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The Influence of Nonlinear Elasticity on the Accuracy of Thermal Shock Damage Evaluation by the Impulse Excitation Technique

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ABSTRACT

This work quantifies the influence of nonlinear elasticity on the accuracy of the thermal shock damage evaluation of high-alumina refractory castables based on dynamic Young's modulus and damping characterization data obtained via the impulse excitation technique (IET).The nonlinear elasticity leads to shifts in the Young's modulus and damping values in dependence of the impulse intensity.
An IET apparatus was employed according to the ASTM E1876-07 standard; the method was improved by nonlinear analysis based on the excitation control. Two arbitrary coefficients were defined to evaluate the nonlinearity with respect to the impulse intensity. Three high-alumina refractory castables were submitted to progressive thermal shock damage and evaluated. The results show that the influence of nonlinear elasticity on the Young's modulus is significant and that nonlinear elasticity is a determining factor for the accuracy of the damping characterization (up to -0.93 % and +114 %, respectively, depending on the damage level).

KEYWORDS

thermal shock, impulse excitation technique, nonlinear elasticity, Young's modulus, damping
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1 Introduction

The impulse excitation technique (IET) is a standardized [1] and widespread method that submits the specimen under test to minute strains for the nondestructive evaluation of dynamic elastic moduli at room, cryogenic and high temperatures. The IET is more practical and accurate than static techniques [2], particularly for brittle materials with rough microstructures, such as refractory materials. In addition, the IET is easily applicable to a broad range of sample geometries and dimensions for research and industrial quality-control purposes.

The latest generation of IET systems – such as the Sonelastic® Solutions (ATCP Physical Engineering, Brazil), Buzz-o-sonic (Buzz-Mac, USA) and RFDA (IMCE, Belgium) – take advantage of the advances in personal computers to incorporate simultaneous damping measurements using the logarithmic-decay and bandwidth methods [3]. This advancement has allowed researchers who work with ceramic materials to use the sensitivity of damping to enhance the evaluation of thermal shock damage and to detect events not easily detectable when only the elastic moduli are monitored [4]. However, damping characterizations performed using the IET are not standardized or as well established as the elastic moduli. Furthermore, reports of the occurrence of nonlinear elasticity in rocks and concrete have appeared in the literature [5–6]. Rocks and concrete have microstructures similar to those of refractory castables; nonlinear elasticity has already been observed in the damping characterization of refractory castables, as we have briefly reported elsewhere [7].

The manifestation of nonlinear elasticity comprises, among other indicators, shifts in the dynamic elastic moduli and in the damping in dependence of the specimen's vibration amplitude [8]; the vibration amplitude, in turn, depends on the impulse excitation intensity applied by the IET apparatus. These shifts can lead to errors related to the impulse intensity randomness, particularly when manual excitation is applied and

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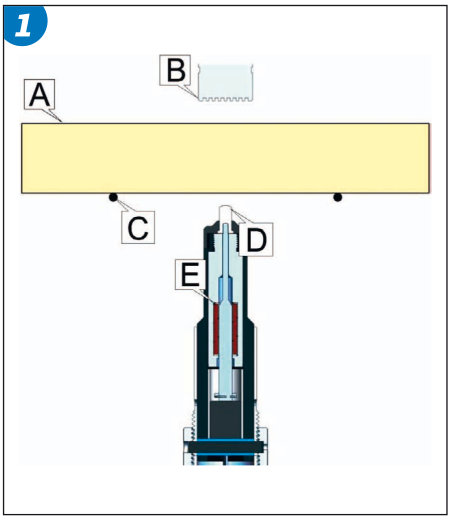

Fig. 1 • Cross section of the apparatus's main parts arranged to excite at the specimen's flexural mode; A: Specimen, B: Microphone, C: Wire, D: Plunger tip. E: Electromagnetic coil

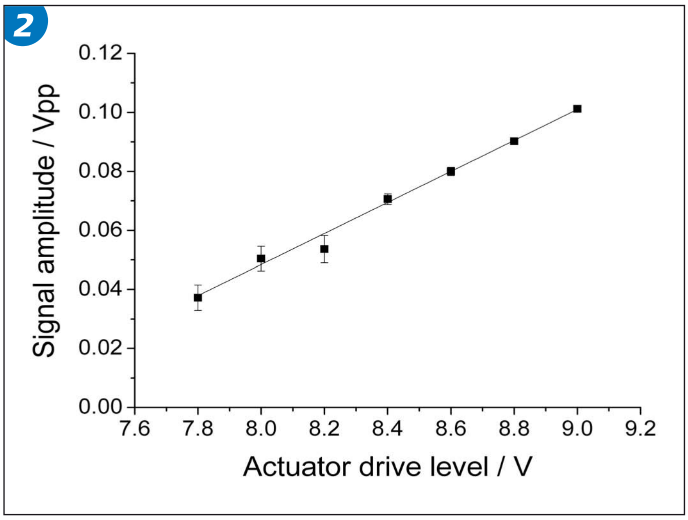

Fig. 2 • Acoustic response peak-to-peak amplitude of a dense alumina ceramic as a function of the electromagnetic impulser driving voltage

different instruments are used. These facts raise concerns about the reliability of the IET when applied to materials with the potential to exhibit nonlinear elasticity; the list of such materials includes refractory materials in general.

Nonlinear elasticity can be classified as atomic or structural, depending on the major nonlinear component. When the structural nonlinear component is involved, it exhibits a significantly stronger effect than the atomic component and is associated with the presence of grains or aggregates that have been consolidated by a mesoscopic bond system which contains cracks, frictional contacts and microstructural defects [8]. These defects act as on/off switch elements that exhibit hysteretic behaviour, and the state of these defects depends on the instantaneous and past stress and strain imposed on the material [9–10]. The presence of these switching elements results in the material becoming softer and dissipating more energy when the amplitude of vibration increases. Refractory materials exhibit the typical microstructural characteristics for the occurrence of structural nonlinear elasticity phenomena, especially when damaged.

The published standards and related literature [2] contain no discussions about the influence of the impulse excitation intensity as a relevant parameter for the characterization accuracy of materials due to potential interference from nonlinear effects.

The objective of this work was to investigate the influence of the impulse excitation intensity on the accuracy of the IET results with respect to the dynamic Young's modulus and damping characterizations of three high-alumina refractory materials that were progressively damaged by thermal shock (water quenching). This work also aimed to quantify this nonlinearity so that this phenomenon can be used in the future to advance the understanding and monitoring of thermal shock damage. The characterization of nonlinear elastic behaviour is already used to evaluate damaged concrete [6].

Regarding the materials used here, two of the investigated refractory castables were prepared with electrofused alumina aggregates with a maximum size of 2 or 8 mm. The third castable was prepared with tabular alumina aggregates with a maximum size of 3 mm.

2 Experimental

2.1 The impulse excitation technique

In this technique, a specimen subjected to proper mechanical boundary conditions in accordance with the expected mode of vibration is excited by a short and light mechanical impulse. The acoustic response is sensed by a microphone and processed according to the frequency and attenuation rate detection. For bars with square cross-sections excited at the flexural mode of vibration, the Young's modulus (Y) is calculated using the Piquet equation [1, 11]:

$$Y = 0.94642 \frac{\rho l^4}{t^2} f^2 T \qquad (1)$$

The parameter f is the flexural frequency, t is the specimen thickness perpendicular to the vibration direction, l is the specimen length, p is the density and T is a geometrical correction factor that depends on the aspect ratio of the specimen and the Poisson's ratio. The shear modulus (G) is calculated in a similar manner from the torsional frequency. The Poisson's ratio (v) is calculated by the relation $v = [(Y / 2G) - 1]$ with an interactive algorithm, which also improves the Y and G precision. The damping (ζ) is commonly calculated using the logarithmic decrement method [3]:

$$\zeta = \frac{\delta}{\omega} \qquad (2)$$

The parameter δ is the angular coefficient of the specimen's acoustic response attenuation on a logarithmic scale, and ω is the angular frequency. The bandwidth method is also applied to high-damping materials. Advanced signal processing is applied to permit proper signal conditioning and curve fitting.

2.2 IET apparatus with controlled impulse

The Sonelastic® PC-based apparatus (ATCP Physical Engineering, Brazil) was employed with voltage-controlled impulse excitation.

The control of the impulse is performed by changing the amplitude of a square voltage pulse applied by the apparatus's electronics to drive an electromagnetic impulser. The vertical cross section of the impulse device's main parts is shown in Fig. 1.

The electromagnetic impulser is located 5 mm from the specimen surface. The useful range of the adjustable drive voltage is from 7.8 to 9.2 V. A voltage less than 7.8 V is insufficient to induce the impulser action, and a voltage greater than 9.2 V causes double hitting. Figure 2 presents the microphone signal peak-to-peak amplitude that corresponds to the acoustic response of a dense alumina ceramic bar as a function of the electromagnetic impulser drive voltage [7]. Five acquisitions were performed at each voltage. The error bars correspond to the standard deviation, and the line corresponds to a polynomial first-order fitting.

If the dense alumina specimen and the microphone are linear, a direct correlation exists between the impulse intensity and the electromagnetic impulser drive voltage, although the absolute amplitude of the vibration is undetermined.

2.3 Nonlinear coefficients

Two arbitrary coefficients were defined to quantify the influence of the nonlinear elasticity on the accuracy of the IET results. The α-coefficient describes the ratio between the percentage shift in the Young's modulus and the change in the impulser driving voltage:

$$\alpha = \frac{\Delta Y}{\Delta V} \quad (3)$$

The β-coefficient serves the same purpose with respect to the damping shift:

$$\beta = \frac{\Delta \zeta}{\Delta V} \quad (4)$$

where ΔY is the percentage change in the Young's modulus, Δξ is the percentage change in the damping ξ and ΔV is the change in the impulser driving voltage. The nonlinear coefficients are determined after a curve fitting over five measurements performed along the useful range of the impulser driving voltage (7.8–9.2 V) and equally spaced at 0.25 V. The reference values for the calculations are the values that correspond to the lowest driving level.

2.4 Materials and thermal shocks

Three high-alumina castables were studied. Two of the castables were prepared with white electrofused aggregates (from Rio Tinto Alcan, Elfusa, Almatis and Alcoa) with a maximum size of 2 mm (named A2) and 8 mm (named A8); both of these castables obey the Andreasen packing model with a distribution coefficient of 0.26. A third castable was prepared with tabular alumina aggregates (Rio Tinto Alcan) with a maximum size of 3 mm (named AT) that obeys the Andreasen packing model with a coefficient of 0.245. The cement of calcium aluminates, CA-Cement (Secar 71, Kerneos), and aggregate content of the materials is shown in Table 1. Citric acid and deflocculant (FS 40, BASF) were used as additives. Twenty-four prismatic bars of each material were prepared with dimensions of 25×25×150 mm.

After the A2 and A8 specimens were moulded, cured for 48 h in a humid environment and dried at 120 °C for 24 h, the specimens were sintered at 1,450 °C for 10 h at a heating rate of 3 °C/min. The AT was sintered at 1,500 °C for 6 h at a heating rate of 2 °C/min. After being fired, the specimens' top surface was ground to improve the parallelism and the accuracy of the elastic moduli characterization.

Thermal shocks were applied using water quenching. The specimen was first heated from room temperature to the desired temperature in a muffle furnace at a rate of 3 °C/min. After a dwell time of 15 min, the specimen was abruptly moved from the muffle furnace into circulating water at room temperature. The specimens of each material were divided into eight groups of three specimens, each named from A to H. Each group of three specimens was subjected to one thermal shock cycle at the temperature variation ΔT specified in Table 2, except for group A, which was chosen as the reference group.

Figure 3 shows the microstructure of the AT castable before (a) and after severe thermal shock damage (b and c). The aggregates consolidated by a mesoscopic bond system that contains cracks, frictional contacts and other defects, constitute the typical microstructural characteristics for the occurrence of structural nonlinear elasticity phenomena.

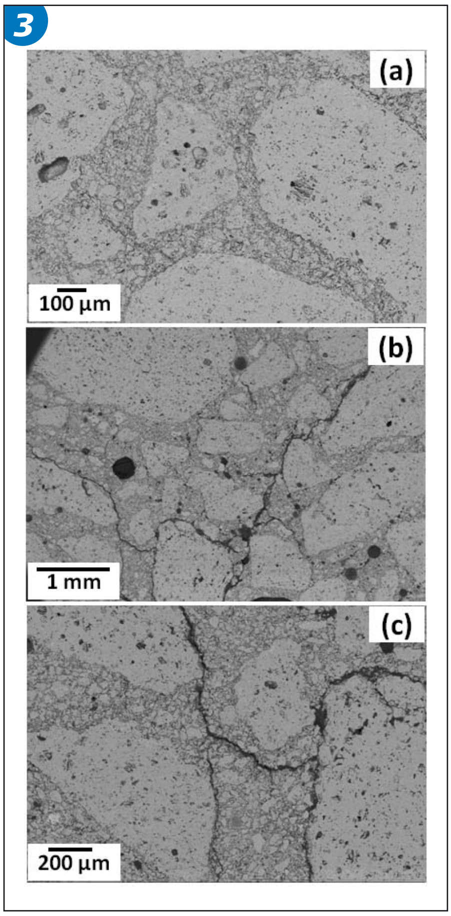

Fig. 3 • AT castable microstructure after sintering (a) and after severe thermal shock damage (b) and (c). The aggregates consolidated by a mesoscopic bond system that contains cracks, frictional contacts and other defects

3 Results and discussion

Figures 4 and 5 show the retained Young's modulus values and the damping changes as a function of the thermal shock temperature variation for each material group. These results were obtained with the lowest impulse excitation amplitude for the IET apparatus (7.8 V). The reference values are shown in Table 3 and correspond to the 0 °C temperature variation point in the graphs. The error bars in these graphs and in the following graphs correspond to the standard deviation, whereas the symbols correspond to average values; both the standard deviations and average values were calculated over three specimens.

An almost linear decrease to values of 50–60 % is observed for the Young's moduli obtained for wide temperature variations (Fig. 4). The percentage decrease is higher for the A8 material, followed by that for A2 and AT. The damage at the interfaces between large aggregates and the castable matrix explains the decrease of the Young's modulus (Fig. 3b, c for the AT material). The absence of a critical temperature variation for A2 and A8 indicates the occurrence of damage during cooling after the firing process, which does not appear to occur for the AT material. This hypothesis is corroborated by the higher Young's modulus and lower initial damping values of the AT material (Table 3).

Table 2 • Temperature variations (DT) applied to the specimen groups

Group	DT / °C
A	0
B	100
C	250
D	400
E	500
F	600
G	700
H	800

Table 1 • Cement and aggregate content of the refractory materials used in this work

	A2	A8	AT
Maximum aggregate size / mm	2.3	8	3
Aggregate content / mass-%	58	67	52.5
CA-Cement / mass-%	4	4	5

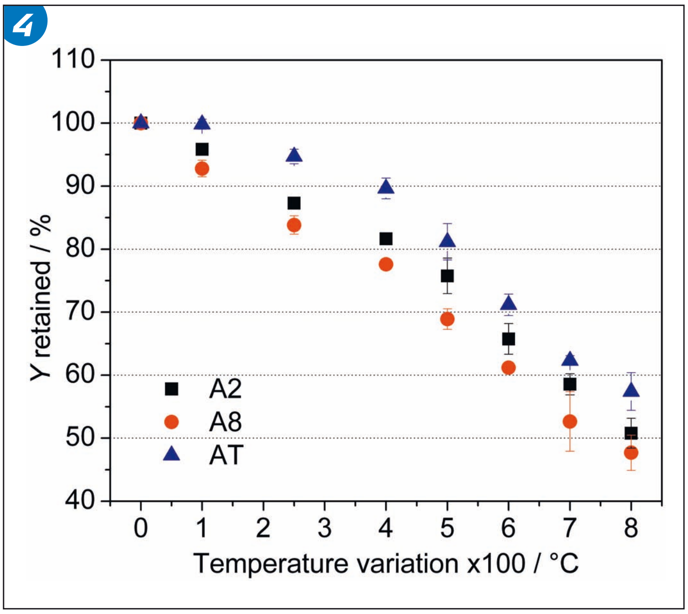

Fig. 4 • Retained Young's modulus (Y) vs. the thermal shock temperature variation (ΔT)

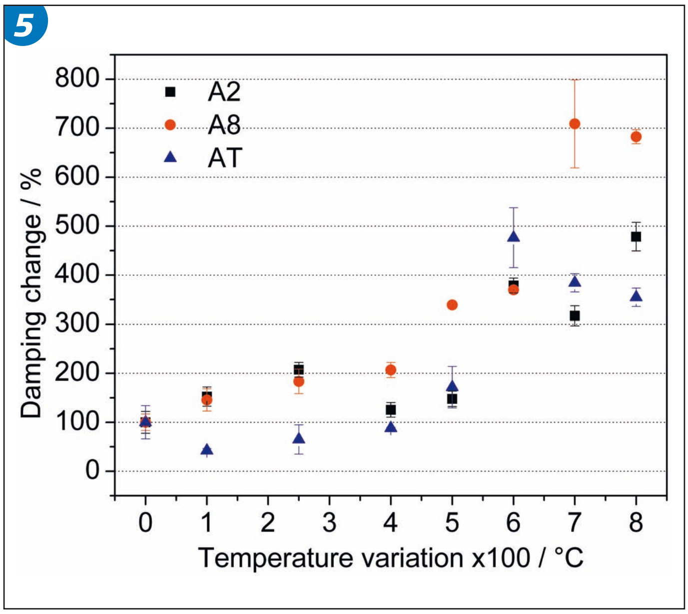

Fig. 5 • Relative damping change vs. the thermal shock temperature variation (ΔT)

As expected, the damping changes follow the inverse tendency of the retained Young's modulus and increase with the formation of cracks and microcracks. The A8 material shows the greatest damping changes, followed by A2 and AT. The percentage difference between the changes in damping after damage is much greater than the difference between the retained Young's modulus. The increase in the AT damping values between 600 and 700 °C may be explained by the formation of the calcium aluminate phase from the cement, which did not react during the sintering process.

The arbitrary nonlinear coefficients that were defined to quantify the influence of the nonlinear elasticity on the accuracy of the IET results were evaluated for each material and group of specimens after damage in the same manner as the Young's modulus and damping values. The α-coefficient and β-coefficient results are shown in Figs. 6 and 7, respectively. These results allow an estimation of the extent to which the Young's modulus and the damping could be changed only by variations in the impulse intensity.

The α-coefficient is always negative, and its absolute value increases with the damage as the cracks propagate, increasing the material's nonlinear elasticity. The increasing rate of the absolute value depends on the magnitude of the temperature change and is higher for temperature changes greater than 400 °C. For the reference group, the α-coefficient is -0.01 %/V for AT, -0.03 %/V for A2 and -0.09 %/V for A8. After the damage, the maximum absolute value was -0.93%/V for A2. In this case, where the impulser amplitude range was 2.4 V, the maximum uncertainty in the retained Young's modulus shown in Fig. 7 associated with the impulse excitation would be approximately 2 % at a temperature variation of 800 °C. This 2 % uncertainty may be not sufficient to compromise the analysis of the results; however, it is relevant and of the same magnitude as the typical uncertainty in IET associated with ceramic specimens characterization [2]. The β-coefficient is always positive and increases with the damage as the cracks propagate, increasing the material's nonlinear elasticity. For the reference group, the β-coefficient is approximately 3.8 %/V for AT, 5.8 %/V for A2 and 21.4 %/V for A8. The damage causes the β-coefficient to reach values as high as 113.9 %/V for the A8 material after the 700 °C thermal shock. Therefore, nonlinear elasticity is a determining factor for the characterization of the damping of the studied materials by IET and may compromise the results of the analysis of thermal shock damage using this technique.

Fig. 6 • The a-coefficient vs. the thermal shock temperature variation (ΔT) for materials A2, A8 and AT

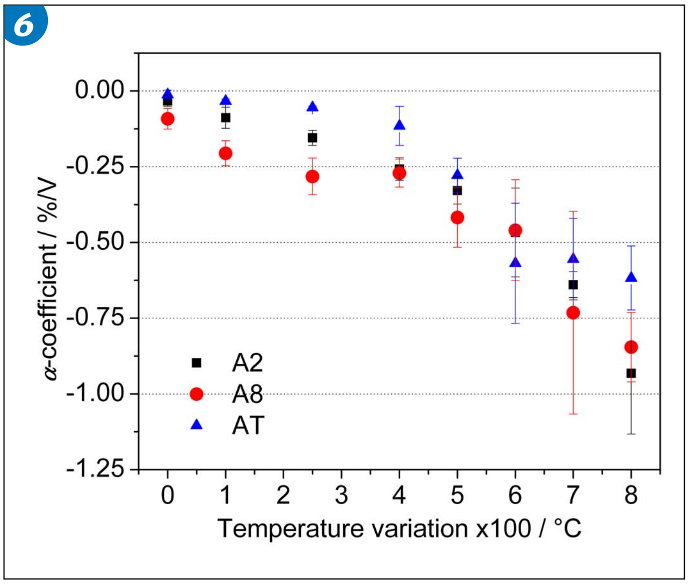

Table 3 • Reference values

Material	A2	A8	AT
Y / GPa	120.4	112.5	126.8
Damping x	0.0014	0.0015	0.0007

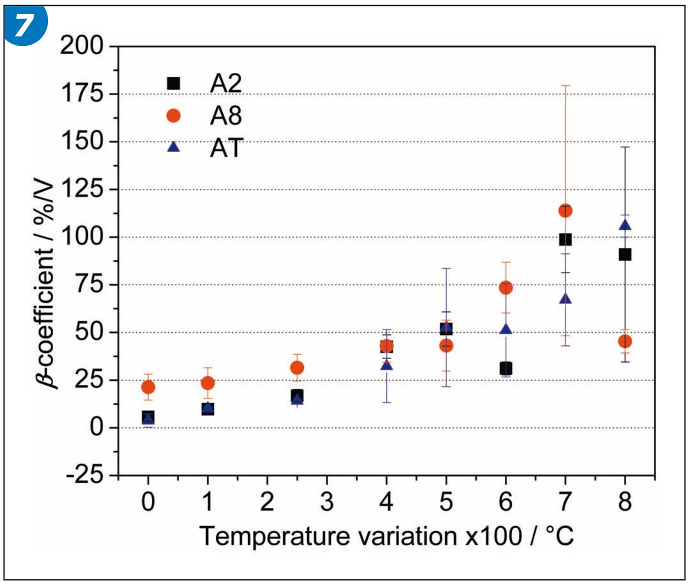

Fig. 7 • The b-coefficient vs. the thermal shock temperature variation (ΔT) for materials A2, A8 and AT

The thermal stress, the anisotropy of the aggregates and the expansion mismatch between the aggregates and the matrix cause the formation of cracks in the materials studied. These cracks appear to be primarily responsible for the phenomenon of nonlinear elasticity intensification due to the nonlinear hysteretic behaviour of the crack walls. The relative movement of the crack walls depends on the difference between the walls' static and dynamic coefficients of friction and on the stress and strain history of the material; the crack walls act like hysteretic on–off switches and lead to the material's nonlinear elasticity behaviour [10].

4 Conclusions

The nonlinear elasticity phenomenon exhibited by refractory materials should be taken into account in the design of IET experimental procedures and in the development of related standards. Characterizations and results analyses based on damping measurements via IET should be conducted carefully because of the one-order-of-magnitude uncertainty associated with the impulse excitation intensity.

The nonlinear elasticity phenomenon correlation with the formation of cracks and microcracks can be used in the future to advance the understanding and monitoring of thermal shock damage.

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References

[1] ASTM 1876-07: Standard Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio by Impulse Excitation of Vibration. ASTM International (2007), 15 pp

[2] **Morrel, R.:** NPL Measurement Good Practice Guide – Elastic Module easurement. UK National Physical Laboratory Report no. 98 (2006), 100 pp

[3] **Silva, C.W.:** Vibration Damping, Control, and Design, 1st edition. Taylor & Francis, Vancouver (2007), 16–25. ISBN 978-1-4200-5321-0

[4] **Tonnesen, T., Telle, R.:** Thermal shock damage in castables: Microstructural changes and evaluation by a damping method. Ceramic Forum International **84** (2007) [9] E132–136

[5] **Johnson, P.A., Zinszner, B., Rasolofosaon, N.J.:** Resonance and elastic nonlinear phenomena in rock. Journal of Geophysical research **101** (1996) [B5] 11553–11564

[6] **Abeele, K.V.D., Visscherb, J.:** Damage assessment in reinforced concrete using spectral and temporal nonlinear vibration techniques. Cement and Concrete Research **30** (2000) 1453–1464

[7] **Pereira, A.H.A., Nascimento, A.R.C., Rodrigues, J.A.:** Effect of non-linearity on young's modulus and damping characterisation of high-alumina refractory castables through the impulse excitation technique. Proc. 53rd International Colloquium on Refractories Aachen (Germany) 2010, 90–93, available on CD-ROM.

[8] **Johnson, P.A., Ostrovsky, L.A.:** Dynamic nonlinear elasticity in geomaterials. Rivista del Nuovo Cimento **24** (2001) [7] 1–45

[9] **Guyer, R.A., McCall, K.R., Boitnott, G.N., Hilbert Jr. L.B., Plona, T.J.:** Quantitative implementation of Preisach-Mayergoyz space to find static and dynamic elastic moduli in rock. J. of Geophysical Research **103** (1997) [B3] 5281–5293

[10] **Mayergoyz, J.D.:** Hysteresis models from the mathematical and control theory points of view. J. Appl. Phys. **57** (1985) 3803

[11] **Pickett, G.:** Equations for Computing Elastic Constants from Flexural and Torsional Resonant Frequencies of Vibration of Prisms and Cylinders. Proceedings of Am. Soc. Testing Mats. **45** (1945) 846–865

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