

Failure Stress and Apparent Elastic Modulus of Diesel Particulate Filter Ceramics

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ABSTRACT

Three established mechanical test specimen geometries and test methods used to evaluate mechanical properties of brittle materials are adapted to the diesel particulate filter (DPF) architecture to evaluate failure initiation stress and apparent elastic modulus of the ceramics comprising DPFs. The three custom-designed test coupons are harvested out of DPFs to promote a particular combination of orientation of crack initiation and crack plane. The testing of the DPF biaxial flexure disk produces a radial tensile stress and a crack plane parallel with the DPF's longitudinal axis. The testing of the DPF sector flexural specimen produces axial tension at the DPF's OD and a crack plane perpendicular to the DPF's longitudinal axis. The testing of the DPF o-ring specimen produces hoop tension at the DPF's original outer diameter (OD) and at the inner diameter of the test coupon, and a crack plane parallel to the DPF's longitudinal axis. The testing of these mechanical test coupons also enables the determination of an apparent elastic modulus of the DPF ceramic material. Results consistently show that the apparent elastic modulus of the DPF ceramics is much less (e.g., up to an order of magnitude less) than apparent elastic modulus estimated using sonic- or resonance-based test methods. These specimen geometries, produced stress states, and modes of consequential crack initiation are discussed in context to the DPF's symmetry along with the assessments of each specimen's ease of fabrication, testing, failure stress determination, ultimate viability, and prospects for test standardization. Lastly, an explanation is offered for why the apparent elastic modulus measured with these three mechanical test coupons is relatively low and why their low values are more accurate for predicting thermomechanical stresses in DPFs.

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INTRODUCTION

Diesel particulate filter (DPF) technology enables the fuel efficient diesel engine to meet emission regulations for particulate matter. Examples of DPFs are shown in [Fig. 1](#). The DPF collects particulate from the exhaust stream during operation. It then is periodically regenerated by increasing its temperature to nominally 600°C. This causes the carbon-containing trapped particulate to oxidize and form gaseous CO₂ enabling continued filtration at a lower backpressure. But the regeneration causes thermal gradients in the brittle ceramic which can be problematic.

Two lifetime-limiting consequences of overly severe thermal gradients are crack initiation and propagation. The end crack and ring crack are examples of crack types that form in DPFs and are schematically shown in [Fig. 2](#). The formation and existence of either can compromise the intended filtering function of the DPF and ultimately result in their need for replacement. Therefore, the end-user has interest in managing the operating conditions so the DPF

successfully fulfills its purpose without initiating those cracks.

Two criteria must be satisfied in tandem to promote and achieve maximum service lifetimes in DPFs under severe operating conditions. The ceramic comprising the DPF must have sufficiently high tensile strength and the tensile stresses (such as those caused by operating thermal conditions) must be sufficiently low. Obviously, if the anticipated operating conditions produce high tensile stresses, then a stronger DPF should be considered for use. Or if the only available candidate DPF ceramics have a limited tensile strength, then the operating conditions need to be restrained so that too-high tensile stresses do not arise. In either circumstance, the understandings of both the DPF ceramic strength and the DPF service conditions are needed to successfully employ a DPF.

The developed service stress state in a DPF can be complex owing to its anisotropic architecture. The DPFs examined in this study have a 4-fold rotational symmetry

(C4) that is illustrated in Fig. 3. The thermal gradients (and consequential mechanical stresses) for operation will of course be affected by this anisotropic structure, and out of convenience may be interpreted with respect to cylindrical coordinates; namely, axial (i.e., gas flow direction), radial, and hoop orientations.

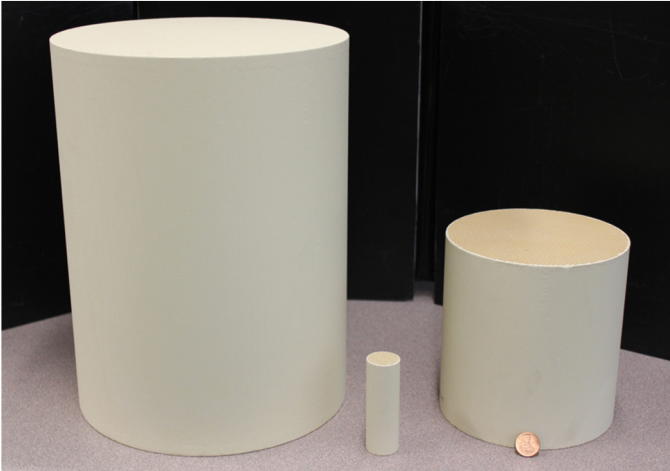


Figure 1. Diesel particulate filters examined in this study.

Ceramics almost always crack because of the existence of a too-high (First Principal) tensile stress, and the formation of the two crack types schematically shown in Fig. 2 may be described in context to that and the DPF's architecture. The plane of the end crack is axial or parallel to the gas flow direction and intersects the DPF's end. The end crack forms as a consequence of sufficiently high radial tensile stresses existing at the DPF end. The plane of the ring crack is perpendicular to the gas flow direction and intersects the outside diameter of the DPF. Its location is usually near the middle of the axial length and forms as a consequence of sufficiently high axial tensile stresses existing at the exterior of the DPF. In general, when ceramics crack in the presence of a thermal gradient (particularly when there is concurrent confinement), crack initiation usually occurs near the cold side of that gradient and often when there is a sufficiently rapid heating or cooling transient (i.e., thermal shock).

To successfully predict thermomechanical stresses (and predict crack or failure initiation), knowledge of the thermoelastic properties, elastic properties, and tensile failure strength of the DPF ceramic must be known or measured. For measuring tensile failure strength, a standardized test method, ASTM C1674 [1], is available. The failure stress is determined using classical beam bending analysis and follows without *a priori* knowledge of the material's elastic modulus. The specimen for that test method is a prismatic bar, harvested from the interior of a DPF as shown in Fig. 4, and is mechanically tested in 4-point flexure. Several have reported on failure stress data on cordierite ceramics DPF using its method or precursors to it [2,3,4,5].

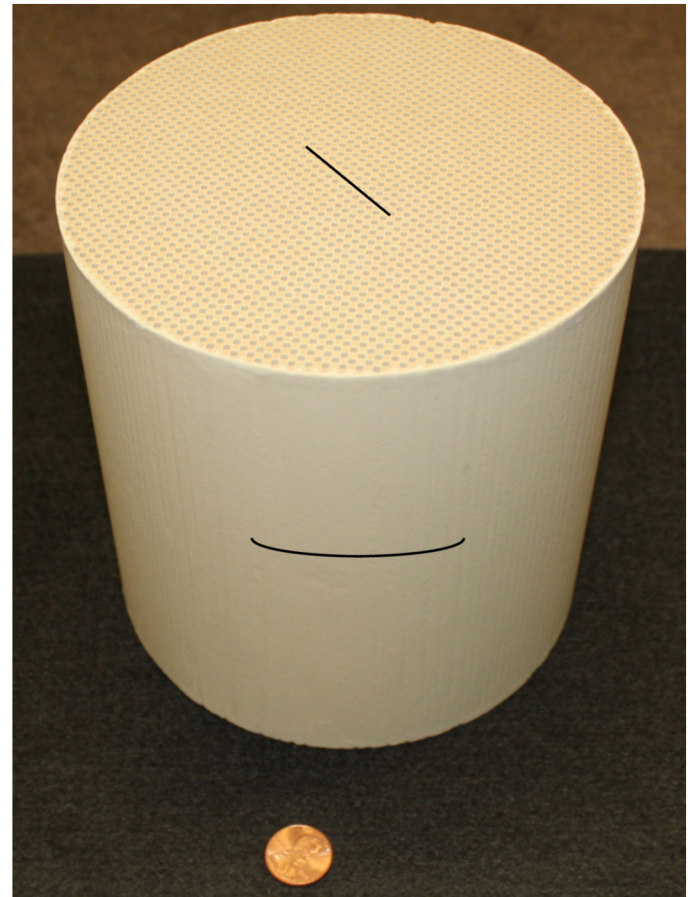
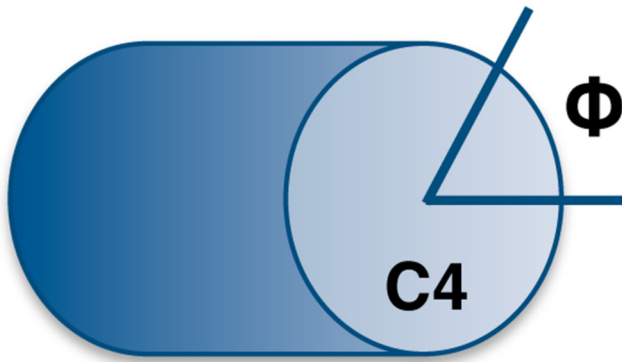


Figure 2. Schematics of (top) end crack and (middle) ring crack superimposed on a DPF. The plane of the end crack is parallel to the DPF's longitudinal axis and intersects the DPF's end face. The ring crack is perpendicular to the DPF's longitudinal axis and intersects the outside diameter usually near the middle of the DPF's length.

A limitation with ASTM C1674 is that it only evaluates tensile failure stress of the interior strut structure of a DPF; such a measured tensile failure stress is perhaps not relevant to either of the crack types shown in Fig. 2 nor of use for predicting the conditions where their crack types initiate. Therefore, a motivation exists to develop mechanical test coupons whose combination of produced crack-plane and crack propagation direction represents the two crack types in Fig. 2.



Such porous structures often have an asymmetric response to tensile and compressive loadings. Tensile loading of the compliant porous structure can readily cause initiation, coalescence, and accumulation of local microcracking resulting in a gradual compliance increase with additional tensile straining. While compressive loading will also cause microcracking, additional compressive straining beyond its initiation can lead to little change in compliance with additional microcracking. The tensile failure stress, like for almost all ceramics, has a much lower magnitude than compressive failure stress, so rationale exists to focus attention on the tensile failure stress for appropriate and conservative DPF reliability analysis.

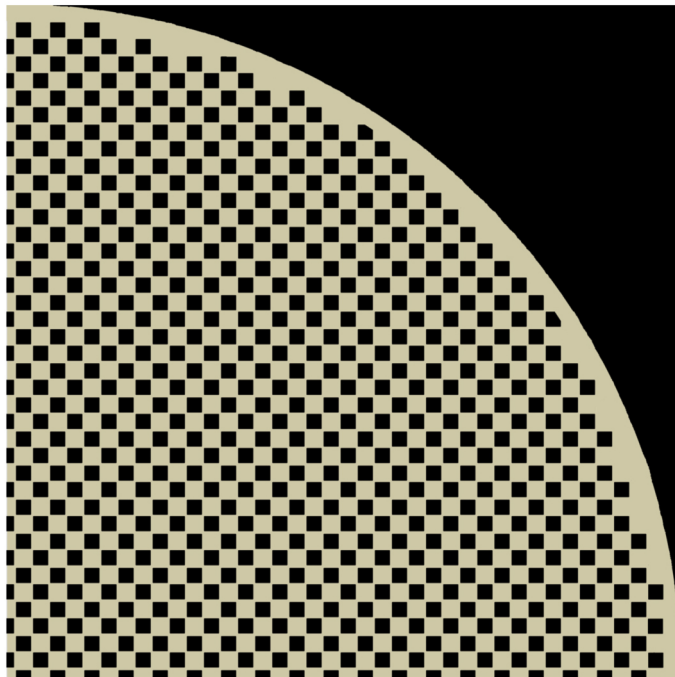


Figure 3. DPFs have a 4-fold rotational symmetry (designated as “C4”). Bottom image shows 50% channel fill for the end structure of a DPF for a $\phi = 90^\circ$.

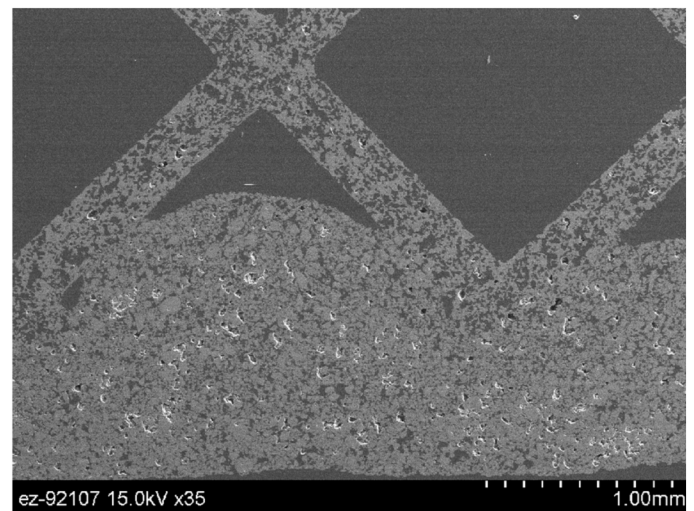


Figure 5. Polished microstructure of a cordierite DPF showing porous open structure in cross-sectioned struts and the closed porous structure of the outer skin (bottom).

There were three primary goals to this study. The first was to employ test specimens whose mechanical loading would produce tensile-stress-induced failure modes consistent with those of the crack types shown in Fig. 2 and that would enable quantification of tensile failure stress associated with their formation. Flexure testing works well for achieving that and tends to provide greater ease of valid experimentation than uniaxial tension testing does, and also is readily adaptable to high temperature testing too. Test specimen geometries, unlike the prismatic bar featured in C1674, used in the mechanical evaluation of other types of ceramics were adapted to the mechanical testing of the DPF architecture and ceramic to better facilitate the understanding of the end and ring crack in Fig. 2. The second goal was to also use analyses that enable the estimation of the DPF ceramic's elastic modulus - a property needed for any eventual modeling of the DPF's operational stress state. The third goal was to satisfy the first and second goals by evaluating the cordierites in three different sized DPFs (shown in Fig. 1). Their geometrical characteristics are shown in Table 1.

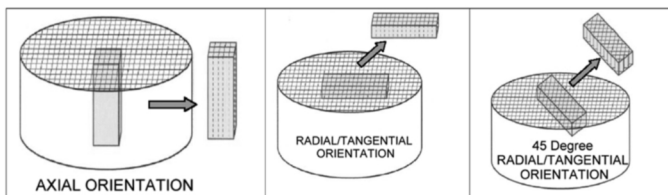


Figure 4. Prismatic bar harvesting for flexure testing described in ASTM C1674.

While the porous microstructure of DPF ceramics is a requirement for exhaust gas filtering it also produces a complex response when subjected to (thermo) mechanical loading. The open and continuous (filtering-capable) porous structure of the interior square struts and the closed, less porous, structure of the exterior skin are shown in Fig. 5.

Table 1. Characteristics of the three evaluated DPF sizes.

Nominal Diameter (mm)	Length (mm)	Measured # of Cells per sq. in.	Manuf. Reported Porosity
25	75	256	59%
141	156	200	52%
229	305	256	52%

THREE ALTERNATIVE SPECIMENS

The descriptions of the equibiaxial flexure specimen, sector flexure specimen, and the o-ring flexure specimen are presented along with their mechanical testing and respective analyses used to determine elastic modulus and tensile failure stress. All three of those specimen geometries were prepared from the three DPF sizes shown in Table 1 producing nine different specimen type and size combinations. All three are as easy, or easier, to harvest out of a DPF as the prismatic bar geometry used in ASTM C1674 (Fig 4).

APPROACH

The method to estimate elastic modulus and tensile failure stress with all three alternative test specimens is represented by the flowchart in Fig. 6. The method combines analysis (either analytic or finite element) with mechanical testing. The mechanical testing of all the test coupons produces a record of compressive force (all three test methods are flexure tests and compressive loading coupled with appropriate fixturing activates their flexure) and an associated linear specimen deflection up through a maximum compressive force where crack initiation begins. All three specimen geometries responded linear elastically up to that maximum compressive force. The coupled pair of maximum-compressive-force and deflection data is recorded for every specimen after the important correction (subtraction) for machine compliance. The geometry of that actual specimen is then modeled using FEA (equibiaxial specimen and o-ring specimen) or classical beam bending (sector flexure specimen) and displacement as a function of compressive force and elastic modulus is predicted. Elastic modulus is then iteratively varied until a produced force-displacement couple finally matches the force-displacement couple measured from the flexure testing. That elastic modulus (secant elastic modulus actually) is then used again in the FEA to estimate the maximum tensile failure stress in that specimen when failure initiation began.

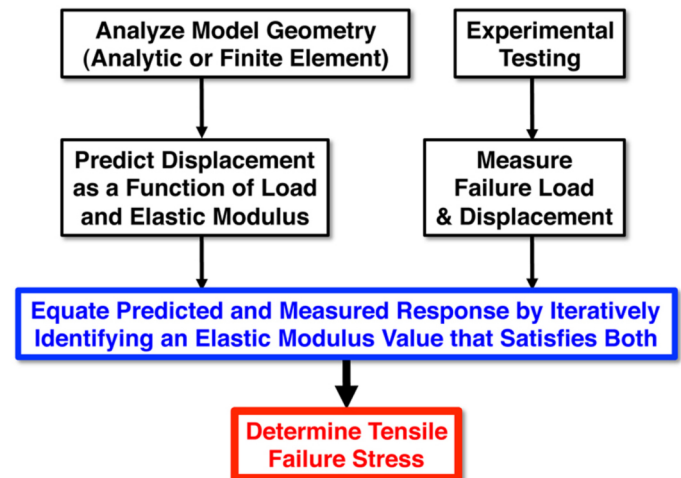


Figure 6. Analysis of all three alternative test coupons were combined with their experimental testing to (1) estimate secant elastic modulus of the DPF cordierite and (2) failure stress.

EQUIBIAXIAL FLEXURE (RADIAL TENSION)

The testing of the equibiaxial flexure specimen subjects the DPF's end structure (or the interior strut structure if the disk is cut from the interior) to a biaxial radial tensile stress, and its measured response is relatable to the interpretation of end-crack formation illustrated in Fig. 2.

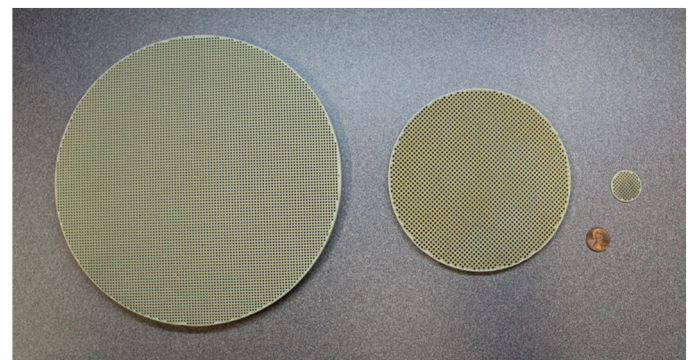


Figure 7. Examples of equibiaxial flexure disks sliced out of three different DPF diameters.

The equibiaxial flexure specimen and test are a standard method (ASTM C1499) used for measuring failure stress in dense ceramic materials [6] and it was adapted here to measure radial tensile failure stresses in DPFs. An advantage of its use with disks harvested out of a DPF is its produced biaxial flexure stress state will result in failure of the weakest direction in the biaxially stressed plane as compressive force is increased. Examples of the equibiaxial flexure specimens and tested coupons are shown in Figs. 7-8, respectively. The geometry, applied compressive force, and resulting disk deflection for the equibiaxial flexure specimen (aka, ring-