#### MECHANICAL PROPERTIES AND TEST METHODS OF ADVANCED CERAMICS

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### CERAMICS

 $\blacktriangleright$  Always composed of more than one element (e.g., Al<sub>2</sub>O<sub>3</sub>, SiC, SiO<sub>2</sub>)

➢Bonds are partially or totally ionic, can have combination of ionic and covalent bonding

- ➤Generally hard and brittle
- Generally low density (compared to steel)
- ➤Generally electrical and thermal insulators
- > Can be optically opaque, semi-transparent, or transparent
- Traditional ceramics based on clay (china, bricks, tiles, porcelain), glasses.

Structural ceramics – "New ceramics" for electronic, computer, aerospace industries.

# WHY CERAMICS



### WHY CERAMICS



### WHY NOT CERAMICS



# SERVICE ADVERTISED ON THE WEB

Mechanical Characterization of Ceramics, Brittle Materials and Components: Materials

#### Ceramics

- monolithic
- reinforced (with particles, whiskers, fibers, nano-fibers, CNT)
- conductive, non-conductive, piezo-electric

#### Composites

- ceramic-ceramic (CMC)
- metal-ceramic (MMC)

#### Ceramic laminates

- macro (e.g. wear parts)
- micro (e.g. sensors)
- coatings

#### Joined materials (brazed, glued)

- ceramic with ceramic
- ceramic with metal

#### and many more, e.g.

- porcelain (e.g. isolator)
- glass (e.g. accessories, controls, instruments)
- long fibers
- porous bodies and foams
- green-bodies



Tensile strength test of a Metal-Ceramic joint



BSE: Si<sub>3</sub>N<sub>4</sub>-MoSi<sub>2</sub> composite



Ceramic laminate. The white outer layers have a thickness of  ${\sim}50~\mu\text{m}.$ 

# SERVICE ADVERTISED ON THE WEB

Mechanical Characterization of Ceramics, Brittle Materials and Components: Properties

#### Strength up to 1500°C

- 3-point and 4-point bending
- biaxial flexural (ring on ring)
- ball-on-three-ball (small discs)
- C-ring
- shear

#### Fracture toughness

- SEVNB: Single Edge V-Notched Beam up to 1500°C
- SCF: Surface Crack in Flexure
- SEPB: Single Edge Pre-cracked Beam
- edge chipping

#### Young's modulus, Shear modulus, Poisson's ratio

- natural frequency up to 1000°C
- bending up to 1500°C (Young's modulus)
- instrumented indentation (Young's modulus)

#### Hardness

- Vickers and Knoop
- dynamic hardness

#### and many more, e.g.

Lifetime, e.g. subcritical crack growth under

- static or cyclic load
- constant stress rate
- creep resistance up to 1600°C
- tensile load thermal shock resistance



Elastic modulus depends on the microstructure and interatomic bonding forces.



Ceramics, glasses,:	(GPa)	Melting Temp. (C)
Diamond (C)	1000	
Tungsten Carbide (WC)	450 - 650	2870
Silicon Carbide (SiC)	450	
Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> )	390	2072
Berylium Oxide (BeO)	380	
Magnesium Oxide (MgO)	250	
Zirconium Oxide (ZrO)	160 - 241	
$Mullite(Al_6Si_2O_{13})$	145	
Silicon (Si)	107	
Silica glass (SiO <sub>2</sub> )	94	
$Soda-lime\ glass\ (Na_2O-SiO_2)$	69	
Metals:		
Tungsten (W)	406	3400
Low Alloy Steels	200 - 207	
Stainless Steels	190 - 200	
Cast Irons	170 - 190	
Copper (Cu)	124	1084
Titanium (Ti)	116	
Brasses and Bronzes	103-124	
Aluminum (Al)	69	660

The values given are indicative only

For many ceramic fabrication techniques, the precursor material is in the form of a powder. During the ensuing heat treatment some residual porosity will remain.



The elastic modulus can be measured by the **impulse excitation of vibration method** (ASTM C 1259).

This test method measures the fundamental resonant frequency of test specimens of suitable geometry by exciting them mechanically by a singular elastic strike with an impulser. A transducer senses the resulting mechanical vibrations of the specimen, which provides a numerical reading that is (or is proportional to) either the frequency or the period of the specimen vibration.



ELECTRICAL SYSTEM

An impulse-excited solid might be expected to vibrate at several resonant frequencies simultaneously. However, for simple shapes, such as beams and plates, it is possible to support them and excite them in such a way that only one mode of vibration is prevalent. The sample is struck at one of the antinodes, and the frequency of vibration is then measured at another antinode using a microphone and frequency analyzer





All surfaces on the rectangular specimen shall be flat. Opposite surfaces across the length and width shall be parallel within 0.01 mm or 0.1 %, whichever is greater. Opposite surfaces across the thickness shall be parallel within 0.002 mm or 0.1 %, whichever is greater.

where

E= Young's modulus (Pa) m= mass of the bar (g)

 $E = \frac{0.9464mf_f^2 L^3}{ht^3}$ 

b=width of the bar (mm) L= length of the bar (mm)

- t= thickness of the bar (mm)
- $f_f$  = fundemental resonant frequency of bar in flexure (Hz)
- $T_1$  = correction factor for fundemental flexural mode to account for finite thickness of bar, Poisson's ratio, etc.



FLEXURE

NODE LINE



$$G = \frac{4mf_t^2 L}{bt} B$$

G = shear modulus (Pa) f<sub>t</sub>= fundemental resonant frequency of bar in torsion (Hz)

$$B = \frac{b/t + t/b}{4(t/b) - 2.52(t/b)^2 + 0.21(t/b)^6}$$

More detailed information on measuring elastic modulus of ceramics can be found in:

ASTM C 1259 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Impulse Excitation of Vibration

ASTM C 1198 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Sonic Resonance

Theoretical strength is  $\sim E/10$ .

We would expect strength of ceramic materials at the order of 10 GPa.

In practice, strength of ceramic materials are much lower in tension.



Typical stress-strain behavior for aluminum oxide and glass.

Ceramic materials usually fail by brittle fracture.

Strength is a function of the fracture toughness and the defect size

$$\sigma_f = \frac{K_{Ic}}{Y\sqrt{\pi c}}$$

For a given toughness, an increase in the defect size results in a decrease in the strength of the material.



Strength of a ceramic material depends on the size of the largest defect present in the structure.



The influence of porosity



The influence of grain size

As the volume of the ceramic material subject to stress decreases, probability of the presence of a larger defect also decreases; therefore strength increases.



Only part of the total volume, *V*, of the flexure specimen on the left is in tension. Only a smaller fraction of this region (depicted by the shaded area) is subjected to higher stresses.

	Tensile strength (MPa)	Flexural strength (MPa)	Compressive strength (MPa)
Silicon Nitride	350-415	930	2100-3500
Silicon Carbide	390-450	634	1035-1725
Aluminum Nitride	_	200	1400-2100
Tungsten Carbide	344	1930	2683
Titanium Oxide	51.6	137	688
MgO stabilized Zirconia	352	620	1750
Aluminum Oxide (98%)	150	300	2500
Aluminum Oxide (99%)	180	400	3000
Zirconia toughened alumina (ZTA)	_	912	_
Boron Carbide	_	450	470
Titanium Diboride	_	277	470
Zirconia	_	800-1200	2000

Tensile Strength Test (ASTM C 1273) requires

- a very careful alignment of the load train.
- a uniform interface between the grip components and the gripped section of the specimen
- a special geometry



3 *PL* 

 $S = \frac{1}{2bd^2}$ 

#### Flexural Strength Test (ASTM C 1161)



	TABLE 1 Fixture Spans	
Configuration	Support Span (L), mm	Loading Span, mm
A	20	10
В	40	20
С	80	40

TABLE 3 Specimen Size

Configuration	Width (b), mm	Depth (d), mm	Length ( <i>L<sub>T</sub></i> ), min, mm
A	2.0	1.5	25
B	4.0	3.0	45
С	8.0	6.0	90



Configuration	Crosshead Speeds, mm/min
A	0.2
В	0.5
С	1.0



42-

C: L = 80 mm

Specimen Preparation—Depending upon the intended purpose specimens can be tested As-Fabricated or Polished

Cross-sectional dimensional tolerances are  $\pm$  0.13 mm for B and C specimens, and  $\pm$ 0.05 mm for A. The parallelism tolerances on the four longitudinal faces are 0.015 mm for A and B and 0.03 mm for C.

The four long edges of each specimen shall be uniformly chamfered at 45°, a distance of 0.12±0.03 mm. They can alternatively be rounded with a radius of 0.15±0.05 mm. Edge finishing must be comparable to that applied to the specimen surfaces. Strength correction factors are provided in the standard.



ASTM C 1273 Test Method for Tensile Strength of Monolithic Advanced Ceramics at Ambient Temperatures

ASTM C1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature

ASTM C1323 Test Method for Ultimate Strength of Advanced Ceramics with Diametrally Compressed C-Ring Specimens at Ambient Temperature

ASTM C1499 Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature

• Also known as 'ring on ring test'

ASTM C1424 Test Method for Monotonic Compressive Strength of Advanced Ceramics at Ambient Temperature

# STATISTICAL ANALYSIS OF STRENGTH

There is usually considerable variation and scatter in the fracture strength of ceramic materials.



The frequency distribution of observed fracture strengths for a silicon nitride material.

$$\sigma \leftarrow \bigcirc \circ \sigma$$

The chain fails if the weakest link fails (Weibull, 1939)

Survival probability of one link:  $P_s = [1 - (\sigma / \sigma_0)^m]$ 

m: Weibull modulus  $\sigma_0$ : Weibull strength

Survavival probability of a specimen with volume V<sub>0</sub>:







the generalized strength distribution law can be given by:



Testing a longer Gauge Length on Strength Distribution gives the illusion of lower strength because a greater sample volume is tested, exposing more lower strength flaws



Weibull plots for strength data of sintered alumina measured with different specimen sizes

More detailed information on statistical analysis of strength can be found in:

ASTM C 1239 Standard Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics

ASTM C 1683 Standard Practice for Size Scaling of Tensile Strengths Using Weibull Statistics for Advanced Ceramics

Hardness: The measure of a material's resistance to deformation by surface indentation or by abrasion.

Ceramics hardness is much higher than metals.

Fo	For Steel	
Vickers HV	Approximate Tensile Strength (MPa)	
660	2199	
620	2061	
580	1923	
540	1792	
500	1655	
460	1517	
420	1379	
380	1241	
340	1110	
300	972	
260	834	
220	696	
200	634	
180	579	
160	517	
140	455	

For Ceramics		
	Hardnes (HV)	
Silicon Nitride	1800	
Silicon Carbide	2300	
Aluminum Nitride	1110	
Tungsten Carbide	1600	
Titanium Oxide	800	
MgO stabilized Zirconia	1200	
Aluminum Oxide (98%)	1800	
Aluminum Oxide (99%)	1800	
Zirconia toughened alumina (ZTA)	1500	
Boron Carbide	2700	
Titanium Diboride	2700	
Zirconia	1200	



10 - Diamond

The values given are indicative only

Knoop hardness (ASTM C 1326) and Vickers hardness (ASTM C 1327) are preferred hardness measurement methods.

*Vickers indenter* is a square-based pyramidal-shaped diamond indenter with face angles of 136°.



Vickers hardness is generally given as the applied force on the indenter divided by the area between the four faces of the pyramid indenter and the surface (measured after the indenter is removed)

Hence, the Vickers hardness number is computed by  $HV = 1.8544 \frac{P}{d^2}$ P= load, kgf d= average length of the two diagonals of

indentation, mm

The hardness symbol HV shall be supplemented by a number indicating the test force used (in kgf) Ex: HV1 means Vickers hardness for an applied force of 1 kg (9.81 N)

The Vickers hardness reported with units of **GPa** is determined as follows:  $HV = 0.0018544 \frac{P}{d^2}$  ASTM C 1327 P= load, N

Some in the ceramic community use the projected area in defining the Vickers hardness. In this case, hardness reported with units of GPa is determined as follows:

 $H = 0.002 \frac{P}{d^2}$ P=load, N

B. Lawn, Fracture of Brittle Solids

In reporting Vickers hardness it would be useful to state whether the projected area or the contact area is used since there is a 7.9 % difference in hardness values.

Closest permitted spacing between Vickers indentations







*Knoop indenter is* a rhombic-based pyramidalshaped diamond indenter with edge angles of 172° 30' and 130° 00'.







Knoop hardness is obtained by dividing the force applied to the Knoop indenter by the projected area of the permanent impression made by the indenter.

Hence, the Knoop hardness number is computed by  $HK = \frac{P}{d^2 C_p}$ 

P= load, kgf

d= length of the long diagonal of the indentation, mm C<sub>p</sub>=correction factor related to the shape of the indenter, ideally 0.070279

The Knoop hardness reported with units of GPa

$$HK = 0.001 \left(\frac{P}{d^2 C_n}\right)$$

P= load, N

d and  $C_p$  as defined above.

More detailed information on measurement of hardness of ceramics can be found in:

ASTM C 1326 Test Method for Knoop Indentation Hardness of Advanced Ceramics

ASTM C 1327 Test Method for Vickers Indentation Hardness of Advanced Ceramics

Linear elastic fracture mechanics



Stress near the crack tip  $\sigma_y = \frac{\sigma\sqrt{\pi c}}{\sqrt{2\pi r}}f(\theta)$  $K_I = Y\sigma\sqrt{\pi c}$ 

 $K_I > K_{IC} \implies$  crack propagation

- $K_{I}$ : stress intensity factor (MPa $\sqrt{m}$ )
- K<sub>IC</sub>: Critical stress intensity factor (toughness)
- $\sigma$  : Applied stress (MPa)
- 2c : Crack length (m)
- Y : Stress intensity factor coefficient

The fracture toughness of ceramic materials are low because they do have limited energy dissipation mechanisms.



**ASTM C 1421** covers the determination of fracture toughness of advanced ceramics at ambient temperature

The fracture toughness values are determined using beam test specimens with a sharp crack. The crack is either a semi-elliptical surface crack, a straight through crack, or it is propagated in a chevron notch.



The precrack must be on the tension (bottom) surface.

#### The Precracked Beam Method

Either the machined notch, a Vickers indent, or a series of Vickers indents act as the crack starter.

Place the test specimen in the compression fixture with the surface containing the notch or indent(s) over the groove Load the test specimen in the compression fixture until a distinct pop-in sound is heard and/or until a pop-in precrack is seen.

The fracture force of the test specimen is determined in flexure test and the fracture toughness,  $K_{lpb}$ , is calculated from the fracture force, the test specimen size, and the measured precrack size.



a) Notch detail - side view b) Multiple indents - tensile surface view Precracked Beam Precracking Arrangement



Suggestion for Bridge Compression Fixture

$$K_{Ipb} = f \left[ \frac{P_{\max}[S_o - S_1] 10^{-6}}{BW^{3/2}} \right] \left[ \frac{3[a/W]^{1/2}}{2[1 - a/W]^{3/2}} \right]$$

f=f(a/W)

Surface Crack in Flexure Method

A beam test specimen is indented with a Knoop indenter. Specimen is tilted to make crack more visible in post fracture measurement.

It is polished (or hand ground), while maintaining surface parallelism, until the indent and associated residual stress field are removed.

The fracture force of the test specimen is determined in flexure test and the fracture toughness,  $K_{Isc}$ , is calculated from the fracture force, the test specimen size, and the measured precrack size.



Measuring precrack size after the fracture:

Fractographic techniques and fractographic skills are needed for this step. The optimum procedure will vary from material to material.

Either an optical microscope or a scanning electron microscope can be used.



(a) SEM and (b) optic microscope images of Knoop Indent Precrack in SiN. (



Dye penetration procedures may be beneficial and are permitted by these test methods. Considerable caution should be exercised in the use of these test methods, since it is difficult to completely penetrate the small, tight cracks in ceramics.

More detailed information on measurement of toughness of ceramics can be found in:

ASTM C 1421 Standard Test Methods for Determination of Fracture Toughness of Advanced Ceramics at Ambient Temperature

# Thanks for listening!

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#### Mangan Katkısının 94(Na0.5Bi0.5)TiO3-6BaTiO3 Çok Katmanlı Piezoelektrik Seramiklerin Elektriksel Yorulma Davranısına Etkisi

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Anahtar Kelimeler Kurşunsuz piezoelektrik çok katmanlı elektriksel vorulmo

mangan Keywords

Lead-free piezoelectric nultilaver electrical fatigue manganese

Piezoelektrik cok katmanlı seramikler evlevici uvgulamalarında vüksek mekanik dayanım ve deplasman özellikleri nedeniyle kullanılmaktadırlar. Bu uygulamalarda <sup>2</sup>Nanotech Ileri Tek Malz. Elk-Elkt. Sis. San. çok fazla elektriksel çevrime maruz kalan malzemelerin elektriksel tepkileri, çevrim sayısına bağlı olarak düşmektedir. Buna elektriksel yorulma denmektedir. Eyleyici olarak kullanılacak olan malzemelerin yorulma davranışının belirlenmesi <sup>3</sup>Eskişehir Teknik Üniversitesi, Malzeme Bil. Ve ve mümkünse dayanımının artırılması gerekmektedir. Eyleyici uygulamalarında genellikle kursun icerikli PZT tahanlı malzemeler kullanılmaktadır. Kursunsuz piezoelektrik malzemeler ise kapasitör uygulamalarında sıklıkla kullanılmaktadır. Toksik etkileri sebebiyle RoHs standartları gereği kurşun içerikli malzemelerin tüm endüstriyel ürünlerde sınırladırılması istenmektedir. Bu sebeple tüm dünyada kurşunsuz piezoelektrik malzemeler geliştirilmektedir. Eyleyici uygulamalarında kullanılmak üzere geliştirilen kompozisvonlardan bir taneşi de 94(Nan Bin 5)TiO3-6BaTiO3kısaca NBT-6BT kompozisyonudur. Bu çalışmada kurşunsuz NBT-6BT kompozisyonuna Mangan ilavesi yapılmış, çok katmanlı seramik üretilmiş ve katkısız NBT-6BT kompozisyonu ile karşılaştırılmıştır. Bu sayede Mangan katkısının NBT-6BT çok katmanlı seramiklerin elektriksel yorulma davranışlarına etkisi araştırılmıştır. Kurşunsuz çok katmanlı seramikler su çözelti sistemiyle ve gümüş-paladyum (Ag-Pd) elektrot kullanılarak 1115-1120°C sinterleme sıcaklığı ve 2-4-6 saat olmak üzere farklı sinterleme sürelerinde sinterlenerek üretilmistir. Mangan katkısının kalıcı polarizasyon değerini artırdığı, histeris eğrisinin kareselliğindeki bozulma oranını düşürdüğü belirlenmiştir.

> Effect of Manganese Additive on Electrical Fatigue Behavior of 94(Na0.5Bi0.5)TiO3-6BaTiO3 Multilayer **Piezoelectric Ceramics**

Abstract

Piezoelectric multilayer ceramics are used in actuator applications related to high mechanical strength and actuating performances. In these applications, the electrical response of materials decreases depending on the number of applied cycles. This is called electrical fatigue. Fatigue behavior of the materials must be determined if possible fatigue strength increased to be used as actuators. PZT based materials with lead content are generally used in actuator applications. Lead-free piezoelectric materials are frequently used in capacitor applications. Due to its toxic effects, it is desirable to limit lead containing materials in all industrial products as required by RoHs standarts. Therefore, lead-free piezoelectric materials are being developed all over the world. One of the compositions developed for use in actuator applications is 94(Na0 5Bio 5)TiO3-6BaTiO3, in short NBT-6BT. In this study, manganese was added to lead free NBT-6BT composition, multilayer ceramics were produced and compared with NBT-6BT multilayer ceramics. Thus, the effect of manganese additive on the electrical fatigue behaviour of NBT-6BT multilayer ceramics was investigated. Lead-free multilayer ceramics were produced with water-based tape casting slurry system and Ag-Pd electrode by sintering at 1115C-1120°C sintering temperature and 2-4-6 hours at different sintering times. It has been observed that manganese additive increases the permanent polarization value, decreases the deformation ratio of squareness of hysterisis.

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#### **Prof. Dr. Servet TURAN**



Eskişehir Teknik Üniversitesi Malzeme Bilimi ve Mühendisliği

#### Problem Çözümünde Farklı Mikroskop Tekniklerinin Stratejik Kullanımı

Malzeme ve yaşam bilimlerinde farklı mikroskopların kullanımları yüzyıllardır devam etmekte olup en önemli buluşlarda ve gelişmelerde mikroskoplar çok önemli yer tutmuş ve bu buluşların birçoğu Nobel ödülü ile taçlandırılmıştır. Mikroskoplarla İlgili bilmek istediğiniz ama sormaya çekindiğiniz her türlü soruyu sorabileceğiniz (her sorunuza cevap veremeyebiliriz!) bir ortamda geçecek olan bu seminerde mikroskop tekniklerinin çalışma prensipleri ile ilgili kısa bir bilgi verildikten sonra farklı problemlerin çözümünde stratejik olarak hangi tekniğin ne zaman ve nasıl kullanılması gerektiği ve elde edilen sonuçların anlamlandırılmasında dikkat edilmesi gerdeken unsurlar farklı örnek problem çözümleri ile anlatılacaktır. Mikroskopta elde edilen her gördüğümüz sonuca inanacak mıyız?, Gördüğüm şey doğru mudur?, Doğruyu nasıl buluruz?, ana ekseni üzerinden hareketle doğru bilinen yanlışlar örneklerle anlatılacaktır.





