**ASE 324L: Aerospace Materials Lab**

# Lab #8: Ceramics

**Purpose:** This lab will provide an introduction to ceramics. The material structures, fabrication processes, and engineering applications will be discussed in lectures. The mechanical properties of alumina sheets will be measured in the lab by a three-point flexure test.

## Background

Ceramics are compounds between metallic and nonmetallic elements, including oxides ($Al_2O_3$), nitrides ($Si_3N_4$), and carbides (SiC). They can be crystalline, partly crystalline, and noncrystalline (amorphous, such as glass). The atomic bonding in ceramics ranges from completely ionic to covalent, with many ceramics exhibiting a combination of both.

## Crystal structures of ceramics

For those ceramic materials for which the atomic bonding is predominantly ionic (e.g., 63% for $Al_2O_3$), the crystal structures may be thought of as being composed of electrically charged ions instead of atoms. The positively charged metallic ions are called ***cations***, and the negatively charged ions are ***anions***. The crystal structures of ceramics depend on both the electrical charge and the relative sizes of the cations and anions. First, the crystal must be electrically neutral. Thus, the chemical formula of a compound indicates the ratio of cations to anions, or the composition that achieves the charge balance. For example, in alumina ($Al_2O_3$), the ratio between aluminum cations ($Al^{3+}$) and oxygen anions ($O^{2-}$) is 2:3. Second, the coordinate numbers (i.e., number of anions in contact with each cation) in the crystal structures depend on the radius ratio between cations and anions. Table 8.1 lists the coordination number and crystal structure for various radius ratios. The most common coordination numbers for ceramics are 4, 6, and 8. For example, the radius for $Na^+$ is 0.102 nm and for $Cl^-$ is 0.181 nm, for which the ratio is 0.56. Thus the coordination number of the sodium chloride (NaCl, also called *rock salt*) crystal is 6 (see Table 8.2 for a unit cell).

Similar to crystalline structures for metals, a number of ceramic crystal structures may be considered in terms of close-packed planes of ions (as opposed to atoms for metals). Table 8.2 lists some common unit cells of ceramic crystals. Typically, the close-packed planes are composed of the large anions, with small interstitial sites in between for the cations.

## Silicate ceramics and glass

Silicates are materials composed primarily of silicon (Si) and oxygen (O), the two most abundant materials in the earth's crust. Consequently, the bulk of soils, rocks, clays, and sand come under the silicate classification. The interatomic Si-O bond has a significant covalent character, which is directional and relatively strong. There exist a variety of silicate structures. The simplest silicate material is silicon dioxide, or ***silica*** ($SiO_2$). Structurally, each atom of silicon is bonded to four oxygen atoms, forming a tetrahedron, as shown in Fig. 8.1; each oxygen atom is shared by two adjacent tetrahedra, forming a three-dimensional network. If these tetrahedra are arranged in a regular and ordered manner, a crystalline structure is formed. There are three primary crystalline forms of silica: *quartz*, *cristobalite*, and *tridymite*. Silica can also be

made to exist as a noncrystalline solid or glass (so-called *fused silica* or *vitreous silica*), having a high degree of randomness in atomic arrangement, similar to liquid. Fig. 8.2 compares the two-dimensional structures for crystalline and noncrystalline silica. The atoms are not closely packed in the silica crystals, resulting in relatively low densities. For example, at room temperature quartz has a density of 2.65 g/cm$^3$, while the silica glass has a density of 2.2 g/cm$^3$.

The common inorganic glasses that are used for containers and windows are silica glasses to which have been added other oxides such as CaO and $Na_2O$. A typical soda-lime glass consists of approximately 70 wt% $SiO_2$, the balance being mainly $Na_2O$ (soda) and CaO (lime).

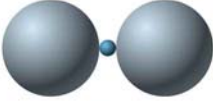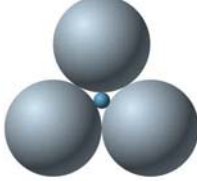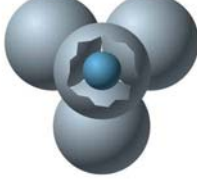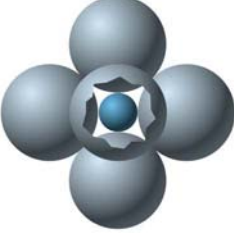Table 8.1: Coordination numbers and crystal structures in ionic ceramic crystals

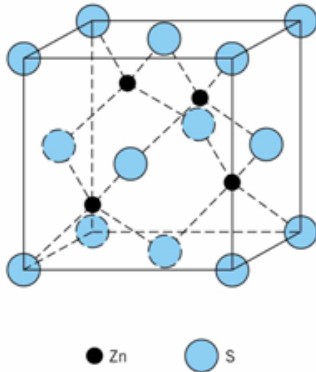| Coordination Number | Cation-Anion Radius Ratio | Coordination Geometry |
|---|---|---|
| 2 | < 0.155 | |
| 3 | 0.155–0.225 | |
| 4 | 0.225–0.414 | |
| 6 | 0.414–0.732 | |
| 8 | 0.732–1.0 | |

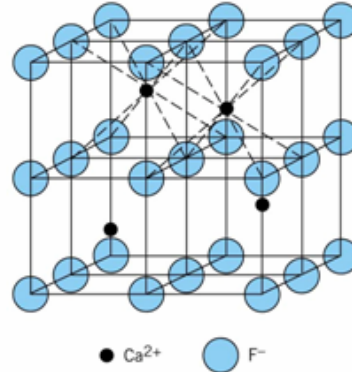Table 8.2: Common unit cells for ceramic crystals
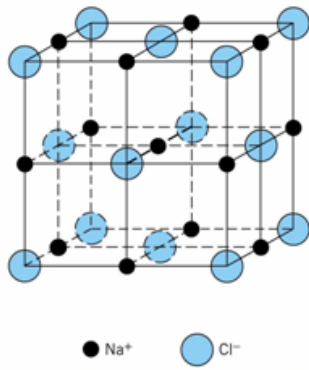


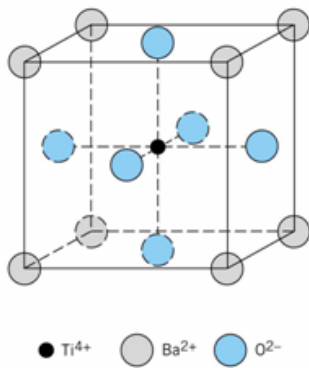(a) Rock salt, or sodium chloride (NaCl)
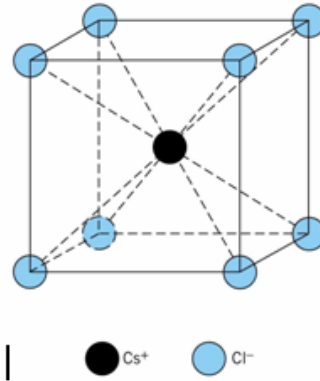


(b) Cesium chloride (CsCl)



(c) Zinc blende (ZnS)



(d) Fluorite (CaF$_2$)



(e) Perovskite crystal structure of barium titanate (BaTiO$_3$)
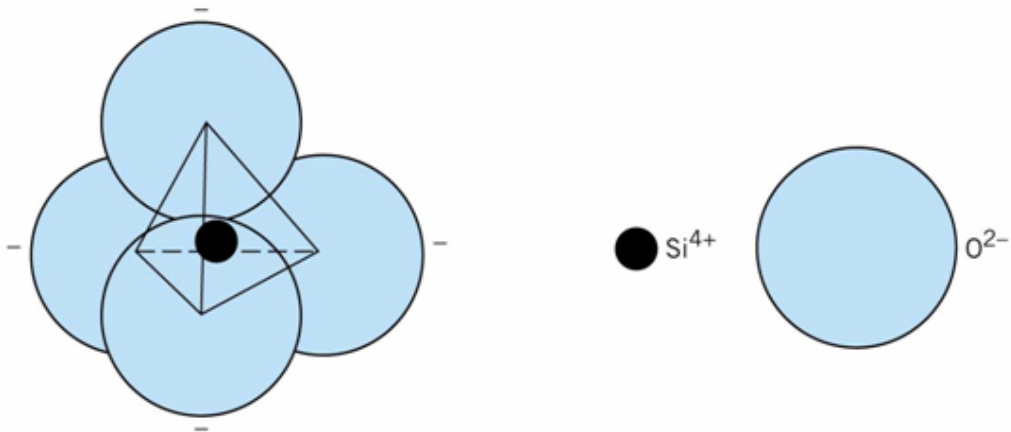
Fig. 8.1: A silicon-oxygen ($SiO_4^{4-}$) tetrahedron
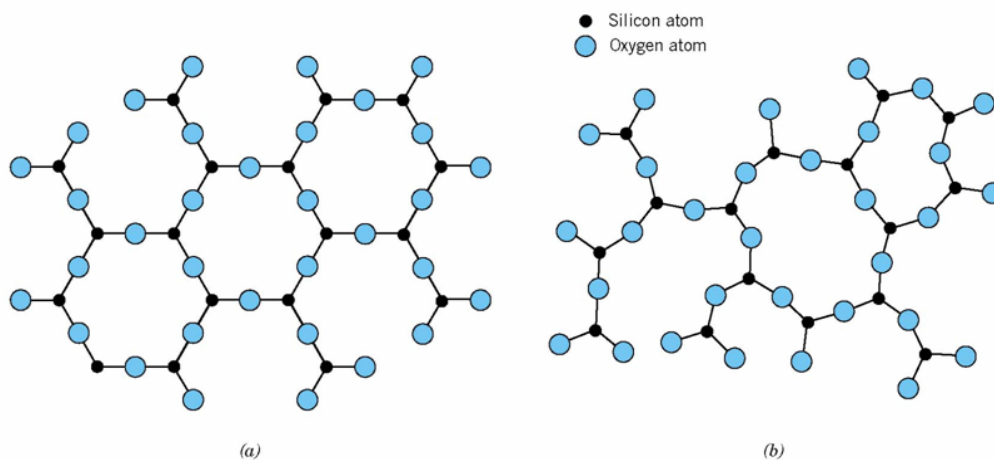


Fig. 8.2: Two-dimensional illustrations of the structure of (a) crystalline silicon dioxide and (b) noncrystalline silicon dioxide.

## Processing of ceramics

Unlike metals, casting and forming by plastic deformation (such as hot rolling or cold working) are normally impractical for ceramics due to their high melting temperatures and brittleness. Some ceramics are formed from powders that are pressed, dried, and fired. Glass shapes are formed at elevated temperatures from a fluid mass that becomes very viscous upon cooling. Cements are shaped by placing into forms as a fluid paste that hardens and assumes a permanent set by virtue of chemical reactions (*cementation*). A taxonomical scheme for several types of ceramic forming techniques is presented in Fig. 8.3.

Fig. 8.3: A classification scheme for ceramics fabrication techniques.

*Power pressing* is a common technique to fabricate ceramics, including electronic and magnetic ceramics. In essence, a powdered mass, usually containing a small amount of water or other binder, is com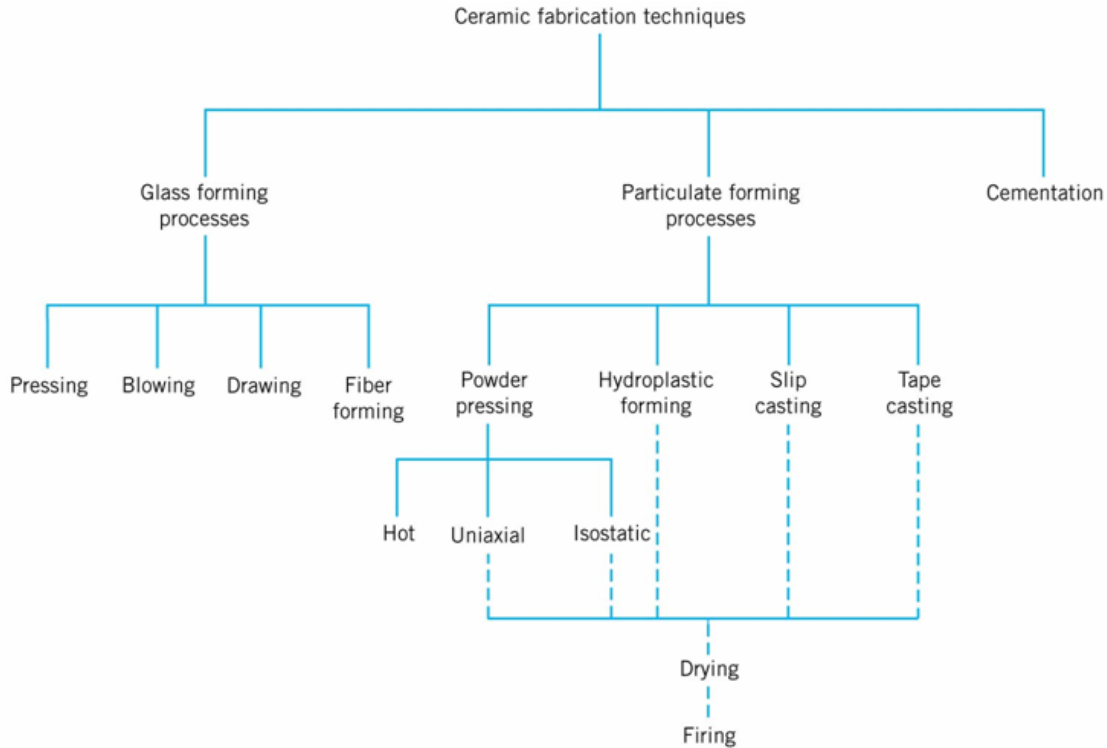pacted into the desired shape by pressure. The degree of compaction is maximized (thus, the fraction of void space between the powder particles is minimized) by using coarse and fine particles mixed in an appropriate proportion. The binder serves to lubricate the powder particles as they move past one another in the compaction process.

There are three basic powder-pressing procedures: uniaxial, isostatic (or hydrostatic), and hot pressing.

- For uniaxial pressing, the powder is compacted in a metal die by uniaxial pressure. The formed piece takes in the shape of the die and platens under pressure. This method is relatively simple, the process is inexpensive, and production rates are high.
- For isostatic pressing, the powdered material is contained in a rubber envelope and the pressure is applied by a fluid isostatically (hydrostatic pressure). More complicated shapes are possible; however, the process is more time consuming and expensive.

For both uniaxial and isostatic procedures, a firing step is required after the pressing. During firing, the formed piece shrinks, leading to reduction of porosity and improvement in mechanical integrity. This occurs by the coalescence of the powder particles into a more dense mass in a process termed *sintering*. The mechanism of sintering is schematically illustrated in Fig. 8.4. A scanning electron micrograph of a sintered alumina is shown in Fig. 8.5.

- With hot pressing, the powder pressing and heat treatment are preformed simultaneously. This method is expensive in terms of time and cost for the mold and dies.
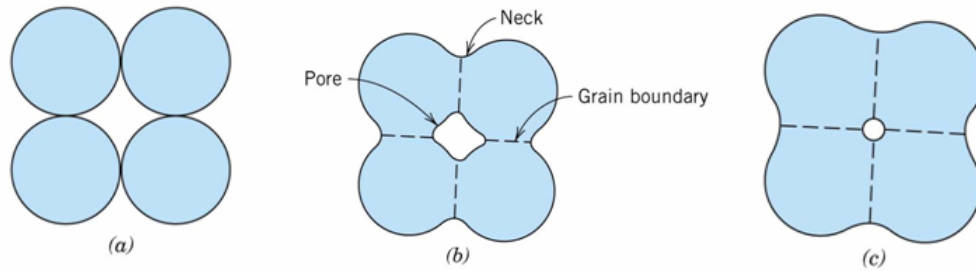
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Fig 8.4: Illustrations of microstructural changes during firing of a powder compact: (a) Powder particles after pressing; (b) Particle coalescence and pore formation as sintering begins; (c) Pores change size and shape as sintering continues.
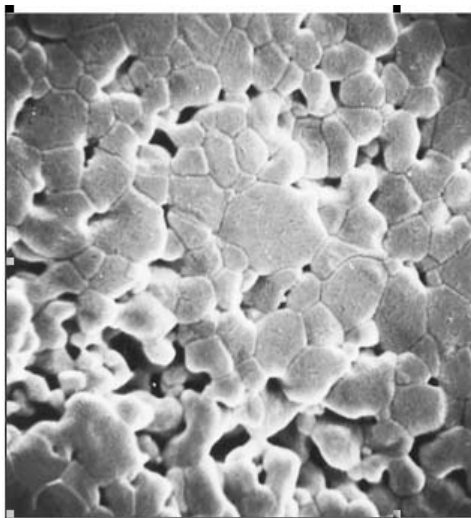


Fig. 8.5: Scanning electron micrograph of an aluminum oxide powder compact that was sintered at 1700°C for 6 min. (5000x)

## Applications of ceramics

Traditional ceramics, for which the primary raw material is clay, have been used in many products such as china, porcelain, bricks, tiles, and glasses. Over the last 50 years or so, significant progresses have been made in understanding the process-structure-property relationship of ceramics. As a result, a new generation of ceramics (*engineering ceramics* or *advanced ceramics*) has emerged and applications of ceramics have expanded into electronics, telecommunication (e.g., optical fibers made of extremely high purity silica), and aerospace industries. For example, piezoelectric ceramics (e.g., quartz, barium titanate or $BaTiO_3$, lead zirconate titanate or PZT) can be used as transducers converting mechanical energy to electrical energy and vice versa, with applications in sonar systems for submarines, automotive airbag sensors, smoke detectors, ultrasonic medical devices, and microelectromechanical systems (MEMS). Applications of advanced ceramics in the aerospace industry include thermal protection systems in rocket exhaust cones, insulating tiles for space shuttles, engine components and ceramic coatings that are embedded into the windshield glass of many airplanes.
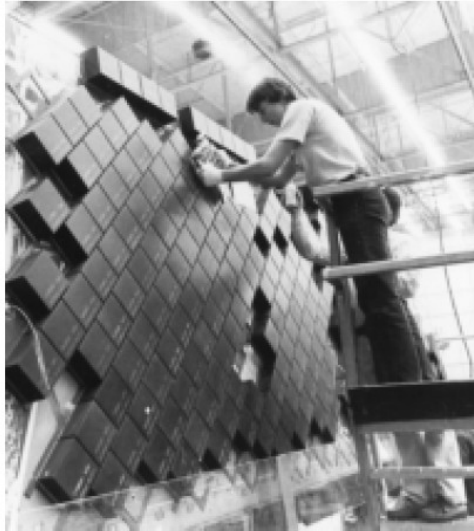
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**Figure 20.17** Photograph showing the installation of thermal protection ceramic tiles on the Space Shuttle Orbiter. [Photograph courtesy the National Aeronautics and Space Administration (NASA).]

**Mechanical properties of ceramics**

While ceramic materials have many advantageous properties (e.g., high melting temperatures and excellent resistance to thermal shock and corrosion), their applications are often limited by mechanical properties. The principal drawback is brittleness, meaning catastrophic fracture with very little energy absorption. The brittle fracture process consists of formation and propagation of cracks, which will be discussed in more details in the next Lab. In this week's lab, we will carry out a **three-point flexure** test to measure the flexural strength and elastic modulus of ceramic sheets.

**Material and specimen specifications:** Ceramic sheets (Goodfellow AL603051 alumina, 50x50 mm, and 0.50 mm thick) were cut into 10x50 mm beams for the flexure tests. Some material specifications provided by the manufacturer are listed below:

| | |
|---|---|
| Purity | 96% |
| Density | 3.9 g/cm$^3$ |
| Young's modulus | 390 GPa |
| Tensile strength | 260-300 MPa |
| Compressive strength | 2200-2600 MPa |
| Shear strength | 330 MPa |
| Upper continuous use temperature | 1700°C |
| Melting point | 2100°C |

**Test procedures:** The three-point flexure experiment is conducted in displacement control at 1.0 in/min. The applied load is measured with a 10 lb load cell with a calibration factor of 1 lb/volt. The actuator displacement is measured by a linearly varying differential transformer (LVDT) with a calibration of 0.33 in/volt. The load-displacement history is recorded automatically in a PC with a data acquisition board and LabView software.

Since no plastic deformation is expected, the specimen size (length, width, and thickness) can be measured either before or after the test.

The stress-strain behavior of brittle ceramics is not usually measured by a tensile test, for three reasons. First, it is difficult to prepare test specimens having the required geometry. Second, it is difficult to grip brittle materials to apply tension without fracturing them. Third, the very small failure strain (about 0.1%) of ceramics necessitates that tensile specimens be perfectly aligned to avoid bending, which is not practically feasible. Therefore, transverse bending tests are more frequently used for ceramics, such as three-point and four-point flexure tests (Ref: ASTM Standard C1161, "Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature.")



$$\sigma = \text{stress} = \frac{Mc}{I}$$

where $M$ = maximum bending moment
$c$ = distance from center of specimen to outer fibers
$I$ = moment of inertia of cross section
$F$ = applied load

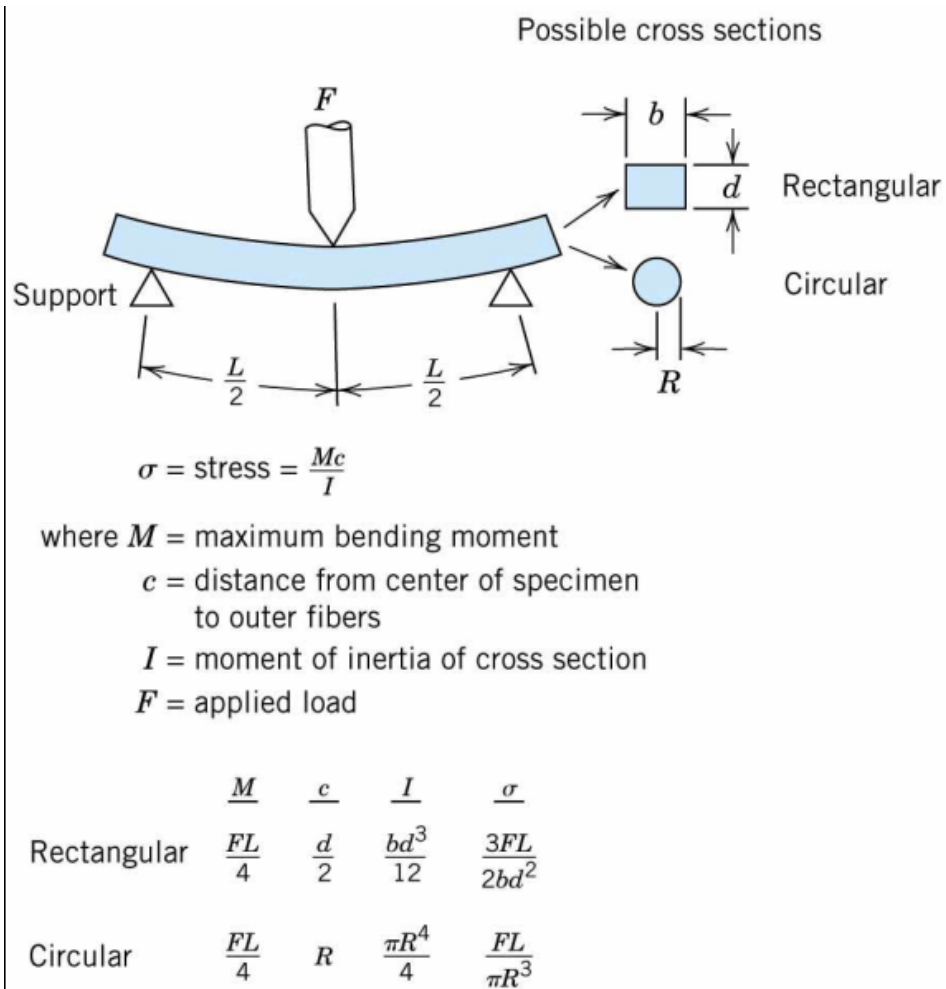| | $M$ | $c$ | $I$ | $\sigma$ |
|---|---|---|---|---|
| Rectangular | $\frac{FL}{4}$ | $\frac{d}{2}$ | $\frac{bd^3}{12}$ | $\frac{3FL}{2bd^2}$ |
| Circular | $\frac{FL}{4}$ | $R$ | $\frac{\pi R^4}{4}$ | $\frac{FL}{\pi R^3}$ |

Fig. 8.6: Scheme of the three-point flexure test for measuring elastic modulus and flexural strength of brittle ceramics.

Figure 8.6 shows the scheme of the three-point flexure test. The specimen has either a circular or rectangular cross section. At the point of loading, the top surface of the specimen is under compression, while the bottom surface is in tension. The stress is computed from the flexural formula for bending beams, as noted in Fig. 8.6. The maximum tensile stress exists at

the bottom surface of the specimen directly below the point of loading. The strain at the same point can be calculated from the measured displacement, $\delta$, at the loading point:

$$\varepsilon = \frac{12c\delta}{L^2}.$$
(8.1)

Therefore, the measured load-displacement curve can be converted to a strain-strain diagram, from which elastic modulus can be determined. Figure 8.7 compares typical stress-strain behavior for alumina and glass. For both materials, the stress is linearly proportional to the strain, while the alumina has a higher elastic modulus than glass.

The elastic moduli of ceramics generally are higher than those of metals, reflecting stronger ionic or covalent bonds in ceramics than the metallic bond. On the other hand, the densities of ceramics are lower, because their atomic structures are not close-packed. As a result, the **specific moduli** (= $E/\rho$) of ceramics are attractively high, especially for aerospace applications. This is one reason ceramic or glass fibers are used in composites.

In Figure 8.7, neither material experiences much plastic deformation prior to fracture. The maximum tensile stress at fracture measured by the flexure test is called **flexural strength**, an important mechanical parameter for brittle materials. From Fig. 8.7, the alumina has a higher flexural strength than glass.
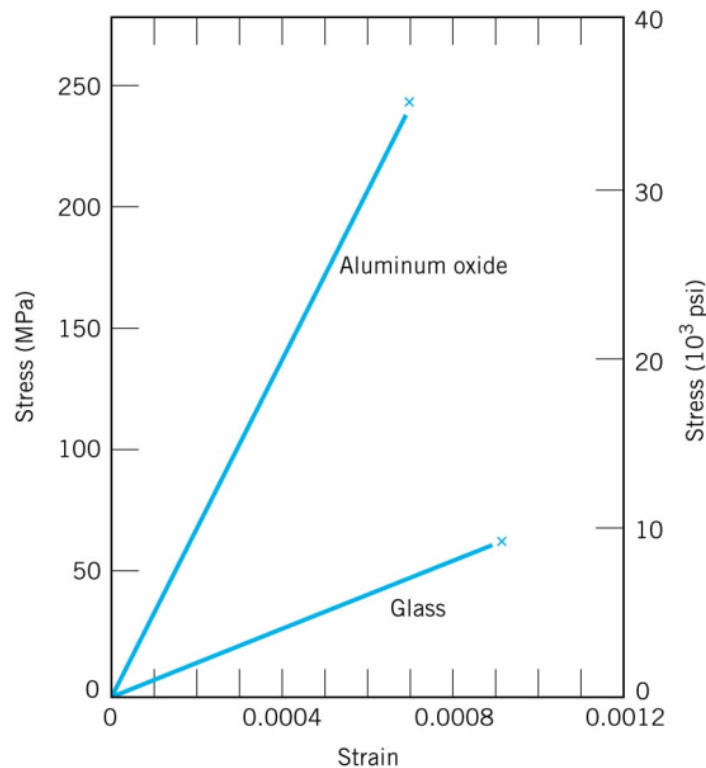


Figure 8.7: Typical stress-strain behavior for aluminum oxide and glass.

**Influence of Porosity**

For some ceramic fabrication techniques, the precursor material is in the form of a powder. Subsequent to compaction of these powder particles into the desired shape, pores or void spaces will exist between the particles. During ensuing heat treatment, much of this porosity will be eliminated; however, it is often the case that some residual porosity remains (as shown in Fig. 8.5). The residual porosity has a deleterious influence on both the elastic properties and the strength.

It has been observed for some ceramics that the elastic modulus $E$ decreases with volume fraction porosity $P$ according to

$$E = E_0\left(1 - 1.9P + 0.9P^2\right), \tag{8.2}$$

where $E_0$ is the modulus of the corresponding nonporous material. Figure 8.8 shows the curve and data for alumina at room temperature.

The flexural strength is influenced by porosity for two reasons: (1) porosity reduces the cross-sectional area of the specimen, and (2) pores act as stress concentrators: for a spherical pore, an applied tensile stress is amplified by a factor of 2. Consequently, the influence of porosity on ceramic strength is rather dramatic: a 10 vol% porosity could decrease the flexural strength by 50%. Experimentally it has been shown that the flexural strength of ceramics decreases exponentially with porosity:

$$\sigma_{fs} = \sigma_0 \exp\left(-nP\right), \tag{8.3}$$

where $\sigma_0$ and $n$ are constants determined from experiments. The experimental data of the flexural strength of alumina as a function of porosity is shown in Fig. 8.9.

The presence of pores and distributed micro-cracks in ceramics leads to a statistical variation in strength of ceramic materials. The probability that a given sample will have a given strength can be described by the Weibull distribution function

$$P_s(\sigma, V) = \exp\left[-\frac{V}{V_0}\left(\frac{\sigma}{\sigma_0}\right)^m\right], \tag{8.4}$$

which gives the *survival probability* of a sample of volume $V$ under a stress $\sigma$, and $V_0$, $\sigma_0$, and $m$ are constants to be determined from experiments. In particular, $m$ is called **Weibull modulus** that tells how rapidly the probability falls as the stress approaches $\sigma_0$. The average strength can then be calculated by

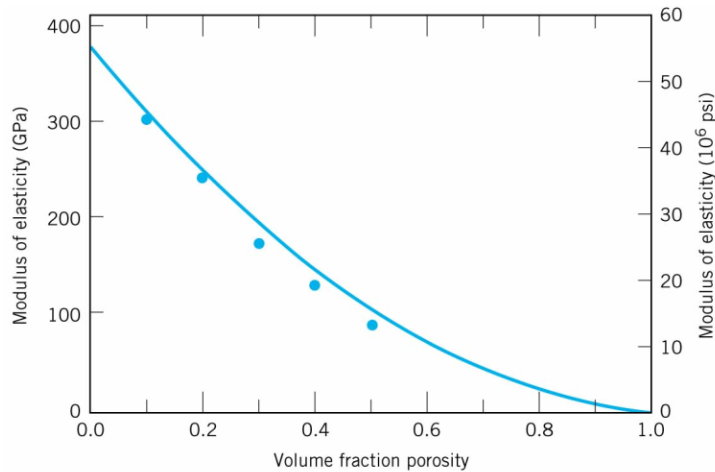$$\sigma_{ave}(V) = \int_0^\infty P_s(\sigma, V)\,d\sigma. \tag{8.5}$$

Fig. 8.8: Influence of porosity on the elastic modulus of aluminum oxide at room temperature.
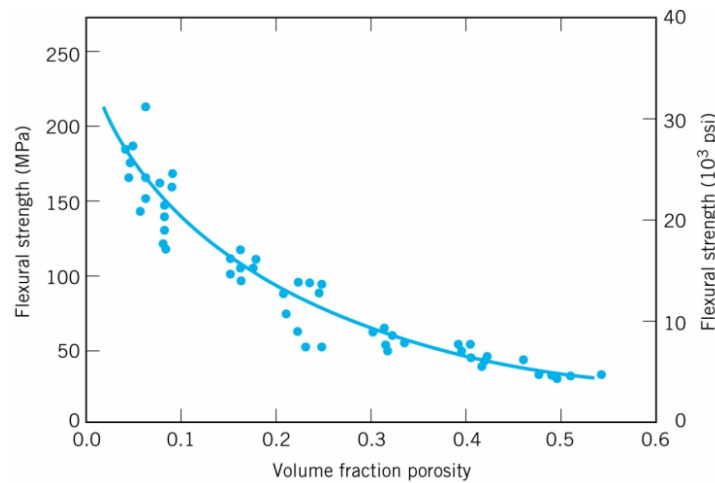


Fig. 8.9: The influence of porosity on the flexural strength of aluminum oxide at room temperature.

**Hardness**

One beneficial mechanical property of ceramics is their hardness, which is often utilized for abrasive or grinding actions. In fact the hardest known materials are ceramics (e.g., diamond).

**Creep**

Like metal, ceramics experience creep deformation under stress at elevated temperatures. However, creep occurs at higher temperature in ceramics due to their higher melting temperatures (typically over 2000°C, with one important exception: ice). High-temperature compressive creep tests are usually conducted to characterize creep behavior of ceramics.

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Homework Assignments

Do not prepare a formal lab report this week, but answer the following questions:

1.  Use the data obtained from the three-point flexure tests to determine the elastic modulus and flexural strength of the alumina sheets. Compare your results to those provided by the manufacturer.

2.  To convert measured load-displacement curves of three-point flexure tests into stress-strain diagrams, the stress is calculated using the formula given in Fig. 8.6 and the strain is computed by Eq. (8.1). Derive these equations for a beam of rectangular cross section. Note that, since the elastic modulus is unknown (to be measured), the stress and strain must be expressed in terms of measurable quantities such as force, displacement, and beam dimensions.

3.  When designing the three-point flexure test for a ceramic specimen, a preliminary calculation was performed to determine the requirements for the load cell and displacement measurements. The specimen's dimensions are set to be 50mm by 5mm by 0.2 mm. The material is estimated to have an elastic modulus of about 300 GPa and a tensile strength of about 800 MPa. The upper limits of the load that can be applied by the available load cells are 1 N, 3 N, 5 N, and 10 N. Which one would you choose for this test? To measure the elastic modulus, at least five data points are needed for the stress-strain curve before fracture. What resolutions in the force and displacement measurements are required for this purpose?

4.  The *specific modulus* of a material is defined as Young's modulus divided by its mass density ($E/\rho$). Similar, the specific strength can be defined as the strength divided by mass density ($\sigma_{fs}/\rho$). For aerospace applications, high specific modulus and high specific strength are desirable. Equations (8.2) and (8.3) give the elastic modulus and flexural strength of porous ceramics. The mass density is simply, $\rho = \rho_0(1-P)$, where $\rho_0$ is the density of the corresponding nonporous material. Plot the specific modulus and the specific strength as functions of the porosity, and determine the porosities that give the maximum specific modulus and the maximum specific strength, respectively. Take $n = 5$ in Eq. (8.3).

5.  Plot the Weibull distributions of ceramic strength with different Weibull moduli: $m = 5$, 10, 20. Take $V = V_0$ and $\sigma_0 = 300$ MPa. Discuss the possible statistical scattering of the strength data for different Weibull moduli.

## Lab 8 – Learning Objectives

By the end of this week, you will be able to do the following:

Level 1 (remembering)

- Describe the types of atomic bonds and crystal structures in ceramics.
- Describe basic mechanical properties (modulus, strength, hardness, etc.) of ceramics in comparison to metals.

Level 2 (Understanding)

- Explain why the fabrication processes of ceramics are different from those for metals.
- Explain why the uniaxial tensile test is not applicable for ceramics.

Level 3 (Applying)

- List at least five ceramic materials in daily life.
- List at least three engineering applications of advanced ceramics.
- Measure elastic moduli and flexural strengths of ceramic materials by three-point flexure tests.

Level 4 (Analyzing)

- Explain why ceramics are not used for the main component in airplane engines.

Level 5 (Evaluating)

- none

Level 6 (Creating)

- Design three-point flexure tests for brittle materials.