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COMPARISON OF ELASTIC MODULI OF POROUS CORDIERITE BY FLEXURE AND DYNAMIC TEST METHODS

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ABSTRACT

Previous work[1] showed differences in apparent elastic modulus between mechanical flexure testing and dynamic methods. Flexure tests have been conducted using non-contact optical systems to directly measure deflection for calculation of elastic modulus. Dynamic test methods for elastic modulus measurement were conducted on the same material for comparison. The results show significant difference in the apparent elastic modulus for static flexure versus dynamic methods. The significance of the difference in apparent elastic modulus on thermal stress and the hypotheses for these differences will be discussed.

INTRODUCTION

Young's modulus is a physical property derived from the strain response of a material in reaction to an applied stress (Hooke's Law). For a ceramic, this application of stress typically results in a linear strain response until brittle fracture at low strain values. The assumption of a homogeniety and isotropy for a polycrystalline sample is typically valid. The addition of porosity to the ceramic material does not usually change the fundamental application of linear elasticity, and one may assume that the material behaves according to a rule of mixtures, is homogenous and is isotropic. The derived elastic modulus is then an effective modulus. There are a large number of literature articles on relationships of elastic modulus to porosity in ceramics with a good summary given by Pabst[2]. Many semi-empirical relationships have been developed to model the modulus-porosity relationship with primary success for isotropic materials having porosity of 0.10 to 0.40 volume fraction. These relationships break down when higher porosity (<0.5 volume fraction) is encountered, as in the cordierite diesel particulate filter (DPF) materials. Since predictive equations for these higher porosity materials are lacking, experimental methods are necessary to evaluate the effective modulus.

Methods available to measure elastic modulus include static methods such as tensile, flexure, and nano-indentation and dynamic methods such as resonant ultrasound spectroscopy (RUS), dynamic mechanical analysis (DMA), pulse excitation, and sonic velocity. Static and dynamic methods have been compared for dense alumina, glass, aluminum and steel[3] with good agreement between static and dynamic methods. Extending this methodology to porous ceramics resulted in a finding of significant difference between apparent elastic modulus from static and dynamic methods[1]. Further work on cordierite ceramic was undertaken to determine if the differences between static and dynamic methods are due to inherent differences in material response to stress application or an artifact of the test method application. Understanding the difference is important as elastic modulus is used in many stress models to predict material response and fracture. A significant change in modulus would have a pronounced effect on the predicted stresses and subsequent reliability calculations.

EXPERIMENTAL

Sample filters of cordierite Duratrap AC[*] (nominal size 200 cpsi/8 mil wall[†]) were used to create specimens for testing. Multiple flexure bars were cut from two filters and randomized. The flexure bars were approximately 12 x 25 x 150 mm. Each flexure bar was tested for elastic modulus using a Buzz-o-sonic[‡] tester in accordance with ASTM E1876-09[4] for Out-of-Plane Flexure. Four specimen bars were then sectioned to make multiple two cell x four cell x 60 mm specimens for Dynamic Mechanical Analysis (DMA) testing. These specimens were tested using a Q800 DMA tester[§] with a three point bending fixture operated with a deflection amplitude of 5 μm in a frequency sweep with test points at 0.1, 1, 10 and 100 Hz. The remaining flexure specimens were tested in accordance with ASTM C1674-11[5] using a Sintech 20G universal test machine[**] with a 500 N load cell, fully articulating four point bend fixture with 13 mm rollers, 45 mm loading span and 90 mm support span. The crosshead speed for testing was 0.5 mm/min. The flexure specimens were separated into two groups with the deflection of each group monitored by non-contact methods. The two methods were Vision System (VS) and Digital Image Correlation (DIC). Method 1 was a National Instruments (NI) Vision Camera System[††] which measures deflection by identifying a group of pixels with a camera and then tracking the pixel cluster as the specimen deformed. Method 2 was a Vic-3D 2010 Digital Image Correlation System[‡‡] which measures deflection by using two cameras to track a speckle painted target. The speckle painting is similar to the selected cluster of pixels used by the NI Vision system. The center of the samples was tracked for deflection relative to the same left outer support. The camera systems were synced to the load cell output from the Sintech 20G load frame to develop the load deflection curve for determination of the elastic modulus.

Since the above test methods were developed for solid cross section specimens, the honeycomb structure of the specimens must be accounted for in the elastic modulus and stress calculations. To determine the moment of inertia, I, a minimum of ten measurements of the ceramic wall thickness and cell pitch were taken on each end of every specimen using a Keyence[§§] VHX-500 digital microscope calibrated at the magnification and focal length used for measurements.

The output from the different tests was fundamental response measurements of frequency and/or load and deflection. These measurements were then converted to apparent elastic modulus.

The resonant frequency from pulse excitation was used with the measured dimensions and mass and calculated moment of inertia to determine the apparent elastic modulus by Equation 1.

Equation 1[6]:

$$E = \frac{f^2 m L^3}{12.674 I}$$

where E, *f*, m and L are the Young's modulus, fundamental frequency, sample mass and length, respectively. For DMA, the apparent elastic modulus was recorded directly as the storage modulus in the results as calculated by Equation 2. Inputs to the program included the moment of inertia and cross section area of the honeycomb specimen. Cross section area was determined using the area calculation function in ImageJ[7].

Equation 2:

$$E' = \frac{\sigma_0}{\varepsilon_0} \cos \delta$$

where E', σ_0, ε_0 and δ are the Storage modulus (apparent Young's modulus), applied stress (corrected for moment of inertia), measured strain and phase angle between applied stress and measured strain, respectively. The apparent elastic modulus for mechanical tests was calculated from the load and deflection of the specimens by Equation 3. The deflection for VS and DIC were taken directly from the measurement output of the camera systems. The deflection measured by the crosshead movement was corrected for machine compliance using a known modulus material as a reference[8].

Equation 3[9]:

$$E = \frac{PL_S^3}{69.8Iy}$$

where P, L_S and y are the applied load, support span length and deflection distance, respectively, for a sample loaded in four point flexure with a moment arm of $L_S/4$.

RESULTS

The calculated apparent elastic modulus values for the dynamic and static test methods are shown in Figure 1. These results include corrections for moment of inertia of the honeycomb structure for all methods and machine compliance for the four point flexure method using crosshead deflection. Machine compliance was calculated using a copper bar with known elastic modulus of 124.5 GPa. The static test method (flexure) shows much lower elastic moduli (~50%) than the dynamic test methods (pulse excitation and DMA). Representative test outputs for each of the methods are shown in Figure 2.

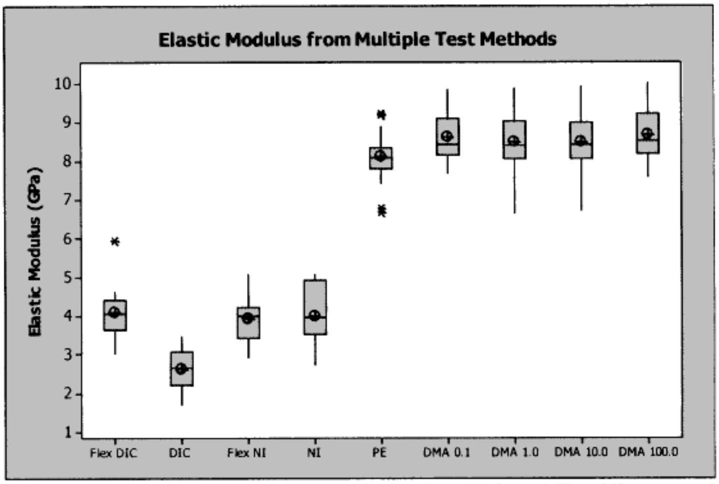

Figure 1 – Apparent Elastic Modulus comparison. Flex DIC and Flex NI are results using crosshead movement to calculate deflection. DIC (digital image correlation) and NI (National Instruments vision system) are results using non-contact measurement of deflection.

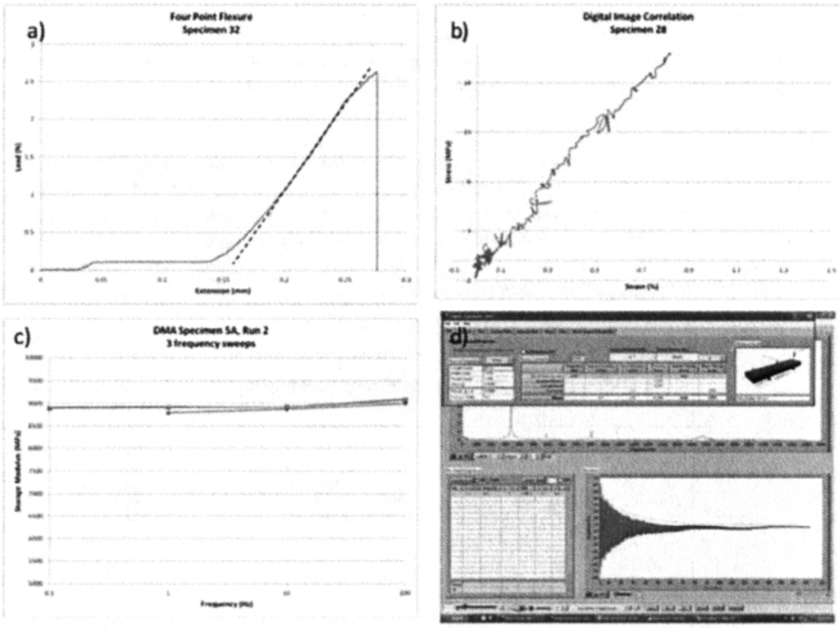

Figure 2 - Representative test output for each method. Outputs are different for each method a) load, b) stress, c) storage modulus, d) frequency.

DISCUSSION

A summary of the calculated elastic modulus data is shown in Table 1 with the t-test analysis between the different test methods. The comparison shows statistical differences in the sample populations between the static and dynamic test methods. The non-contact Vision System correlates with the two data sets for four point flexure using crosshead deflection. This is an indication that the correction for machine compliance works. The Digital Image Correlation has a lower value than the four point flexure and Vision System, and DIC is found to be significantly different. Review of the DIC set-up showed that the image area was restricted to approximately 65 mm of the support span due to design of the fixture occluding the view of the specimen surface. The reduced length of image area may be introducing an error in the deflection measurement. Further work on DIC is needed where the entire specimen surface is in view of the camera during the test to ensure accurate recording of the specimen deflection.

Comparison of the dynamic methods (pulse excitation and DMA) showed that there is a statistical difference between the methods. Additional comparison of the DMA method over four different frequencies showed no statistical difference between frequencies of 0.1 to 100 Hz.

Table I – Elastic Modulus Summary

Test		Elastic Modulus			t-test	
		Mean (GPa)	St Dev (GPa)	# data points	Comparison	P value*
1	4 point flex A	4.10	0.652	16		
2	Digital Image A	2.63	0.499	14	1 vs 2	0.000
3	4 point flex B	3.94	0.634	14	1 vs 3	0.483
4	Vision System B	4.01	0.724	15	3 vs 4	0.783
5	Pulse Excitation	8.10	0.547	40	5 vs 7	0.001
6	DMA 0.1 Hz	8.62	0.583	98		
7	DMA 1 Hz	8.50	0.634	122	6 vs 7	0.156
8	DMA 10 Hz	8.50	0.646	123	6 vs 8	0.154
9	DMA 100 Hz	8.68	0.610	137	6 vs 9	0.438

* - P value is a statistical measurement for correlation of sample populations. P value < 0.05 indicates that the sample populations being compared do not have equivalent mean values.

Since there is a statistical difference between the static and dynamic methods, there is now a question of why these results are different. The cordierite material has a microstructure of distributed porosity and intentionally formed microcracks which results in the desired low thermal expansion behavior (< 1 ppm/°C between 25 and 1000 °C) for application in exhaust aftertreatment systems. While the distributed porosity would be expected to behave as an additive phase in contribution to the apparent elastic modulus (rule of mixtures) the microcracks present an additional contribution. A hypothesis would be that the microcracks participate in the measurement of the apparent modulus if there is sufficient time for the crack to open or close during active measurement. At high frequency, with very low strain, there would not be sufficient time for crack movement and the microcracks would have no effect on the apparent modulus value and the assumptions of linear elasticity in a homogeneous, isotropic material are valid.

Another hypothesis would be that the mechanical test produces larger strains and the interaction of the microcrack opening/growth with larger strains would cause the load-deflection response to deviate from linear elasticity. In an attempt to test the possible frequency effect, the DMA test was run over a range of frequencies. This showed no difference in apparent modulus. However, the minimum DMA frequency of 0.1 Hz still is much greater than the effective frequency in a flexure test (on the order of 0.0006 Hz) so the frequency difference is too great to draw any conclusions. Tests where the strain application is on the order of 0.01 and 0.001 Hz are needed to provide further insight. Mechanical testing by flexure to reach strain applications in the range of dynamic measurements will require higher precision in the deflection measurement. A non-contact system (Vision System or Digital Image Correlation) with a higher resolution load cell should provide this capability.

In addition to four point flexure testing, there is current work[10] using O-ring compression and biaxial flexure with micro-FEA to determine the apparent elastic modulus of porous honeycomb cordierite ceramic. This work has also confirmed that the apparent mechanical modulus determined from a static (mechanical) test method is significantly lower that the apparent modulus from a dynamic test method. The biaxial flexure results are nearly equivalent to these four point flexure results.

The significance of understanding apparent elastic modulus is in the impact which a change in modulus has on the final application of the material. The specific use of cordierite in diesel aftertreatment systems for particulate filters utilizes elastic modulus as a modeling input to convert temperature profiles into stress profiles. An example of this is shown in Figure 3.

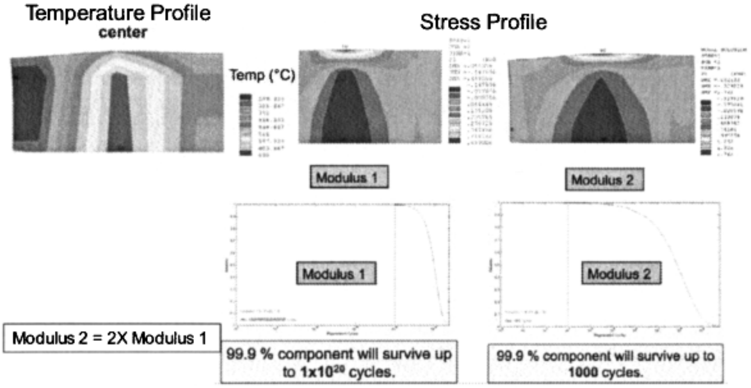

Figure 3 - Thermal Stress and Durability[11]

The stress profile and apparent elastic modulus are also used as inputs to the durability models which have been created as tools to predict the lifetime use for the final aftertreatment components. The effect of a modulus change on the durability prediction is shown in the lower section of Figure 3 where a change of modulus by a factor of two results in a prediction of 17 orders of magnitude reduction in durability. From this result, it can be seen that the use of an accurate and reasonable value for material elastic modulus is required to have a prediction of useful life. In addition, a fundamental rethinking of the meaning of linear elastic constants in the case of porous microcracked ceramics may be necessary.

CONCLUSIONS

Dynamic measurement (resonance) and static measurement (mechanical) produce different values for elastic modulus of porous cordierite ceramic. The elastic modulus from resonance is a measure of the material response at very low strain which is different from the material response in a mechanical test with relatively large strain. The apparent elastic moduli for dynamic versus static test methods in this study are different by a factor of two. This result has significant impact on calculated stress and life in an aftertreatment component.

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[*] Corning, Inc, Corning, NY

[†] Standard terminology for extruded honeycomb is English units - cells per square inch (cpsi) and mil (0.001 inch)

[‡] BuzzMac International, Glendale, WI

[§] TA Instruments, New Castle, DE

[**] MTS Corporation, Eden Prairie, MN

[††] National Instruments, Austin, TX

[‡‡] Correlated Solutions, Inc., Columbia, SC

[§§] Keyence Corporation, Osaka, Japan