

Prediction of Refractory Strength Using Nondestructive Sonic Measurements

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In recent years the use of nondestructive sonic testing of industrial refractory products has become a generally accepted technique for evaluating and comparing product properties, quality, and uniformity. Because of the technical validity of the data, the ease of use, and the portability of the equipment, the method has the potential for even wider use and increased significance. With the ongoing trend toward higher cost and higher quality refractories, it is expected that the use of nondestructive sonic testing will increase significantly in coming years, because of the increasing expenses associated with manpower expended, equipment costs, and the loss of product (by destructive testing). Likewise, ongoing research on sonic test equipment can be expected to result in improved analytical capabilities and better measurement sensitivity to permit increased utility of the method. Through the use of sonic testing it is possible to greatly expand the scope and significance of refractory testing, in a cost and time effective manner, while also enhancing the understanding of the refractory production process. Based upon the initial establishment of valid property correlations, using the relationship(s) of destructive test results and nondestructive sonic test data, it is possible to thereafter utilize only the sonic test data to characterize and monitor selected properties of refractories. Sonic testing also allows the detection of internal laminations, areas of low density, and other defects, which means that the technique(s) permits very good overall characterization of the quality of refractories.

Various publications and presentations have dealt with the practical application of sonic testing to characterize and monitor the properties of industrial refractory materials nondestructively. Works by Davis^{1,2} reported the use of sonic data to determine the correlation between modulus of elasticity and flexural strength for fireclay brick. Other studies followed on a limited basis, but only in recent years has the use of sonic testing become common. Semler³ discussed the use of ultrasonic data to monitor the strength degradation/damage in high alumina refractories resulting from thermal shock exposure in lab tests. Judd et al.⁴ discussed a testing program which included correlation of ultrasonic velocity measurements with the porosity and density of refractory pouring tubes; they determined statistically valid confidence limits for several tube suppliers which became the basis for 100% testing (acceptance/rejection) of all incoming pouring tubes. A presentation by Whittemore⁵ considered the use of ultrasonic data to evaluate pouring pit refractories. Dunworth⁶ discussed the use of ultrasonic data to monitor in-plant the production of slide gates. Miller⁷ described a program in which correlations were developed between porosity, density, and strength properties of various refractory types. Lawler et al.⁸ conducted an extensive evaluation of fireclay coke oven refractories, in which ultrasonic measurements were correlated with crushing strength to give statistically valid criteria for field evaluation of the quality of the complex shapes. Semler⁹ considered the general aspects of the ultrasonic testing of refractories and discussed several practical examples of applications, including the statistical correlation of ultrasonic velocity and refractory strength. Canelli and Monti¹⁰ discussed the use of ultrasonics to inspect slide gate products, especially for monitoring and

Mathematical relations were evaluated and equations are presented correlating non-destructive sonic measurements, using either ultrasonic velocity or modulus of elasticity, with modulus of rupture for five representative fired refractory product types, including high alumina (90%), mullite (70% alumina), super-duty fireclay (42% alumina), vitreous silica (99% silica), and carbon. The experimental relations were tested for additional brands and found to yield predicted strengths that were within 0.5% to 6% of the actual strengths for refractory products having properties similar to the model refractory product used to develop the correlation equation. For refractory products with properties that were dissimilar to the model refractory, the predicted strengths differed from the actual strengths by >20%.

maintaining control of production parameters. Petit¹¹ reports the results of an evaluation of ladle refractories in which sonic resonance modulus of elasticity data were correlated with porosity, density, and strength properties to establish a basis for ongoing nondestructive evaluation of the quality of future shipments. Kawai et al.¹² discuss the detection of flaws in sintered SiC plates using an ultrasonic probe; a relationship was established between sonic response and the flexural strength of the plates. Russell and Morrow¹³ report the results of ultrasonic testing of slide gates and shroud tubes, performed to establish the statistical basis of evaluating the quality of the pieces received nondestructively; the data were correlated with the porosity, density, and strength of the refractories.

As shown by many of the above studies, a direct relationship can be demonstrated between sonic measurements, both ultrasonic velocity and modulus of elasticity by sonic resonance, and various physical properties. Davis^{1,2} determined the ratio E/B (E = modulus of elasticity and B = modulus of rupture) for different kinds of aluminosilicate refractories based on the fact that the room temperature stress-strain relationship for these brittle materials is linear up to the breaking point, i.e. the breaking point lies on a straight line given by the following equation:

$$\sigma_c = \epsilon_c E$$

where σ_c = breaking stress, E = modulus of elasticity, and ϵ_c = strain at failure.

The study described herein was conducted to evaluate and illustrate in more detail the correlation of sonic measurements with the mechanical strength of a broader range of industrial refractory products than has been reported in previous studies.

Experimental Procedure

Five general categories of fired commercial refractory products were chosen for study, including high alumina (90%), mullite (70% alumina), super-duty fireclay (42% alumina), vitreous silica (99% silica), and carbon. Multiple samples of each representative refractory type were evaluated by two nondestructive sonic techniques followed by determination of the modulus of rupture (flexural strength by destructive test). The test results were evaluated to

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Table I. Model Refractory Types and Property Ranges Included in Study

Refractory type	MOR (MPa)	Ultrasonic velocity (m/s)	MOE (GPa)
High alumina (90%)	8.2–26.0	2130–4900	27.5–65.5
Mullite (70% Al ₂ O ₃)	9.6–23.0	3050–5500	37.9–79.5
Fireclay (42% Al ₂ O ₃)	2.4–12.4	1830–4120	5.5–31.0
Vitreous silica (99%)	2.4–6.9	1525–2450	4.1–11.2
Carbon	13.8–22.8	2750–3660	11.7–17.2

derive the best mathematical correlation between both of the sonic measurements (ultrasonic velocity and the sonic resonance modulus of elasticity) and the modulus of rupture.

The test procedures and equipment used in this study are described in further detail below.

Ultrasonic Velocity

A commercial ultrasonic testing instrument* of transmission type was used to evaluate each refractory brick sample. The instrument consists of a pulse generator and timing circuit coupled to two transducers (150 kHz) that were firmly positioned manually at opposite ends (0.229 m (9 in.) length) of each test brick. Each transducer had a 0.0016 m (1/16 in.) thick rubberized end covering to help overcome measurement problems due to the roughness of the brick surface. Knowing the pulse time and the path length, the ultrasonic velocity was calculated using the equation: Ultrasonic Velocity (m/s)=Path Length (m)×Time (×10⁻⁶ s).

Modulus of Elasticity (MOE)

The modulus of elasticity by sonic resonance was determined for multiple samples of each type using a commercial testing instrument.† The instrument permits determination of the resonant frequency of a sample by monitoring and evaluating the vibrational harmonics of the sample with a transducer; the vibrations are physically induced in the sample by tapping. The modulus of elasticity was calculated using the experimentally determined resonant frequencies, according to standard test method ASTM C-885.

Modulus of Rupture (MOR)

The modulus of rupture was determined for full-sized brick (0.229 m (9 in.) by 0.114 m (4.5 in.) by 0.064 m (2.5 in.)) using a universal tester‡ with 1.78 × 10⁶N capacity, according to standard test method ASTM C-133. The loading rate was 29.9 N/s, applied automatically, and the sample support span was 0.178 m (7 in.). Calibration of the instrument showed the load readings to be within 1% of standard values.

Results and Discussion

For each of the five representative refractory types included in this work, samples representing as broad a property range as possible were studied. Table I shows the pertinent property ranges for modulus of rupture, modulus of elasticity, and ultrasonic velocity that were included in this study.

The correlation between ultrasonic velocity and room temperature modulus of rupture was evaluated to determine the best mathematical fit (equation) for the data. Both linear and exponential relationships were considered. It was observed in all cases that the exponential relationship gave the higher correlation coefficient, ranging from 0.92 to 0.82. For the linear basis, the correlation coefficients ranged from 0.86 to 0.72.

Table II. Experimentally Derived Exponential Relation Between Modulus of Rupture and Ultrasonic Velocity

Refractory type	Mathematical relation*	Number of samples	Correlation coefficient
High alumina (90%)	MOR = 2.87 e 4.75 × 10 ⁻⁴ V	92	0.92
Mullite (70% Al ₂ O ₃)	MOR = 3.70 e 3.19 × 10 ⁻⁴ V	50	0.91
Fireclay (42% Al ₂ O ₃)	MOR = 2.53 e 3.78 × 10 ⁻⁴ V	95	0.86
Vitreous silica (99%)	MOR = 0.43 e 11.15 × 10 ⁻⁴ V	38	0.92
Carbon	MOR = 5.29 e 4.02 × 10 ⁻⁴ V	35	0.82

*MOR in MPa and V in m/s.

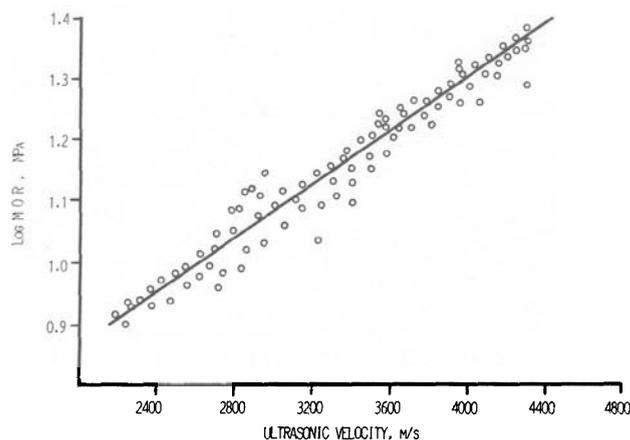


Fig. 1. Plot showing all data points for 90% alumina model refractory product, from which best-fit correlation was derived using an exponential relation. This plot illustrates data trend that was observed for each model refractory product studied.

The correlation of ultrasonic velocity and the modulus of rupture (MOR) was found to best conform to the general exponential equation:

$$\sigma_c = Ae^{BV}$$

where σ_c = modulus of rupture, MPa, A = material-dependent constant, B = slope from semilog plot, and V = ultrasonic velocity, m/s.

The specific exponential equation determined for each of the representative refractory types is shown in Table II. A representative plot of log MOR vs ultrasonic velocity, for the fired high alumina (90%) refractory type, is shown in Fig. 1, to illustrate the relationship observed. Plots of the experimental data for the other fired refractory products showed the same relationship, but are not included herein.

Correlation of the modulus of rupture with the modulus of elasticity per the classical stress/strain relationship is given by the following equation:

$$\sigma_c = KE$$

where σ_c = modulus of rupture, MPa, K = material-dependent constant, and E = modulus of elasticity, GPa.

The correlation coefficients determined for this linear relationship ranged from 0.94 to 0.81. The constant (K) determined for each of the refractory types is shown in Table III. A composite plot of MOR vs MOE for each of the representative refractory types studied is shown in Fig. 2. The general property ranges for each of the refractory products can be observed in this plot. The slope of the line for each of the refractory products gives the K value (constant).

To test the general utility of the experimentally derived equations a predicted modulus of rupture was calculated from nondestructive sonic measurements for several other refractory products different from those for which the mathematical relationships were established. The predicted and actual strength comparison results are presented in Table IV. A summary of these comparative results is shown in Table V.

*V Meter, James Electronic Co., Chicago, IL.
 †Grindosonic, J. W. Lemmens Co., Leuven, Belgium.
 ‡Tinius-Olsen Co., Willow Grove, PA.

Table III. Experimentally Derived Constant for Linear Relation Between Modulus of Rupture and Modulus of Elasticity

Refractory type	Constant, K	Number of samples	Correlation coefficient
High alumina (90%)	409×10^{-6}	55	0.94
Mullite (70% Al_2O_3)	322×10^{-6}	50	0.93
Fireclay (42% Al_2O_3)	400×10^{-6}	85	0.89
Vitreous silica (99%)	570×10^{-6}	37	0.90
Carbon	1245×10^{-6}	35	0.81

The data summarized in Table V show the excellent to very bad correlation of the predicted and actual strength values. Excellent correlation is seen in those cases where the actual and calculated strengths are within 1%. Reasonable correlation is considered to be 1% to 10% difference, and bad correlation is greater than a 10% difference. The basis for the wide range (% difference) between the calculated and actual strength values, from 0.6% to 76%, can seemingly be explained on the basis of differences in the physical and chemical character of the refractories compared. To obtain good correlation of results, a statistically valid mathematical relationship must be used. But the validity of each experimentally derived mathematical relationship is limited to refractories of the same or very similar chemistry and bonding structure. Various examples of this limitation in strength correlation are seen in the data summary shown in Table V. In the case of high alumina (90%), mullite (70%), and superduty fireclay, the additional brands chosen to test the mathematical relationships had similar bulk chemistry, based upon comparison of the product data sheets, and it is felt that the physical structure was very similar to the appropriate model refractory based upon the descriptions of the products, although it was not quantitatively evaluated on a comparative basis; for these similar products, strength correlations of less than roughly 6% difference were observed, for either ultrasonic velocity or modulus of elasticity measurements. The high duty fireclay, silica brick, and carbon brick all differed from the model products and the strength correlations all had a percent difference greater than roughly 20%. The high duty fireclay product had a nominal chemistry similar to the model superduty fireclay

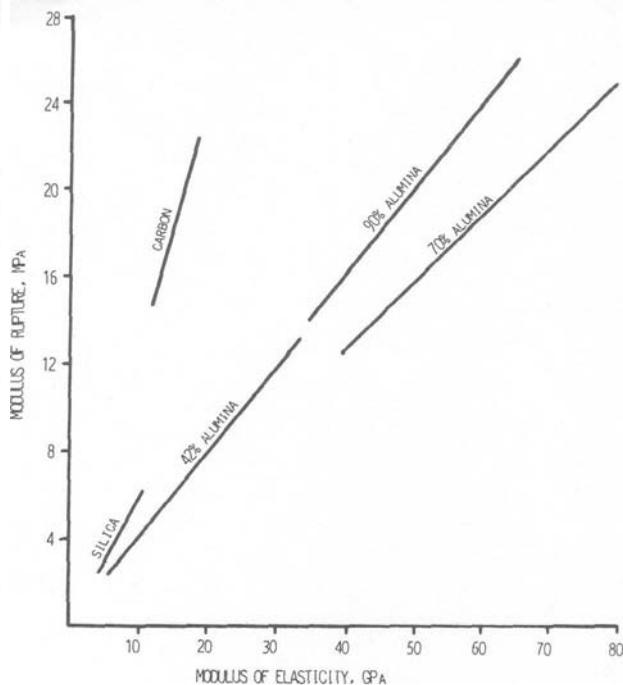


Fig. 2. Plot of modulus of rupture vs modulus of elasticity for all model refractory products tested. Slope of each line gives K constant for product (for range tested), which can be used to calculate modulus of rupture if the modulus of elasticity is known.

refractory, but apparently there was a difference in the physical character of the products, as shown by the 23% difference in calculated and actual strength. The silica refractories compared had generally similar silica content (99% vs 96%), but the 99% product was a slip-cast vitreous silica refractory and the 96% product was a conventional lime-bonded silica brick; the result was a roughly 30% difference in the calculated and actual strength. The two carbon brick products compared were different, probably in both

Table IV. Prediction of MOR Using Experimentally Derived Relations

Refractory type	Ultrasonic velocity (m/s)	MOR predicted from ultrasonic velocity (MPa)	MOE (GPa)	MOR predicted from MOE (MPa)	Actual MOR (MPa)
High alumina (90%)	4040	19.60	47.10	19.24	19.12
Mullite (70% Al_2O_3)	3901	13.13			12.29
Fireclay (42% Al_2O_3):					
Super duty 1	3370	9.00	21.50	9.42	9.37
Super duty 2			19.80	7.91	8.12
High duty	3485	9.43			7.67
Silica brick (96%)	2056	4.29	7.20	4.13	6.13
Carbon	2503	14.45			8.20

Table V. Comparison of Predicted and Actual Strength Data Shown in Table IV

Refractory type	Basis of comparison	Number of samples	Predicted vs actual MOR (% difference)
High alumina (90%)	Ultrasonic-exponential	10	+2.5
	MOE-linear	10	+0.6
Mullite (70% Al_2O_3)	Ultrasonic-exponential	15	+6.8
Fireclay (42-45% Al_2O_3):	Ultrasonic-exponential	10	-3.9
	MOE-linear	10	+0.5
	MOE-linear	5	-2.6
	Ultrasonic-exponential	25	+22.9
Silica brick (96%)	Ultrasonic-exponential	8	-30.0
	MOE-linear	8	-32.6
Carbon	Ultrasonic-exponential	19	+76.2



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the major raw materials used and the bond, although the specific characteristics weren't revealed by the suppliers or the product data sheets.

The derivation of a meaningful mathematical relationship clearly must involve the analysis of an appropriate number of samples representative of the complete property range typical of any given brand or product type; the broader the property range evaluated, the more broadly applicable the relationship should be. Various references, for example, Military Standard 414 and International Standard 5022 are available to guide the determination of the number of test samples that constitutes a representative population. For evaluating only one refractory brand on an ongoing basis, it should be possible to establish a very accurate mathematical relationship whereby sonic measurements could be used to very accurately predict the strength nondestructively, as long as there are no changes in the chemical or physical makeup of the product. It is felt that the mathematical relationships developed in this work are reasonably valid, and certainly representative of the potential for the method, based upon the observed strength correlations of less than 1%, even though only a limited range of samples were available for study. In a production or plant setting, obviously it would be possible to establish a broader and more rigorous mathematical relationship because of the ongoing availability of a broader, more complete range of samples for any given refractory brand or product type.

The limited comparative data generated herein, seemingly suggest that the modulus of elasticity measurements permit slightly better strength correlation than the ultrasonic velocity measurements. This observation is generally supported by previous thermal shock studies on a wide range of refractory products in this lab, in which the modulus of elasticity technique has been found to provide a more definitive evaluation of the induced crack damage than ultrasonic measurements. However the ultrasonic technique is better than MOE for pinpointing the presence and location of internal defects and low density regions in commercial refractory products.

This work has confirmed that sonic measurements can be used to generate valid strength estimates for a variety of refractory products based upon a valid mathematical relationship and an appropriate comparison of materials. In the case of the evaluation

of one brand on an ongoing basis, the comparison should be very straightforward and easy, until there is some significant change in the raw materials or the production history for the product. With any change in the raw materials or the production history, a new mathematical relationship would have to be established. The comparison of multiple brands or similar refractory types appears more limited, requiring that the chemistry and structure be very similar if good correlation is to be expected, although it does appear feasible that this broader comparison is possible. Further work is needed to better document the feasibility of determining and using a more broadly applicable mathematical relationship, based upon nondestructive sonic measurements, for estimating the strength of competitive brands of the same refractory type, such as, slag resistant superduty fireclay, low alkali 70% alumina, bonded alumina-zircon-silica, fired, high purity magnesite, and 60% magnesite-chrome (low silica), to name a few.

Summary

Five fired refractory product types were evaluated using two nondestructive sonic techniques, i.e., ultrasonic velocity and modulus of elasticity. The sonic data were correlated with modulus of rupture data to determine the best mathematical relationship for each of the refractory types, from which the modulus of rupture could thereafter be predicted (calculated) for the same product or an equivalent product having similar chemistry and structure. The mathematical relationships which showed the best correlation between sonic measurements and strength were exponential for ultrasonic velocity and linear for modulus of elasticity. The relationships derived were tested for several additional fired refractory brands, to evaluate their general validity in predicting strength. Seemingly in most cases where the chemistry and structure of the refractory being evaluated are similar to a refractory product for which a valid mathematical relationship has been developed, good correlation between the predicted and actual strength can be expected, with the values differing by less than roughly 5%. In several of the example cases, the predicted and actual strengths differed by less than 1%. For products with nominally similar chemistry, but different makeup and/or structure, the predicted strengths differed widely from the actual strengths. For a given fired refractory product, as well as other very similar products, an experimental mathematical relationship, based on a rigorous, statistically valid analysis, can be determined, which should be valid until some change is made in the composition, particle sizing, or production history. The scope or range of applicability of the mathematical relationship(s) can be broadened by using the widest range of values applicable to any product type(s) of interest.

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