Biaxial Testing: Appropriate for Mechanical Characterisation?

Gary D. Kipling, Andrew Easton and Gareth B. Neighbour
Dept. of Engineering, University of Hull, Cottingham Road, Hull, UK. HU6 7RX
Email: g.d.kipling@2005.hull.ac.uk

Abstract
Material properties are often described as being characteristic and are quoted as such irrespective of scale. However, although the testing of materials is sometimes assumed to be a mature field, there are issues related to the appropriateness of the test, the inherent suitability of the assumptions in determining the material property value, and indeed inherent microstructure of the material concerned. Quasi-brittle materials by their very character show deviation from a truly elastic material and so challenge some of the assumptions being made; and this is true for nuclear graphites. Polygranular graphite is used in Advanced Gas-cooled Reactors (AGRs) primarily as a means of providing moderation for the nuclear reaction, but also as a major structural component in the form of the core bricks. At present, in the nuclear industry, the prediction of when cracked graphite bricks will occur in a nuclear core is largely based on the measurement of mechanical properties from small samples, even though the volume of a typical brick is a factor of 10^4 greater than that of a typical flexural test sample. For polygranular graphites, many models to predict the probability of failure have been generated and these are usually related to a uniaxial value and indeed most tests conducted determine uniaxial values. If sample size restrictions apply, particularly for engineering ceramics, a biaxial stress geometry is sometimes used. This is especially true if the material is used in applications that impose multi-axial stress fields and so to some extent better resemble the engineering duty and reflect the real performance criterion. As an illustration, this paper will also discuss the evaluation and choice of different designs of biaxial test apparatus. Further, for the preferred biaxial testing system, results will be presented that demonstrates the issues discussed above and shows the complexities involved in the small-scale dependence of geometry upon strength.

Keywords

Biaxial Stress, Strength, Mechanical Characterisation

INTRODUCTION

The testing of materials is sometimes assumed to be a mature field. There are, however, issues relating to the appropriateness of the test, the inherent suitability of the assumptions in determining the material property value, and indeed inherent microstructure of the material concerned. Quasi-brittle materials by their very character show deviation from a truly elastic material and so challenge some of the assumptions being made. At present, in the nuclear industry, the prediction of when graphite bricks will crack in a nuclear core is largely based on the measurement of mechanical properties from small samples, even though the volume of a typical brick is a factor of 10^4 greater than that of a typical flexural test sample.

For polygranular graphites, many models to predict the probability of failure have been generated and these are usually related to a uniaxial value and indeed most tests conducted determine uniaxial values. If sample size restrictions apply, particularly for engineering ceramics, a biaxial stress geometry is sometimes used. This is especially true if the material is used in applications that impose multi-axial stress fields and so to some extent better resemble the engineering duty and reflect the real performance criterion. Thus, using a biaxial stress geometry is appealing in many respects, as it represents a more severe stress state than uniaxial stress and is accordingly better suited to conservative design basis. However, whilst biaxial testing has attractions there are additional complications which are the subject of this paper.

BIAXIAL TEST METHODS

There are many test configurations available to determine the biaxial strength of a material. In this work, an extensive literature review was performed searching for all types of biaxial test. After a brief evaluation, 11 candidate methods were identified:

- Diametral Compression Test (*e.g.* see Awaji *et al.* 1987)
- Ball on three ball (*e.g.* see Godfrey, 1985)
- Ring on ring (*e.g.* see Fett *et al.* 2006)
- Ball on ring (*e.g.* see Isgrò *et al.* 2003)
- Internal Pressure Tube / Ring Test (*e.g.* see Perreux and Suri, 1997)
- Bulge Test (*e.g.* see Imaninejad *et al.* 2004)
- Cruciform Test (*e.g.* see Welsh and Adams, 2002)
- Arcan test (*e.g.* see Doyoyo and Mohr, 2003)
- Iosipescu Test (*e.g.* see Kumosa and Han, 1999)
- Cold-Spin Test (*e.g.* see Brückner-Foit *et al.* 1993)
- Scissor Arms Test (*e.g.* see Kumosa and Han, 1999)

A selection process was undertaken to determine the most appropriate test from the 11 candidate tests using a binary dominance method to weight each of the 17 identified selection criteria, including, for example, adaptability to different specimen geometries, potential for edge effects, suitability for brittle materials, *etc*. The most suitable test for the given attributes was found to be the ball-on-three-ball test. After selection, an experimental test programme was defined and the test apparatus was designed using SolidWorks and manufactured to fit existing high specification universal test machines (Lloyds EZ50).

Ball-on-Three-Ball Test Method

The ball-on-three-ball test apparatus comprises of a thin disc sample supported on three equally spaced ball bearings and held in position using alignment pins. The sample is loaded in the centre of the disc using another ball bearing, as illustrated in Figure 1. During testing, the bottom surface of the sample is subject to a biaxial tensile stress. Cheng *et al.* (2003) states that "*the crack extension initiates exclusively on the tensile free surface*" and also that "*compressive stresses normal to a crack will not cause fracture in brittle materials*". Fracture of biaxial samples tested using this method is therefore likely to be initiated at a crack or flaw on or near the surface of the material under tensile-tensile loading.

Analytical Solutions for 'Ball on Three Ball' Test

There are numerous analytical solutions for the ball-on-three-ball test method, each of which appears to be very different in nature. The most commonly used analytical solutions can be found in: Godfrey (1985); F394-78 Standard (1996); Ovri (2000); Higgs *et al*. (2000); Danzer *et al.* (2001); and Pagniano *et al*. (2005). An evaluation of these solutions was undertaken and is briefly described below with Table 1 defining the variables used in the analytical solutions presented herein.

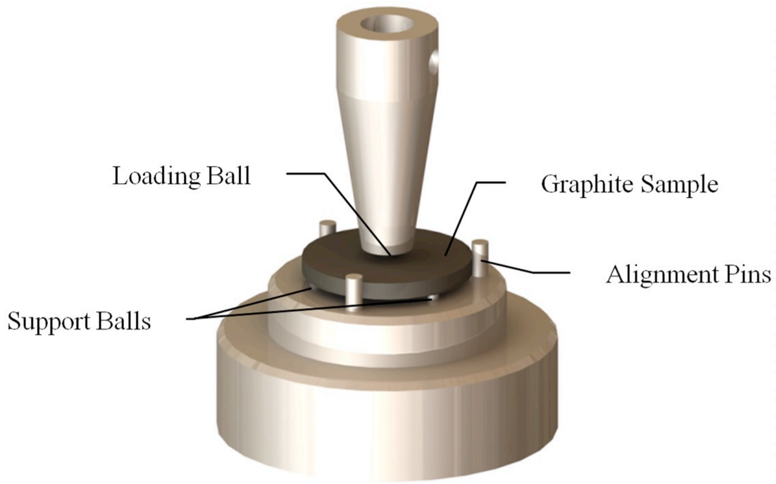

FIGURE 1: Rendered SolidWorks drawing of the Ball on three ball test apparatus

TABLE 1: Variables used in analytical solutions

Variable type	Variable	Symbol	Variable type	Variable	Symbol
Applied Condition	Load	L	Apparatus Conditions	Diameter of ball	D_b
Material Geometry	Thickness	T		Poisson's Ratio (Ball)	ν_b
	Radius	R_d		Young's Modulus (Ball)	E_b
Material Properties	Young's Modulus (Disc)	E_d		Support Radius	A
				Radius of ball	R_b
	Poisson's Ratio (Disc)	ν_d	Contact Radius Approximations	Godfrey	R
				Westergaard	b

Investigation into the derivation of all these solutions revealed that they are similar in their manipulation of both material properties and experimental values. They are in essence approximated based upon 'cylindrical symmetrical thin-plate theory' for truly elastic materials (Kirstein and Woolley, 1966) which predicts an infinite stress amplitude opposite to the load transfer point; and they can all be related to the master equation (Marshall, 1980) for the biaxial stress, σ, such that:

$$\sigma = YL / T^2 \qquad [1]$$

Our evaluation, however, has found that there are key differences regarding contact mechanics between the disc and the loading ball, and also the geometric factors used, *Y*. For example, Pagniano (2005), Equation [2], suggests that the radius of uniform loading at centre is equivalent to the radius of the loading ball.

$$\sigma = 3L\frac{(1+\nu_d)}{4\pi T^2}\left[1 + 2\ln\left(\frac{A}{R_b}\right) + \left(\frac{1-\nu_d}{1+\nu_d}\right)\left(1 - \frac{R_b{}^2}{2A^2}\right)\left(\frac{A^2}{R_d{}^2}\right)\right] \qquad [2]$$

However, another solution, proposed by Godfrey (1985), Equation [3], includes an approximation for the contact radius between the loading ball and the disc described by Equation [4] using Hertzian theory (elastic body interaction) and so takes into account the material properties of the indenter and the disc.

$$\sigma = 0.4775\left(\frac{L}{T^2}\right)(1+\nu_d)\ln\left(\frac{A}{R}\right)+\frac{1}{2}(1+\nu_d)+\frac{0.25(1-\nu_d)(2A^2-R^2)}{R_d{}^2} \quad [3]$$

$$R = 0.721\left[\frac{LD_b(1-\nu_b{}^2)}{E_b}+\frac{(1-\nu_d{}^2)}{E_d}\right]^{\frac{1}{2}} \quad [4]$$

Interestingly, solutions suggested by Higgs *et al.* (2000), Equation [5], and Danzer *et al.* (2001) also require an approximation for the contact radius. Both papers explain that Equation [4] is only valid for values of R which are larger than $1.7T$. Values of R smaller than $1.7T$ can be found by replacing the actual radius by an 'equivalent radius', b. An approximation for this 'equivalent radius' is given by Westergaard (1926), Equation [6].

$$\sigma = 3L\frac{(1+\nu_d)}{4\pi T^2}\left[1+2\ln\left(\frac{A}{R}\right)+\frac{(1-\nu_d)}{(1+\nu_d)}\left(1-\frac{R^2}{2a^2}\right)\frac{A^2}{R_d}\right] \quad [5]$$

$$b = \sqrt{1.6R^2+T^2}-0.675T \quad [6]$$

Further, ASTM F394-78 Standard (1996) and Ovri (2000) do not use any contact radius approximations. Rather, these papers suggest the use of a hardened dowel to apply the load to the sample. The area of applied load is therefore stated as the radius of the dowel. In order to apply these solutions to the ball-on-three-ball method, it may be necessary to substitute an approximation for the contact radius. However, it should be noted that ASTM have now withdrawn this standard.

EVALUATION OF ANALYTICAL SOLUTIONS

For each of the most commonly used analytical solutions, an evaluation was undertaken using MathCAD for the test apparatus illustrated in Figure 1. For notionally the same applied load the solutions were shown to give rise to large differences in predicted biaxial strength as shown in Figure 2. This graph demonstrates the difference in calculated values for biaxial strength by imputing the same variables into the six solutions. Bizarrely, three analytical solutions yield a negative biaxial strength. Evidently these solutions can not be valid for the conditions of the experiment. It is a likely that these solutions are applied to experiments using different materials or indeed samples of a very different geometry. Further modelling of the solutions using surface plots, illustrates the issue of scale when applying these solutions. Figure 3 shows that as the thickness of the sample increases, the sensitively to load decreases.

Figures 4a and 4b illustrate the issue regarding the contact approximation for the solutions. Figure 4a (solution from Godfrey (1985)) shows that this solution would predict a decrease is sensitivity as the thickness increases. The solution from Pagniano (2005) (Figure 4b) again predicts a decrease is sensitivity as the thickness increases. Additionally however, when large balls are use to support the disc, a negative biaxial strength is calculated. Evaluation of the suitability of the solutions reveals that, for this investigation, only Godfrey (1985), Pagniano *et al.* (2005) and Higgs *et al.* (2000) could be applied to this test arrangement. Preliminary calculations using the solutions suggested by these three papers do yield encouraging results, albeit with concerns regarding the scale of the samples. The remaining three solutions are evidently not suitable for the testing of graphite using the suggested sample geometry.

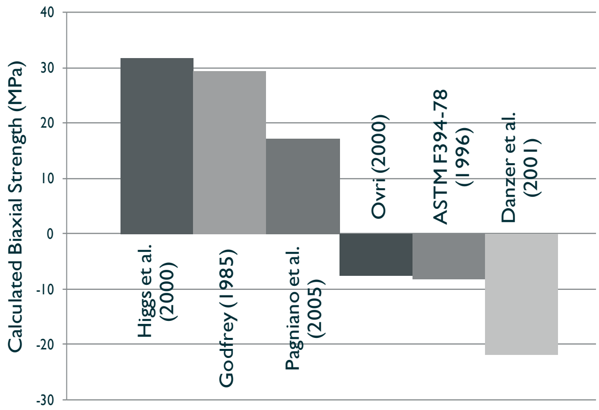

FIGURE 2: Biaxial strength of possible biaxial strength solutions

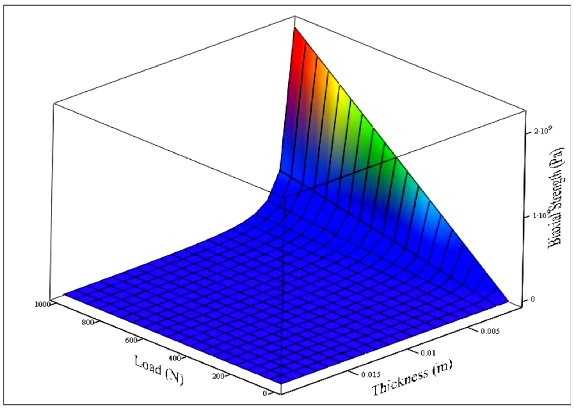

FIGURE 3: Relationship between load, sample thickness and biaxial strength (Godfrey, 1985)

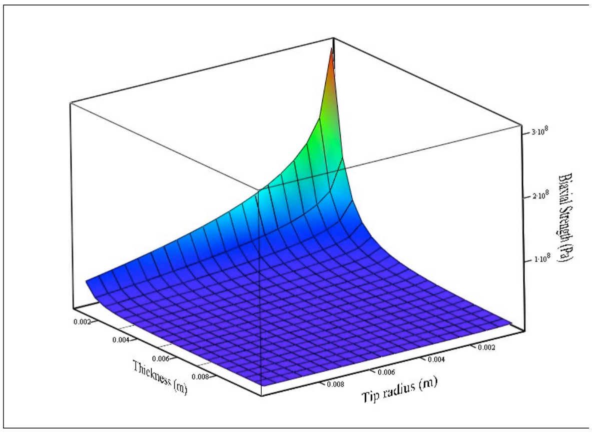

FIGURE 4a: Relationship between load, tip radius and biaxial strength (Godfrey, 1985)

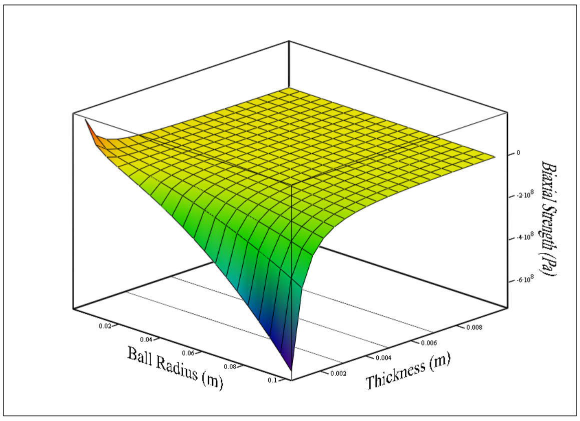

FIGURE 4b: Relationship between load, ball radius and biaxial strength (Pagniano, 2005)

EXPERIMENTAL DETAILS

The material investigated is EY9 grade graphite. The material is supplied in bars of 25.4mm diameter to allow easy machining into suitably sized specimens. Typical properties of the graphite are summarised in Table 2.

TABLE 2: Typical properties of investigated graphite

Property	EY9 (Williams *et al.* 1993)
Porosity (%)	17
Elastic Modulus (GPa)	13.1
Density (kg/m^3)	1677.0 (measured)
Tensile Strength (MPa)	14.11 (measured)
Compressive Strength (MPa)	51.0

Samples of EY9 grade graphite were prepared in thicknesses of 1, 2, 3, 4, 5 and 8 mm with a common diameter of 25mm. These samples were machined using a lathe. The material was first cut to the correct diameter. The individual specimens were then cut at the correct thickness using a 'parting off tool'. This process ensures that the faces of the sample parallel and the thickness is as accurate as possible. The specimens are then smoothed using fine silicon carbide paper.

The three ball bearing supports and loading ball were held in the correct position using grease. The sample was positioned using the pins on the base. The loading ball was lowered into a starting position just above the surface of the sample. A compression test was started, using a speed of 0.5mm/minute. The test continued until the specimen fractured, the load at this point (maximum load) is the value required to calculate the biaxial strength of the sample. NEXYGEN MT materials testing software was used to display and record properties of each test.

Determination of the materials uniaxial strength was achieved using the brittle ring test. This test comprises a ring sample which is compressed between two loading plates until fracture. Kennedy (1993) suggests that the brittle ring test results yield "*realistic estimates of strength for actual components*". The solution suggested by Kennedy to calculate the uniaxial strength of a sample is;

$$\sigma_{nom} = \frac{3P(a+b)}{\pi h(b-a)^2} \qquad \sigma_{\max} = \sigma_{nom} K_t \qquad [6a, 6b]$$

Where, P is the applied load, a is the inner radius of the sample, b is the outer radius of the sample, h is the thickness of the sample and K_t is a value read from a plot in Petterson (1953). Brittle ring samples were placed in the testing machine between two loading plates before a compression test was conducted at a speed of 0.5 mm/min. The test continued until fracture of the specimen occurred; the maximum load at this point was recorded

RESULTS AND DISCUSSION

The biaxial strength of EY9 graphite was calculated using three equations, all of which yielded different relationships between biaxial strength and sample thickness (shown in Figure 5). Higgs *et al*. Godfrey and Pagniano *et al*. showed an increase in biaxial strength as the thickness of the specimen increased, however, the Pagniano equation did result in lower strength values than Godfrey. Godfrey (WG) (Using Westergaard's approximation for contact radius) showed a roughly consistent value for biaxial strength as the thickness increased as did Higgs *et al*. (WG). Generally, the mean biaxial strength of a material tends to be lower than the uniaxial strength. Brocklehurst (1977), states that the biaxial strength of polycrystalline graphite is approximately 80 – 85% of the uniaxial strength. Brocklehurst goes on to explain that "*flaw mechanism of failure is that under a under a biaxial stress there is a greater chance of the larger flaws being critically oriented to a critical stress*".

Test data using the ball on three ball method suggest that the biaxial strength is generally higher than the uniaxial strength. This relationship contradicts the general relationship stated by Brocklehurst, that the biaxial strength is lower than the uniaxial strength.

The relationship shown by the five equations does not adhere to the theory of brittle fracture, whereby an increase in volume will result in a decrease in strength. This effect is attributed to the probability that a larger sample will be likely to contain critical flaws. A possible explanation for this effect is that as the thickness of the specimen increases, the sample experiences more compressive force from loading. Whilst the volume of the specimen is increased, the tensile area remains relatively constant. If this theory is correct, it could be concluded that the ball on three ball test method is not valid for samples which are subject to large amounts of compressive force. There was evidence of localised plastic deformation, caused by large compressive stresses at the point of loaded. This was particularly evident in the 8 mm thick samples which shows Hertzian cone cracking, resulting in a 'cup and cone' feature at the point of contact between ball and disc.

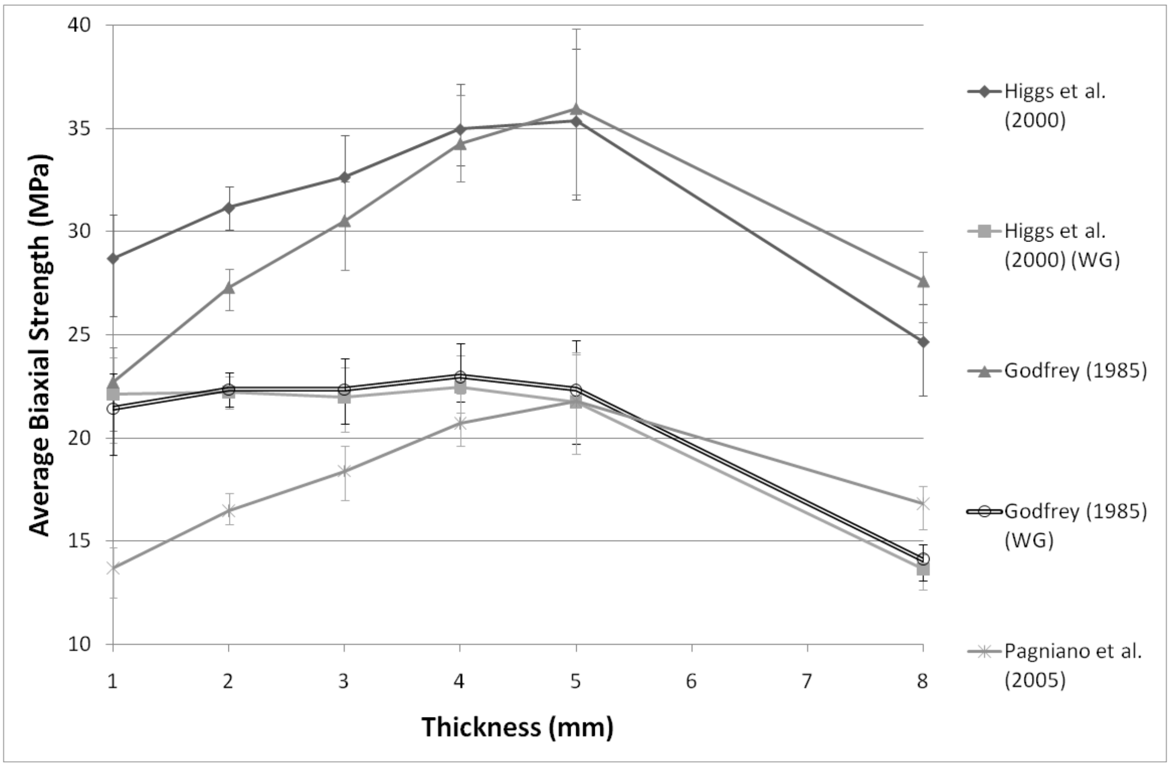

FIGURE 5: Calculated biaxial strength of EY9 grade graphite at varying thicknesses

A total of 10 brittle ring tests were undertaken for EY9 grade graphite. The average uniaxial strength was calculated as 14.443 with a standard deviation of 1.1044. Brocklehurst states that the compressive strength of graphite is typically 3 to 4 times higher than the tensile strength. EY9 Grade graphite has a compressive strength of 51.02 MPa (Williams *et al.* 1993), while the average tensile strength was calculated at 14.443. Using the aforementioned relationship yields an acceptable value of 3.53. The approach undertaken in this paper has also be applied to other materials such as POCO graphite (Easton, 2007) and Duratec 750 (Kipling, 2008). The results of these tests support the findings of this paper.

CONCLUSIONS

The biaxial strength values for EY9 grade graphite were higher than would have been predicted based on the materials uniaxial strength. The relationship between biaxial strength and sample thickness was also contrary to the expected trend. These results can be attributed to the stress distribution of the material. Finite element analysis on the 'ball on three ball' showed that the region of maximum tensile stress is very small, almost pin like Higgs *et al.* (2000). This effect also increases the effect of surface defects, since a flaw around the area of highest stress would have a large effect on the calculated strength of the material.

Disregarding the 8mm samples, which appears are subject to a more complex stress field during loading, the most appropriate results are generated using the solutions suggested by

Godfrey and Higgs and the contact approximation from Westergaard. The values for biaxial strength are higher than would be expected taking into consideration the relationship stated in Brocklehurst. These solutions do however yield the lowest calculated values for biaxial strength and are closest to the expected relationship between uniaxial and biaxial strength.

Analytical assumptions are likely to contribute to the higher calculated stress. The solutions are primarily used to calculate the biaxial strength of ceramics such as alumina, silicon nitride and glass. In each case the material being tested is brittle. Whereas graphite is commonly regarded as quasi brittle and it is unlikely that the analytical assumptions will take account of this factor.

References

ASTM F394-78 (Reapproved 1996). Standard test method for biaxial flexure strength of ceramic substrates.

Adams D. F. and Welsh J. S. (2002). An experimental investigation of the biaxial strength of IM6/3501-6 carbon/epoxy cross ply laminates using cruciform specimens. *Composites: Part A,* 33, 829-839.

Awaji H., Sato S., Kawamata H., Kurumada A., Oku T. (1987). Fracture criteria of reactor graphite under multiaxial stresses. *Nuclear Engineering and Design,* 103, 291-300.

Brückner-Foit A., Fett T., Munz D., Schirmer K. (1997). Discrimination of multiaxiality criteria with the Brazilian disc test. *Journal of the European Ceramic Society*, 17, [5], 689-696.

Cheng M., Chen W., Sridhar K.R. (2003). Biaxial flexural strength distribution of thin ceramic substrates with surface defects. *Journal of Solids and Structures 40* 2249–2266.

Brocklehurst J. E. (1977). Fracture in Polycrystalline Graphite. *Chemistry and Physics of Carbon*, [13], 145-279

Danzer R., Supancic P., Börger A. (2001). The ball on three balls test for strength testing of brittle discs: stress distribution in the disc. *Journal of the European Ceramics Society 22* 1425-1436.

Doyoyo M., Mohr D. (2003). Microstructural response of aluminum honeycomb to combined out-of-plane loading. *Mechanics of Materials*, 35, 865-876.

Easton, A. (2007). Determination of the Effect of Sample Geometry upon the Biaxial Strength of Engineering Materials.

Ernst E., Fett T., Rizzi G., Müller R., Oberacker R. (2006). A 3-balls-on-3-balls strength test for ceramic disks. *Journal of the European Ceramic Society*, (Corrected Proof, unspecified).

Feilzer A. J., Isgrò G., Pallav P., van der Zel J. M. (2003). The influence of the veneering porcelain and different surface treatments on the biaxial flexural strength of a heat-pressed ceramic. *The Journal of Prosthetic Dentistry,* 90, [5], 465-473.

Godfrey D. J. (1985). Fabrication, formulation, mechanical properties, and oxidation of sintered Si_3N_4 ceramics using disk specimens. *Materials Science and Technology,* 1, 510-515.

Han Y., Kumosa M. (1999). Non-linear finite-element analysis of Iosipescu specimens. *Composites Science and Technologies,* 59, 561-573.

Higgs R. J. E. D., Lucksanasombool P., Higgs W. A. J., Swain M. V. (2000). Evaluating acrylic and glass-ionomer cement strength using the biaxial flexure test. *Biomaterials 22,* 1583-1590.

Imaninejad M., Subhash G., Loukus A. (2004). Experimental and numerical investigation of free-bulge formation during hydroforming of aluminum extrusions. *Journal of Materials Processing Technology*, 147, [2], 247-254.

Kirstein, A.F., Woolley, R.M., (1967). Symmetrical bending of thin circular elastic plates on equally spaced point supports. *Journal of Research of the National Bureau of Standards 71C*, 1–10.

Kipling G.D. (2008) Determination of the Effect of Sample Geometry upon the Biaxial Strength of Engineering Materials

Kennedy C.R. (1993). The Brittle-Ring Test for Graphite. Carbon, Vol. 31. No. 3 519-528

Ovri J. E. O. (2000). A parametric study of the biaxial strength test for brittle materials. *Materials Chemistry and Physics,* 66, 1–5.

Pagniano R.P., Seghi R.R., Rosential S.F., Wang R., Katsube N. (2005). The effect of a layer of resin luting agent on the biaxial flexure strength of two all-ceramic systems. *The Journal of Prosthetic Dentistry* 459-466.

Perreux D., Suri C. (1997). A study of the coupling between the phenomena of water absorption and damage in glass / epoxy composite pipes. *Composites Science and Technology,* 57, 1403-1413.

Petterson R.E. (1953). Stress Concentration Design Factors. Wiley, New York, 135.