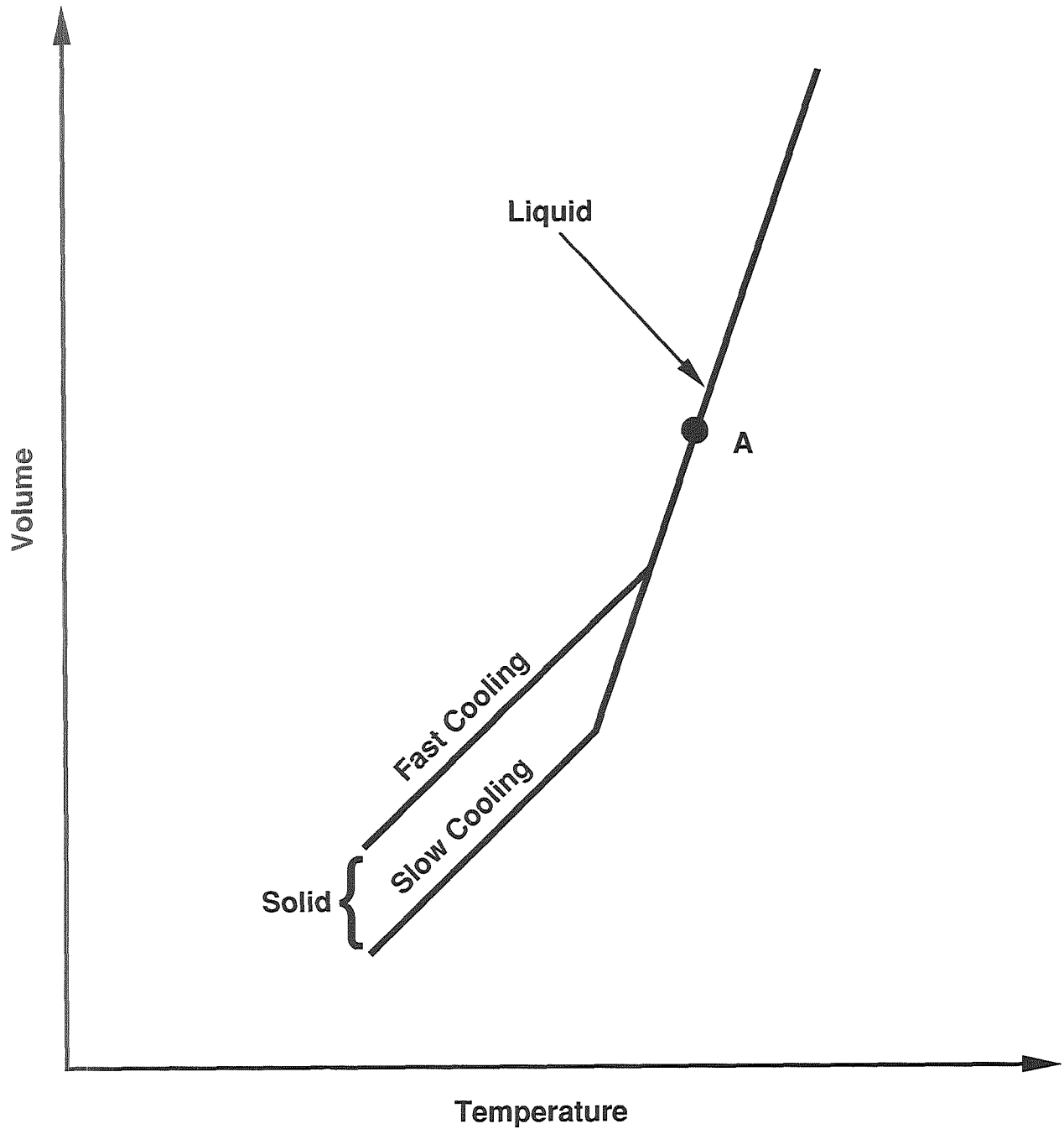


Figure 1. Relationship between temperature and volume for a typical glass. Rapid cooling results in a higher volume (lower density) than obtained with slower cooling, because more time is available for structural rearrangement in the glass.

THERMAL SHOCK OF CERAMIC MATERIALS

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KEY WORDS: thermal shock, Young's modulus, thermal expansion coefficient

PREREQUISITE KNOWLEDGE: This demonstration is suitable for students in materials science at the high school or college levels. The students should understand elementary mechanics concepts, such as Young's Modulus of Elasticity.

OBJECTIVES: To demonstrate the concept of thermal shock in ceramic materials, and to illustrate the pertinent factors of materials selection.

EQUIPMENT AND SUPPLIES: Furnace capable of controlled temperatures up to 500°C; aluminum oxide rod or 2-hole thermocouple insulator, approx. 3 mm diameter x 10 cm long; water; tongs; black or blue ink.

PROCEDURE: Thermal shock is a mechanism often leading to the failure of ceramic materials. Many uses for ceramics involve high temperatures, and if the temperature of a ceramic is rapidly changed failure may occur. Thermal shock failures may occur during rapid cooling or during rapid heating. As an example, consider rapid cooling, which is easier to visualize. If a ceramic material is cooled suddenly, the surface material will approach the temperature of the cooler environment. In doing so, it will experience thermal contraction. Since the underlying material is still hot, the skin material is stretched and so experiences tensile stress. If the resulting strain is high enough (0.01% to 0.1% for most ceramics), the ceramic will fail from the surface and cracks will propagate inward. Even if these cracks do not cause immediate failure, the ceramic will be severely weakened and may fail from mechanical overload of forces it would normally withstand.

After distributing aluminum oxide rods to the students urge them to manipulate and bend the rods with their hands. Soon, popping sounds will be heard in the classroom. These sounds demonstrate how a brittle material fails without warning, with no permanent strain, and at a low-strain value. Next, place about ten of the rods into a stainless steel beaker or a small metal pan and heat to 500°C in a suitable furnace. Remove the container from the furnace and quickly quench the rods in a bucket of water, then dry them overnight at about 100°C. The following day, dip the rods in ink, which acts as a crude dye penetrant to make any cracking visible. Wipe excess ink from the rods, handling carefully to avoid breaking. Again, urge the students to bend the rods and note that the rods are much weaker than before. Also, note from the partial penetration of the ink that the cracks do not extend into the centers of the rods. This is because the cracks start at the surface in a tensile stress area, but propagate into regions of lower stress until they stop. When a quench is performed at lower temperatures (down to about 300°C) crack density is lower and crack length is shorter. A quench temperature that is lower still will not

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result in any detectable damage. This temperature is not a constant, but is a function of both configuration and material.

When comparing different ceramics for thermal shock applications, it is common to use a figure of merit or index of thermal shock performance. This is a number (ratio) that is useful for both choosing materials and for visualizing the thermal shock process. Since the index should be high for a thermal shock resistant ceramic, its numerator should contain properties that are numerically large when good thermal shock performance is exhibited by a material. Tensile strength (S) and thermal conductivity (K) are therefore placed in the numerator, the former for obvious reasons and the latter because a high value of thermal conductivity tends to decrease thermal gradients, other factors being equal. The denominator of the thermal shock index is composed of the thermal expansion coefficient (A) and the Young's modulus of elasticity (E). The thermal expansion is responsible for the failure-causing strain, so it should be as small as possible. The elastic modulus is a measure of the stress resulting from a given strain, so it should be a low value for good thermal shock performance. Combining these factors,

$$\text{Thermal Shock Index (TSI)} = \frac{SK}{AE}$$

where the units of measurement should be consistent within a given comparison. In the case of common glasses, all of the properties except thermal expansion fall into a relatively narrow range. By choosing a glass with low thermal expansion, thermal shock failure can be avoided in most cases. See, for example, the index values for soda-lime glass, borosilicate glass, and fused silica in Table I. Note the large difference between the thermal shock indices of aluminum oxide and graphite. This difference is backed by experience; it is extremely difficult to cause graphite to fail by thermal shock, principally because its Young's modulus is so low and its thermal conductivity is high.

SAMPLE DATA SHEETS: If students quench specimens from a series of temperatures, data sheets might be constructed to record crack frequency as a function of quench temperature. Alternately, ceramic rods might be broken in 3-point bending on a testing machine, and the bending strength plotted as a function of quench temperature.

INSTRUCTOR NOTES: Provided above in Procedure. Follow usual safety precautions to avoid burns.

REFERENCES: Kingery, W. D., H. K. Bowen, D. R. Uhlmann, Introduction to Ceramics, Second Edition, John Wiley & Sons, New York, 1979, Ch. 16.

SOURCES OF SUPPLIES: The alumina should be >95% dense, but can be of any purity greater than 95%. One supplier is Coors Ceramics, Golden, CO.

Table I. Thermal Shock Index for Some Common Ceramic Materials

<u>Material</u>	$K,^{(1)}$ <u>W/cm-°C</u>	<u>S, MPa</u>	$A, ^\circ C^{-1},$ <u>$\times 10^{-6}$</u>	<u>E, GPa</u>	<u>TSI</u>
Soda-lime-silica glass	2E-2	68 ⁽²⁾	9.2	69	2.1
Borosilicate glass	2E-2	68	3.3	63	6.5
Fused SiO ₂	6E-2	68	0.6	72	94
Aluminum Oxide	3E-1	204	5.4	344	33
Graphite ⁽³⁾	1.4	8.7	3.8	7.7	416

(1) Thermal conductivity and expansion coefficients from Thermophysical Properties of Matter, Y. S. Touloukian, ed., Plenum Press, New York, 1970.

(2) Because glass tensile strength is so dependent on surface condition, a single "reasonable" value was chosen for all glass strengths.

(3) Values are typical of nuclear-grade graphite, from Industrial Graphite Engineering, Union Carbide Corp., 1959.