The Impulse Excitation technique, an innovative NDT method for microstructure and mechanical properties characterization of refractory materials used in the aluminum industry.

Bart Bollen
General Manager, IMCE NV, Belgium

16. February 2016, AMAP colloquium, Aachen

IMCE

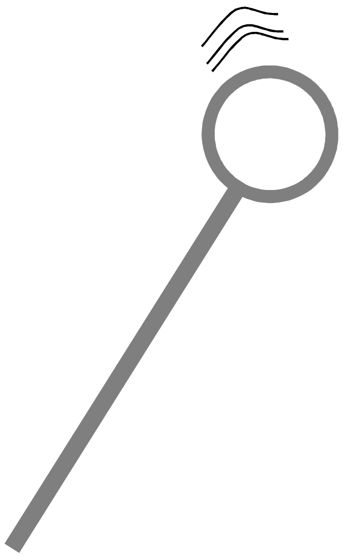
- Since 1995 we focus on the development and production of non-destructive testing devices based on the Impulse Excitation Technique (IET)
- Integration of the IET-systems
  - » Measuring systems
    - Resonant Frequency and Damping Analyser (RFDA)
    - High temperature measurement set-ups
  - » Quality control systems
    - RFDA-Inspector

Outline

- IET: principle
- IET: applications
- IET: cases
- IET: conclusions

IET principle

An ancient technique!?



IET: principle

Dr. Förster established first modern IET principles

ZEITSCHRIFT FÜR METALLKUNDE

Herausgegeben von der Deutschen Gesellschaft für Metallkunde im Verein deutscher Ingenieure, Berlin NW7, Ingenieurhaus
Gegründet von W. Guertler, geleitet von W. Köster und H. Groeck. – V·D·I-Verlag GmbH, Berlin NW7, Dorotheenstr. 40

29. Jahrgang — April 1937 — Heft 4

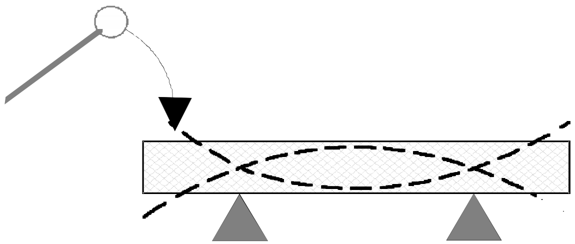
Ein neues Meßverfahren zur Bestimmung des Elastizitätsmoduls und der Dämpfung

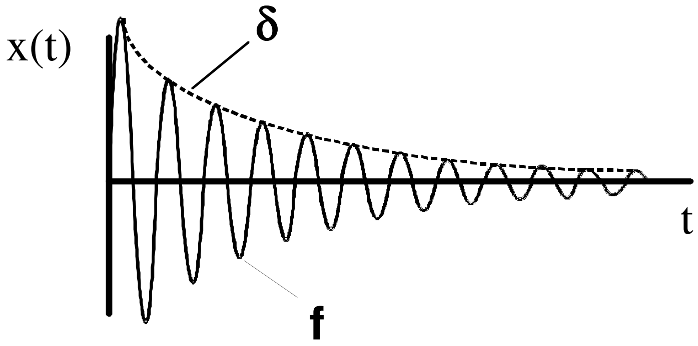
Von Fritz Förster in Stuttgart

(Aus dem Kaiser-Wilhelm-Institut für Metallforschung in Stuttgart.)

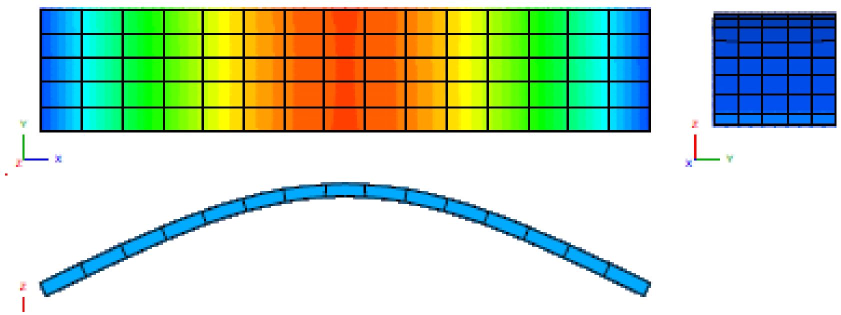
Objektive Bestimmung der Eigenschwingungszahl und der Dämpfung beliebig geformter Prüfkörper — Messung der Dämpfung aus der Resonanzhalbwertsbreite oder Abklingdauer — Einfluß der Aufhängung des Prüfkörpers auf die Dämpfung — Anordnung der Aufhängevorrichtung zur Vermeidung direkter mechanischer und elektrischer Schwingungsübertragung — Messung über und unter Raumtemperatur — Feststellung der Lage von Fehlstellen im Prüfkörper

IET: principle
Measuring Principle

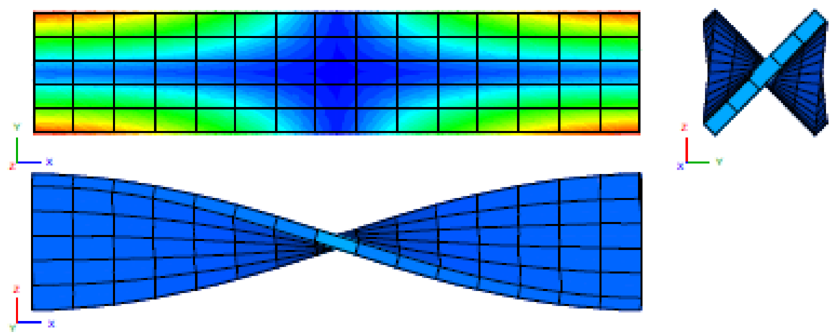




Commonly used vibration modes for a prismatic beam



Fundamental flexure mode



Fundamental torsion mode

IET: principle
Relation resonance frequency - stiffness

Transverse vibration of homogenious isotropic prismatic bars

Euler-Bernoulli Rayleigh Timoshenkko

$$E\, r_z^2 \frac{\partial^4 v}{\partial x^4} + \rho \frac{\partial^2 v}{\partial t^2} - r_z^2 \frac{\partial^4 v}{\partial x^2 \partial t^2} - r_z^2 \left(\frac{E}{K'G} \frac{\partial^4 v}{\partial x^2 \partial t^2} - \frac{\rho}{K'G} \frac{\partial^4 v}{\partial t^4} \right) = 0$$

Deformation energy
E = Young's modulus
r_z = radius of gyration

Energy of motion
ρ = density

Rotatory inertia

Shear deformation contribution
G = shear modulus
K' = cross-section shape factor

J.D. Lord, R. Morrell, A National Measurement Good Practice Guide, No. 98, Elastic Modulus Measurement, National Physical Laboratory, Teddington (2006) / A2.2 p. 79/92

IET: principle

Relation resonance frequency - stiffness

Theoretically there is no analytical solution to Timoshenko's differential equation

In the early 1960's Spinner and Teft proposed following approximate equation:

$$E = 0.9465\,\rho\, f_r^2 \left(\frac{L^4}{t^2}\right) T_1$$

E	= Young's modulus
f_r	= flexural frequency
ρ	= density
L	= length, t = thickness
T_1	= correction factor
	= f (t/L, Poisson's ratio ν)

This formula has been accepted as a standard:

ASTM C 1259 – ASTM E 1876 – ISO 12680

Slightly modified in EN 843-2 (2006)

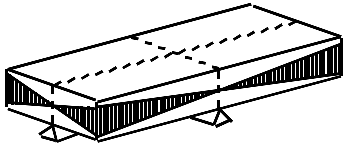
For refractory material: **ASTM C 1548-02**

IET: principle

Relation resonance frequency - stiffness

- For rectangular bars, cylinders in torsion mode (ASTM E 1876-15 , ASTM C 1548-02, ISO 12680-1, EN 843-2)

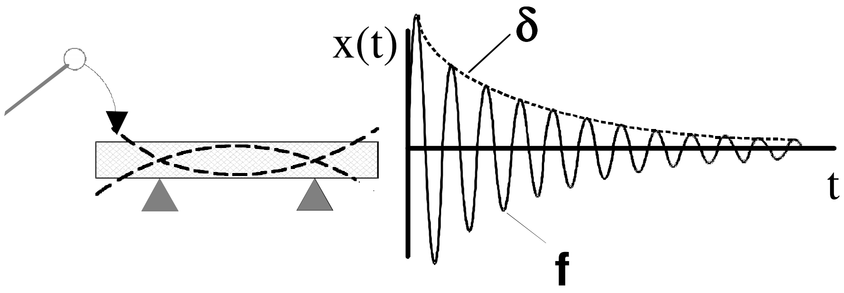
Torsion mode



$$G = \frac{4 L m f_t^2}{b t}\left[\frac{B}{(1 + A)}\right]$$

- For rectangular bars, cylinders in longitudinal mode (ASTM E 1876-15)
- For discs (ASTM E 1876-15)
- For grinding discs and pipes (described in literature)
- For coatings (ISO 20343)

IET: principle
Digital Signal Processing



$$x(t) = \sum A_i e^{-k_i t} \sin(\omega_i t + \phi_i)$$

f = 1/T = ω/2π : resonant frequency

k : exponential damping

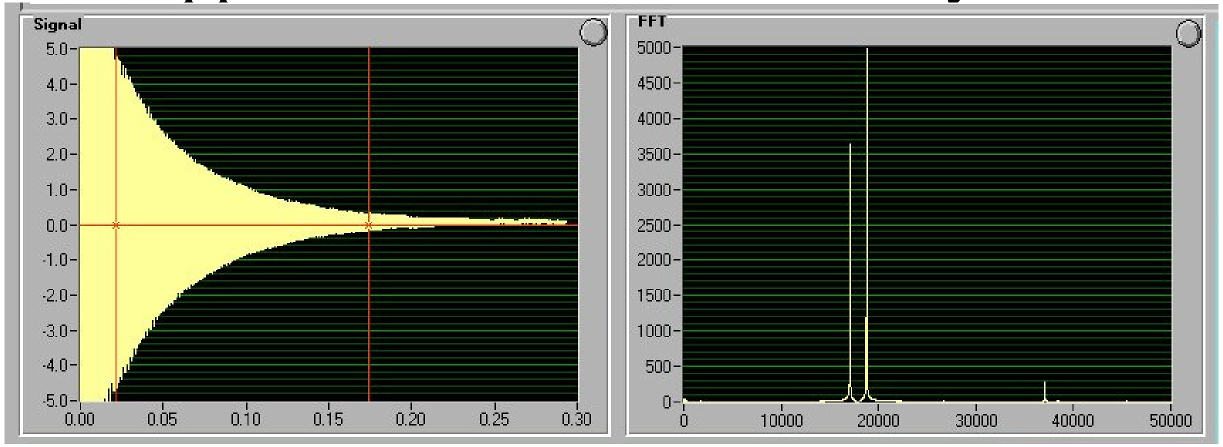
$$Q^{-1} = \frac{\Delta W}{2\pi W} = \frac{1}{\pi} \ln\left(\frac{x_1}{x_2}\right) = \frac{\delta}{\pi} = \frac{k_i}{\pi f_i}$$

Resonant Frequency and Damping Analyser (RFDA)

- Records ‘acoustic’ signal in time domain
- The software transforms signal to frequency domain and determines a first set of approximatively f_r by FFT
- Performs an iterative simulation of the time-domain signal as a sum of exponentially damped sinusoidal functions, to obtain for each selected f_r the corresponding value Q^{-1}.
- **→ Digital Signal Processing**

IET: principle
Simultaneous multiple frequencies and damping analysis

- Computer supported IET measurement systems



Signal in time domain

Signal in frequency domain

FFT →Resolution FFT: $\Delta f = \frac{f_s}{N}$

Example: vibration signal lasts 0.5 s => $\frac{44100}{22050} = 2$ Hz

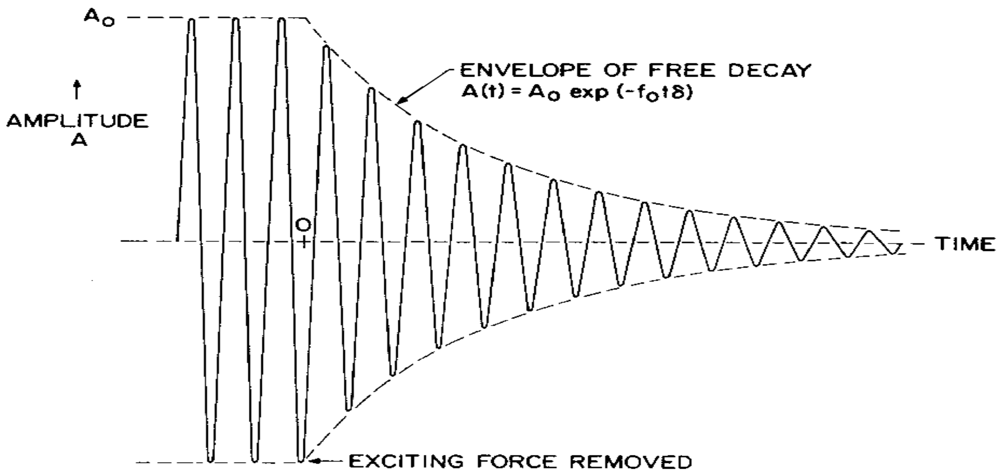
vibration signal lasts 0.1 s => $\frac{44100}{4410} = 10$ Hz

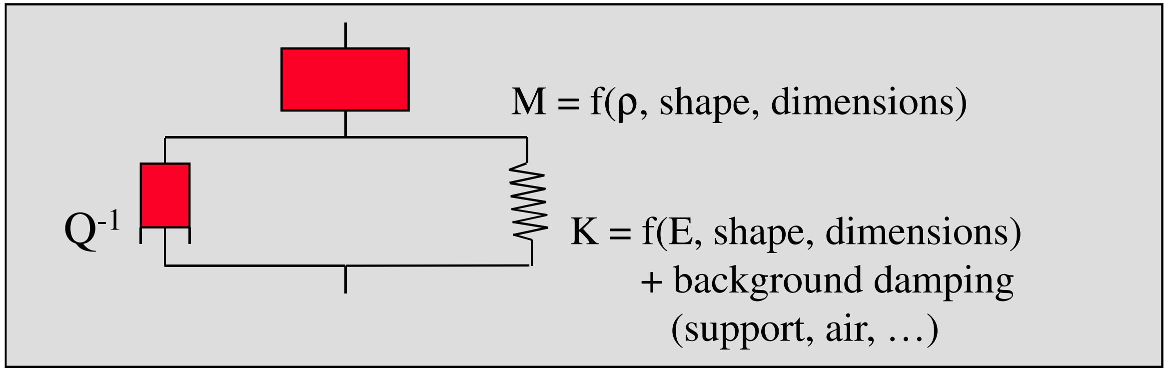
- Enhanced algorithms (RFDA)→ $\frac{\Delta f}{f} < 10^{-4}$

IET: principle
Internal friction measurement

Amplitude loss due to internal friction:

Damping or Internal friction (Q^{-1}) is energy dissipation in a material due to the movement of microstructural features.





IET: principle
Measuring results

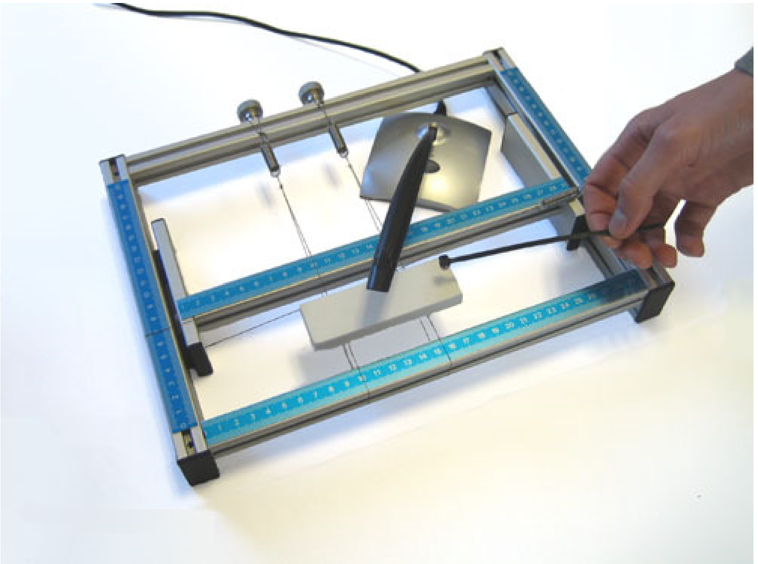
Resonant Frequency and Damping Analyser results

- Absolute information
  (Predefined shapes)
  - Elastic properties
    - Young's Modulus
    - Shear Modulus
    - Poisson's Ratio
  - Damping

- Relative information
  (Complex shapes)
  - Use the frequency spectrum and damping as a fingerprint of the part
    - Correlation analysis between frequency spectrum and physical properties

IET: principle

Room Temperature Measurement systems



RFDA Basic

RFDA Professional

- Manual excitation
- Frequencies up to 16 kHz
- USB microphone
- No DAQ-card
- No PC included

- Automatic excitation
- Frequencies up to 100 kHz
- Special IMCE microphone
- DAQ-card
- PC included

IET: principle
Summary

IET:

- Fast measurement
- Principally ‘simple’ measurement set-up
- Limited ristrictions on sample geometry and dimensions
- Non-destructive measurement of stiffness and damping properties
- Automatic digital signal processing

Suitable for repeated measurements as function of time, temperature or other actions on the material

IET: applications

High Temperature Measurement systems

RFDA HT650

- Up to 650 ºC (optionally to 1050 ºC)
- Heating rate 1-5 ºC/min
- Air atmosphere (optionally with gas flow)

RFDA HT1600
RFDA HT1750

- Up to 1600 ºC or 1750 ºC
- Heating rate 1-5 ºC/min
- Air atmosphere (optionally with gas flow)

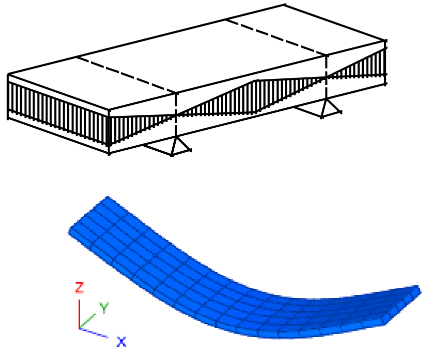
RFDA HTVP1600
RFDA HTVP1750C

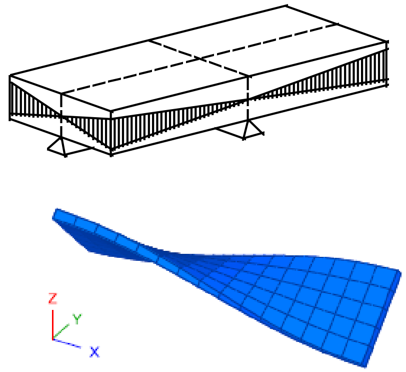
- Up to 1600 ºC or 1750 ºC
- Heating rate 1-5 ºC/min
- HTVP1600: air, inert, reducing
- HTVP1750C: inert or vacuum

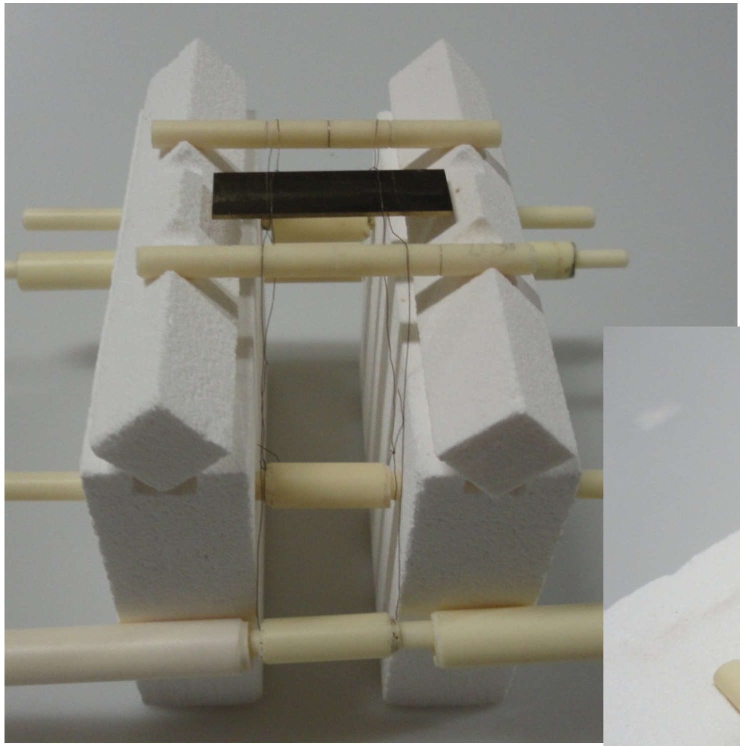
RFDA LTVP 800

- From -50 ºC to 800 ºC
- Heating rate 1-60 ºC/min
- Vacuum (10^{-5} mbar)

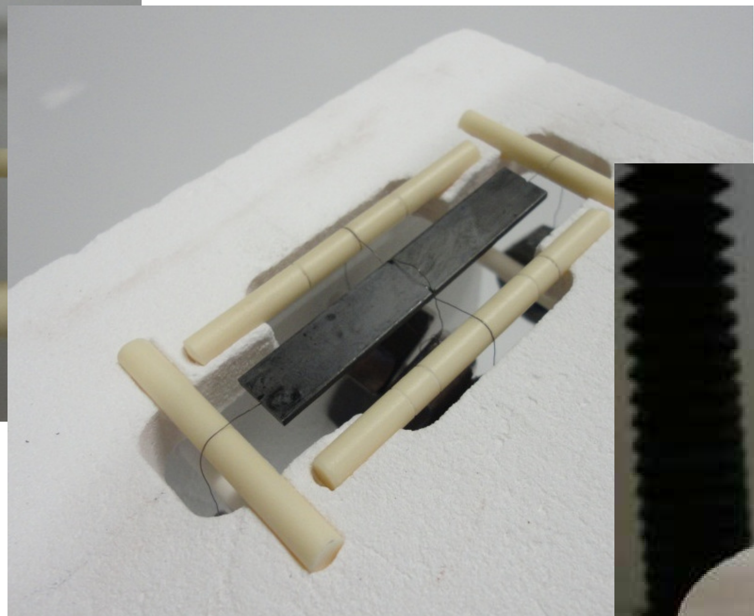
IET: applications
Excitation mechanism and sample support



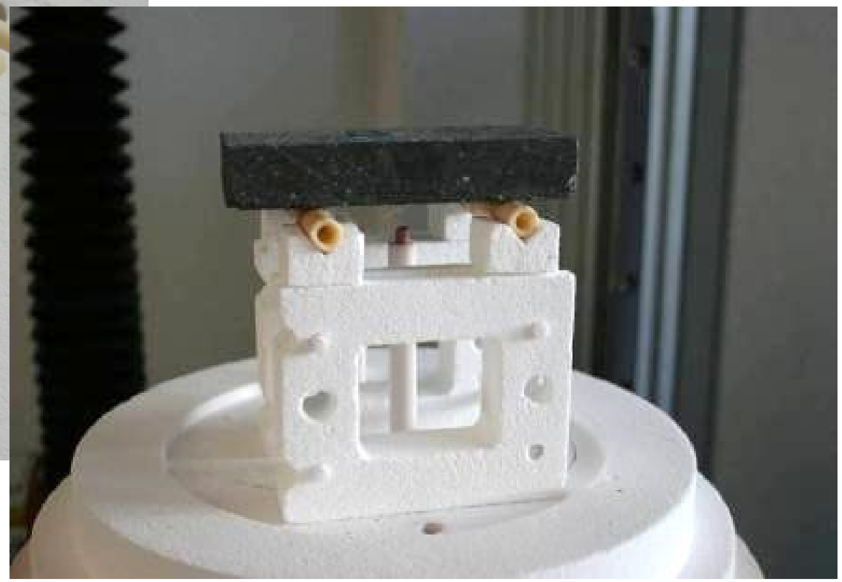




Small sample – flexural mode



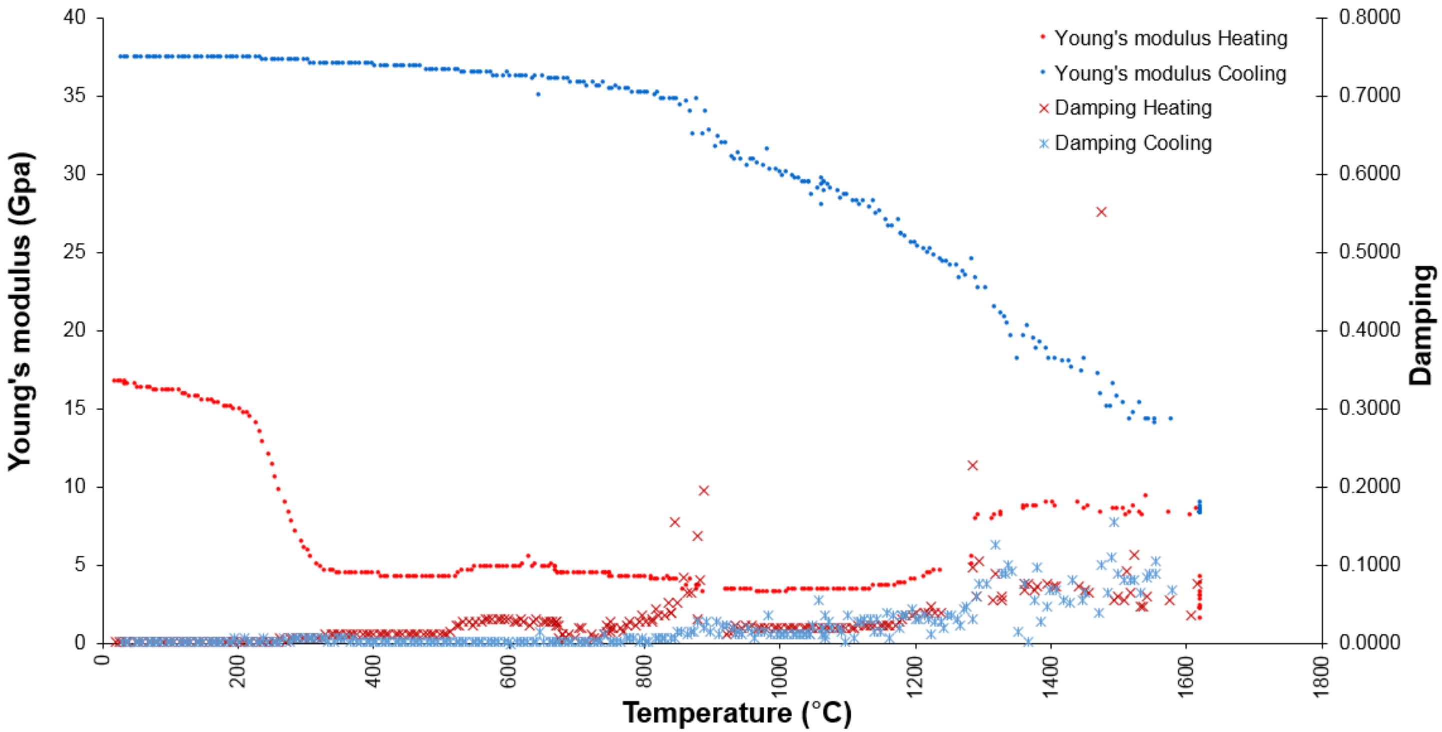
Small sample – torsion mode



Large sample – flexural and torsion mode

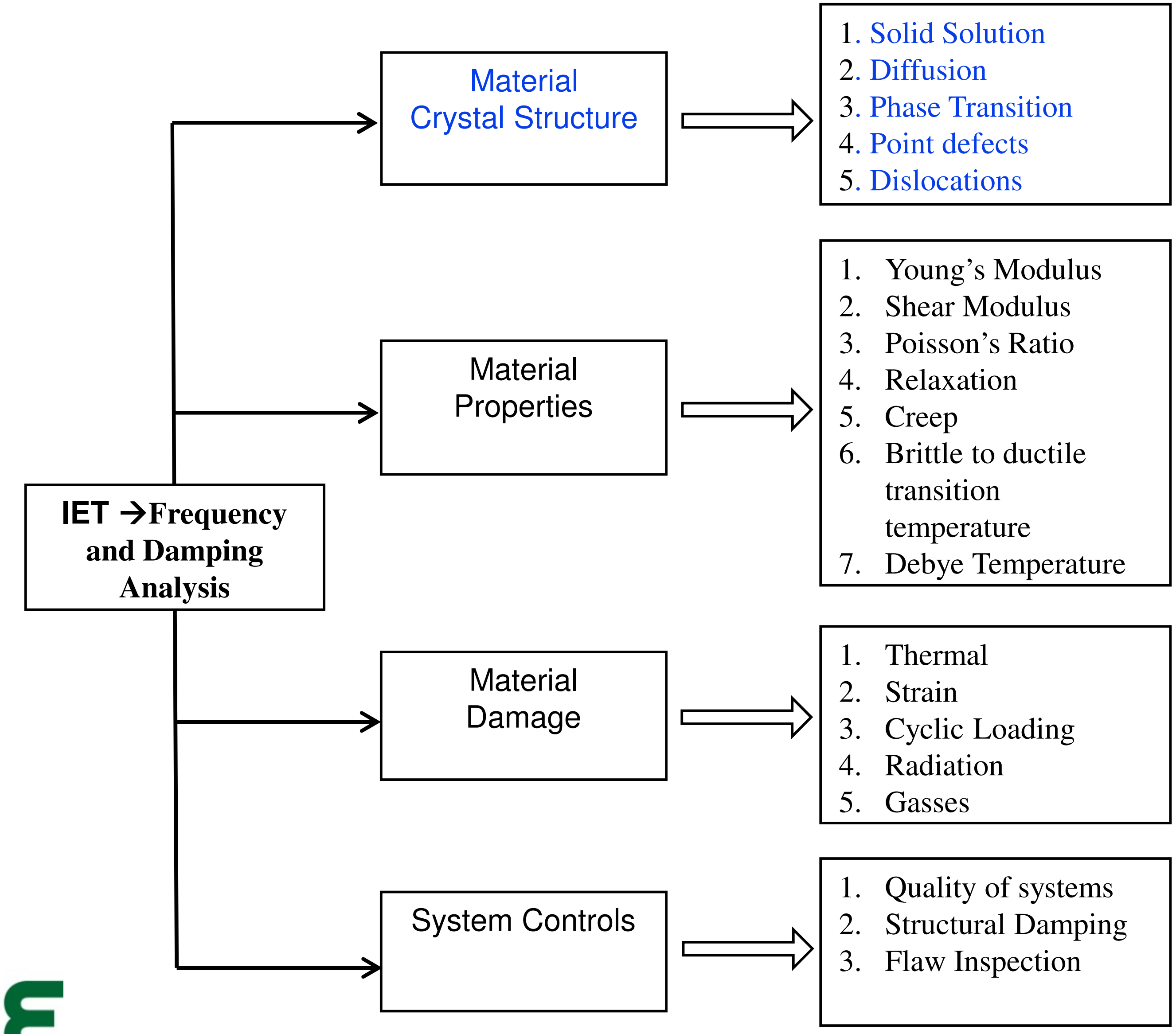
IET: applications
High Temperature Measurement system RFDA HT1600

Example: High alumina castable material



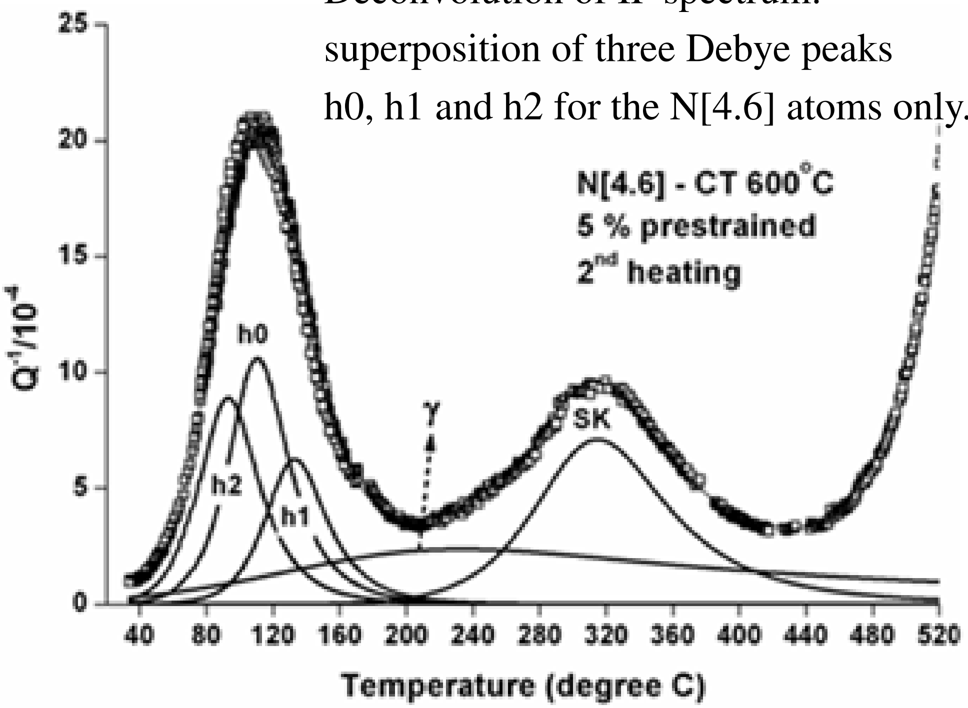
IET: Cases

Application domains and results



IET: Cases
Material crystal structure

Deconvolution of IF spectrum:
superposition of three Debye peaks
h0, h1 and h2 for the N[4.6] atoms only.

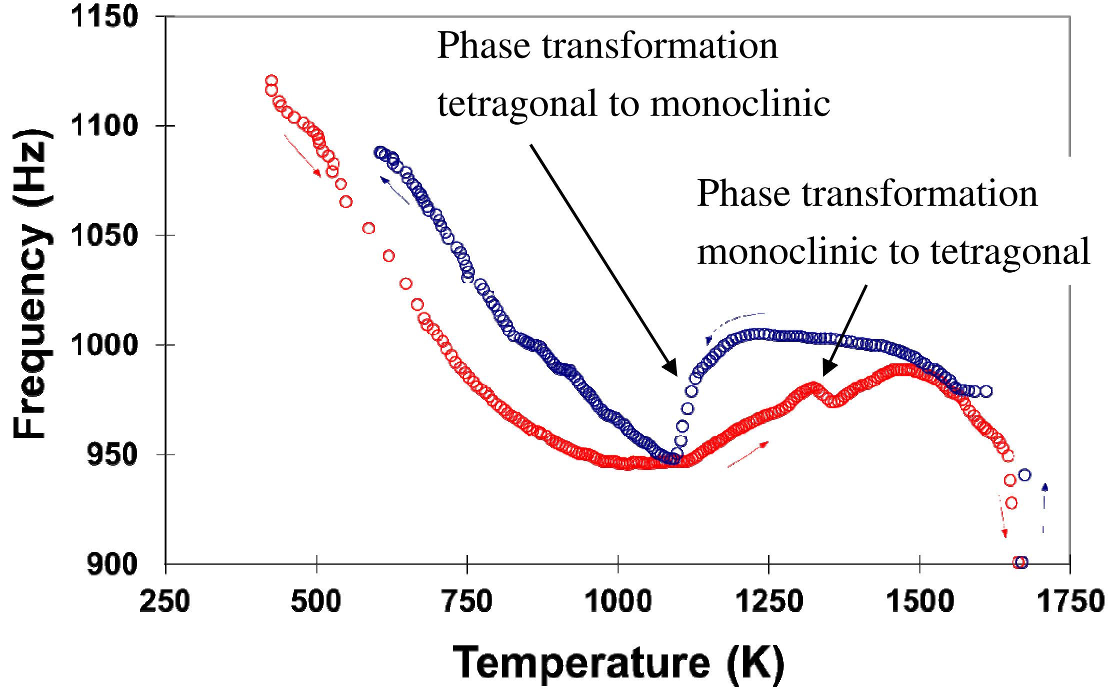


Study:

- ❑ Dislocations
- ❑ Relaxation
- ❑ Interstitials
- ❑ ……

J. Jung, E. Kozeschnik, S. Ho Han, B.C. De Cooman, "Analysis of the Mechanical Properties of N-Added CMn Structural Steel by the Impulse Internal Friction Technique", Metallurgical and Materials Transactions A, vol. 43A (2012), 4587 - 4600

ZrO_2 partially stabilized with 2.7% MgO



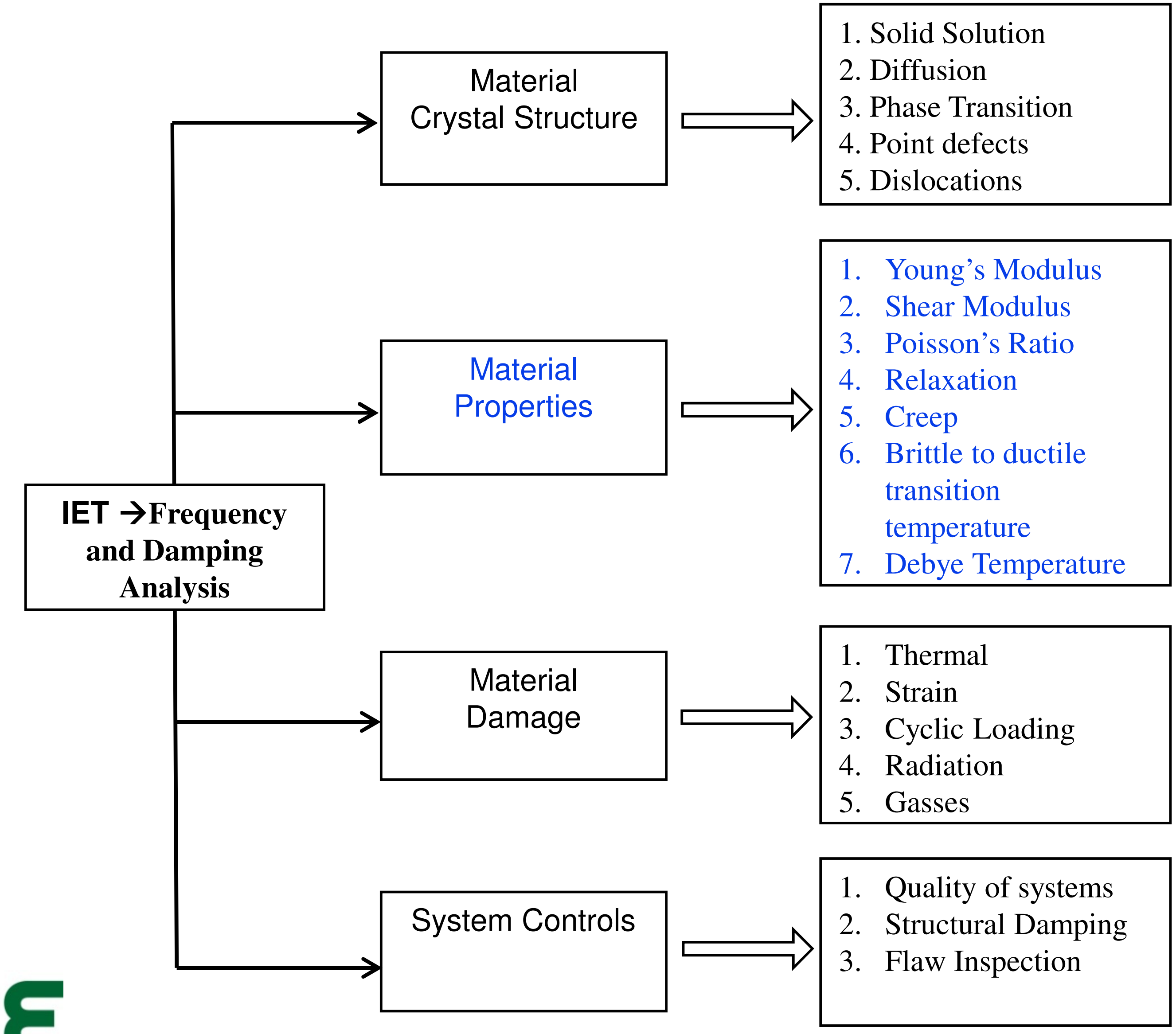
Change of resonant frequency is explained by the phase transformation of the material.

Study:

- ❑ Phase Transformations
- ❑ Grain boundaries
- ❑ ……

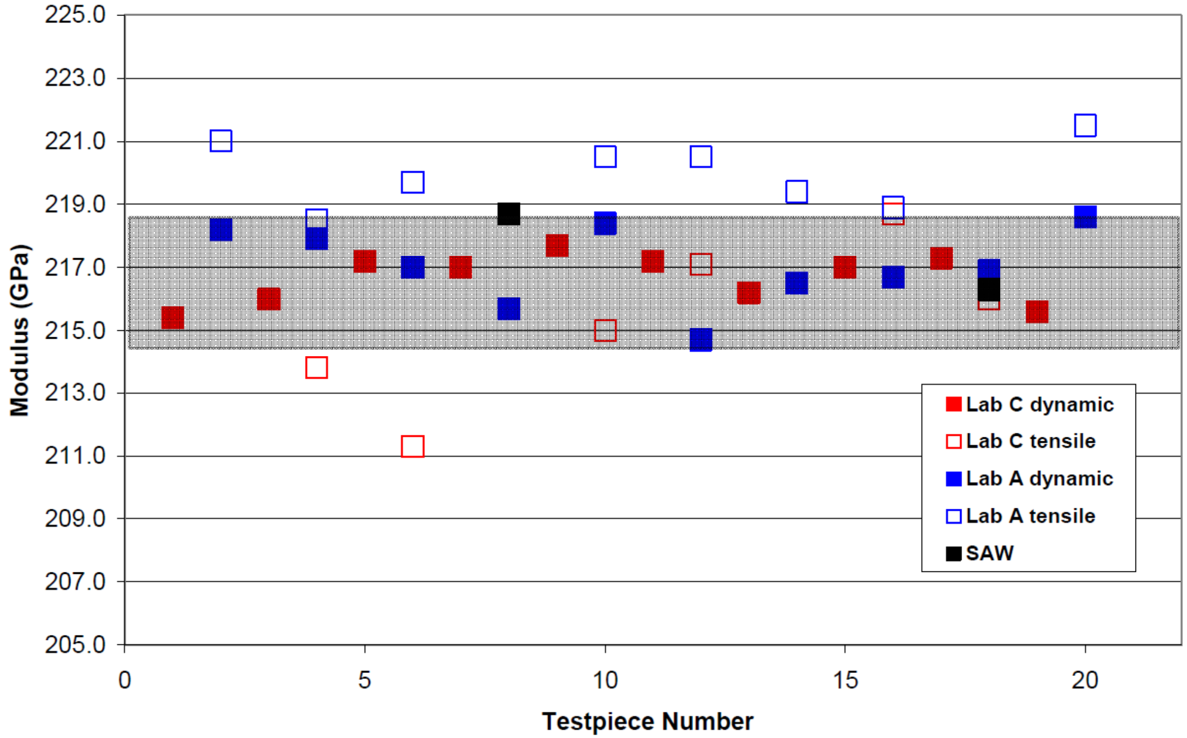
IET: Cases

Application domains and results



IET: Cases
Material properties

Comparison of the dynamic, tensile and SAW measurements on flat machined testpieces – BCR Nimonic 75 (CRM 661) tensile reference material



Study:

- ❑ Dynamic Young's modulus
- ❑ Dynamic Shear modulus
- ❑ Poisson's ratio

J.D. Lord, R. Morrell, A National Measurement Good Practice Guide, No. 98, Elastic Modulus Measurement, National Physical Laboratory, Teddington (2006)

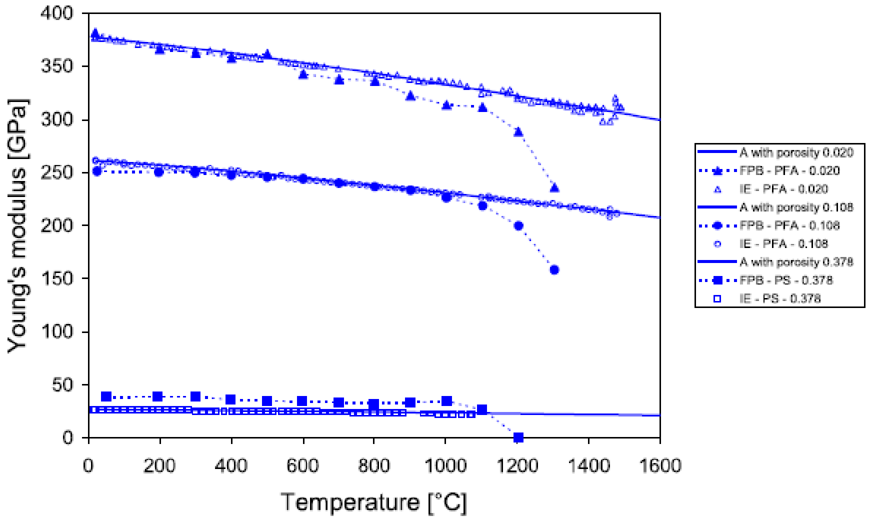
IET: Cases
Material properties

Comparison of the dynamic and static Young's modulus

	Temperature [°C]	E_{STAT} [GPa]	E_{DYN} [GPa]
Alumina	25	400.3	400.8
	500	374.3	375.7
	1000	345.9	348.1
Zirconia	25	209.2	209.6
	500	186.9	187.8
	1000	139.6	140.5

Difference < precision of measurements

Temperature dependence of the Young's modulus for alumina ceramics with different porosity

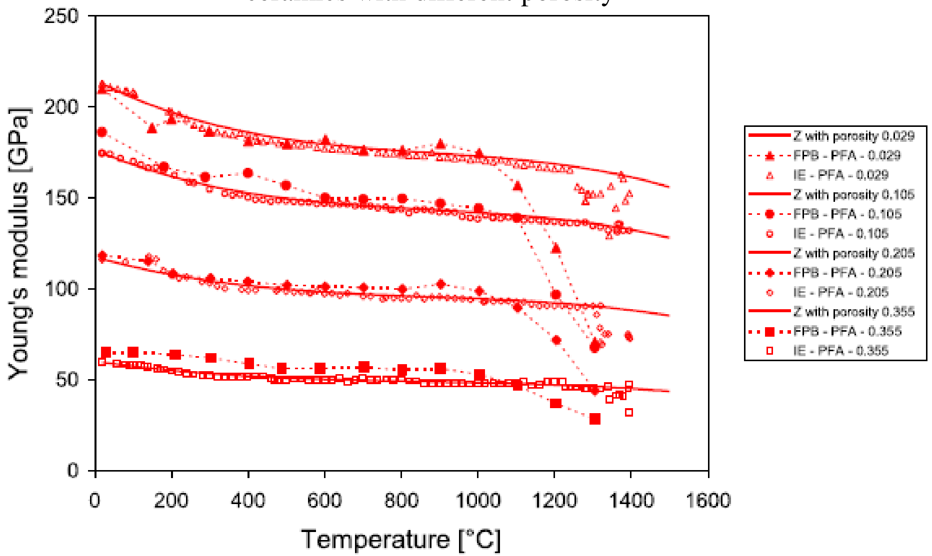


Linear dependency between porosity and E

$$E = E_0(1 - 2\phi)$$

Porosity dependency can be decoupled from the temperature dependency of E

Temperature dependence of the Young's modulus for zirconia ceramics with different porosity



Willi Pabst, Eva Gregorová, Marting Cerný, Isothermal and adiabatic Young's moduli of alumina and zirconia ceramics at elevated temperatures, Journal of the European Ceramic Society 33(2013) 3085-3093

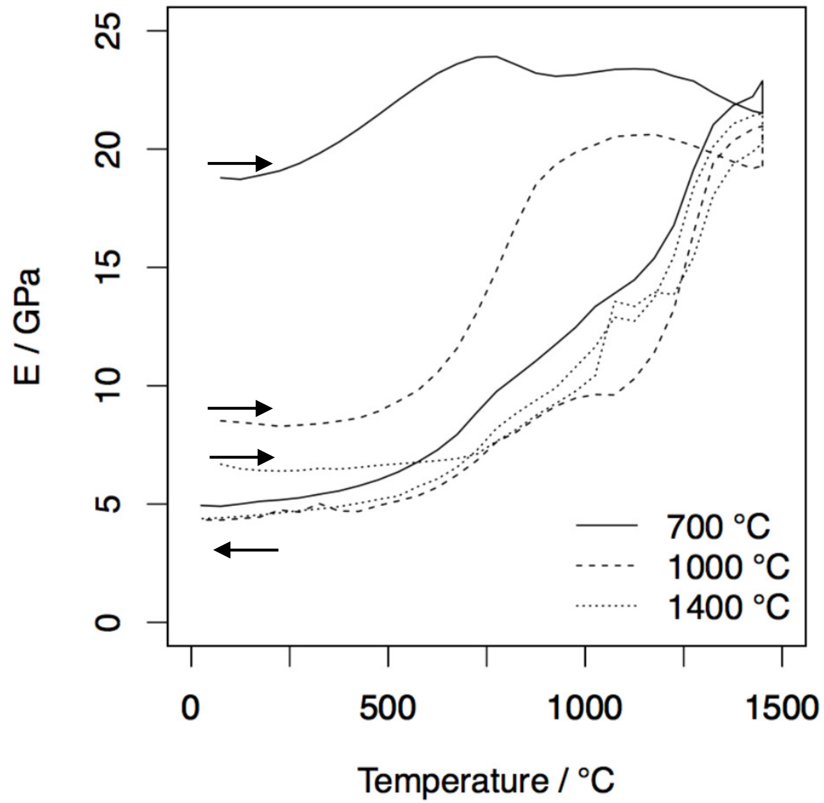
IET: Cases
Material properties / process parameters

Carbon bonded alumina containing 30 wt% carbon treated with 3 different pyrolysis temperature.

Due to mismatch of thermal expansion coefficients of alumina, graphite and carbonized phenolic resin, the alumina particles detach from the surronding matrix during cooling after the pyrolysis. This disturbed microstructure determines the low mechanical properties of the refractory material at room temperature

- Significant dependence on E(T)
- Reducing hysteresis with an increasing pyrolysis temperature
- The maximum E and residual E are not dependent on the pyrolysis temperature

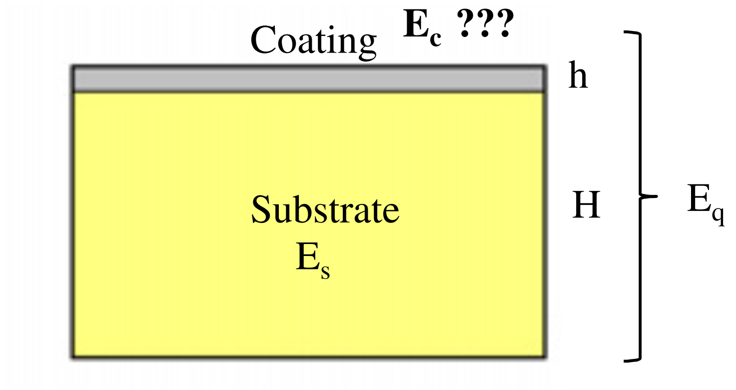
Temperature dependence of the Young's modulus for different pyrolysis temperatures



J. Werner, C.G. Aneziris, The influence of pyrolysis temperature on the Young's modulus of elasticity of carbon-bonded alumina at temperatues up to 1450 C, Ceramics International, Volume 42, Issue 2, Part B, 2016, pg 3460-3464

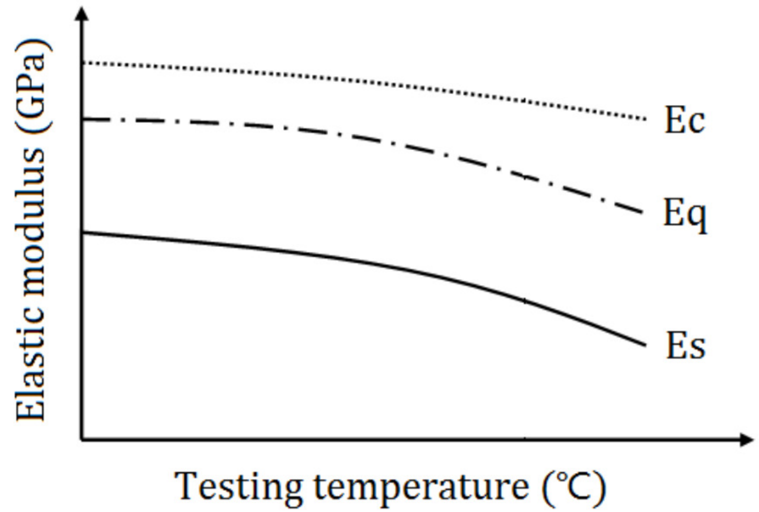
IET: Cases
Coatings

Young's modulus of coating: comparison between the elastic properties of the uncoated and coated sample



$$\frac{E_c}{E_s} = \frac{-U + \sqrt{U^2 + V}}{2R^3}$$

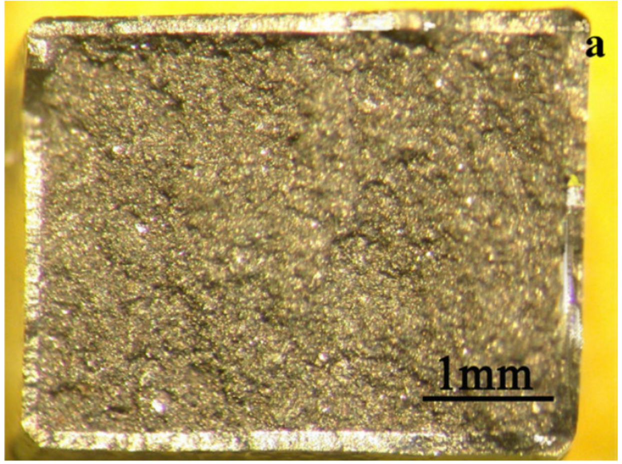
U, V and R are constants only dependent on h, H, E_q and E_s



ISO 20343 standard

IET: Cases
Coatings

Young's modulus of coating: comparison between the elastic properties of the uncoated and coated sample

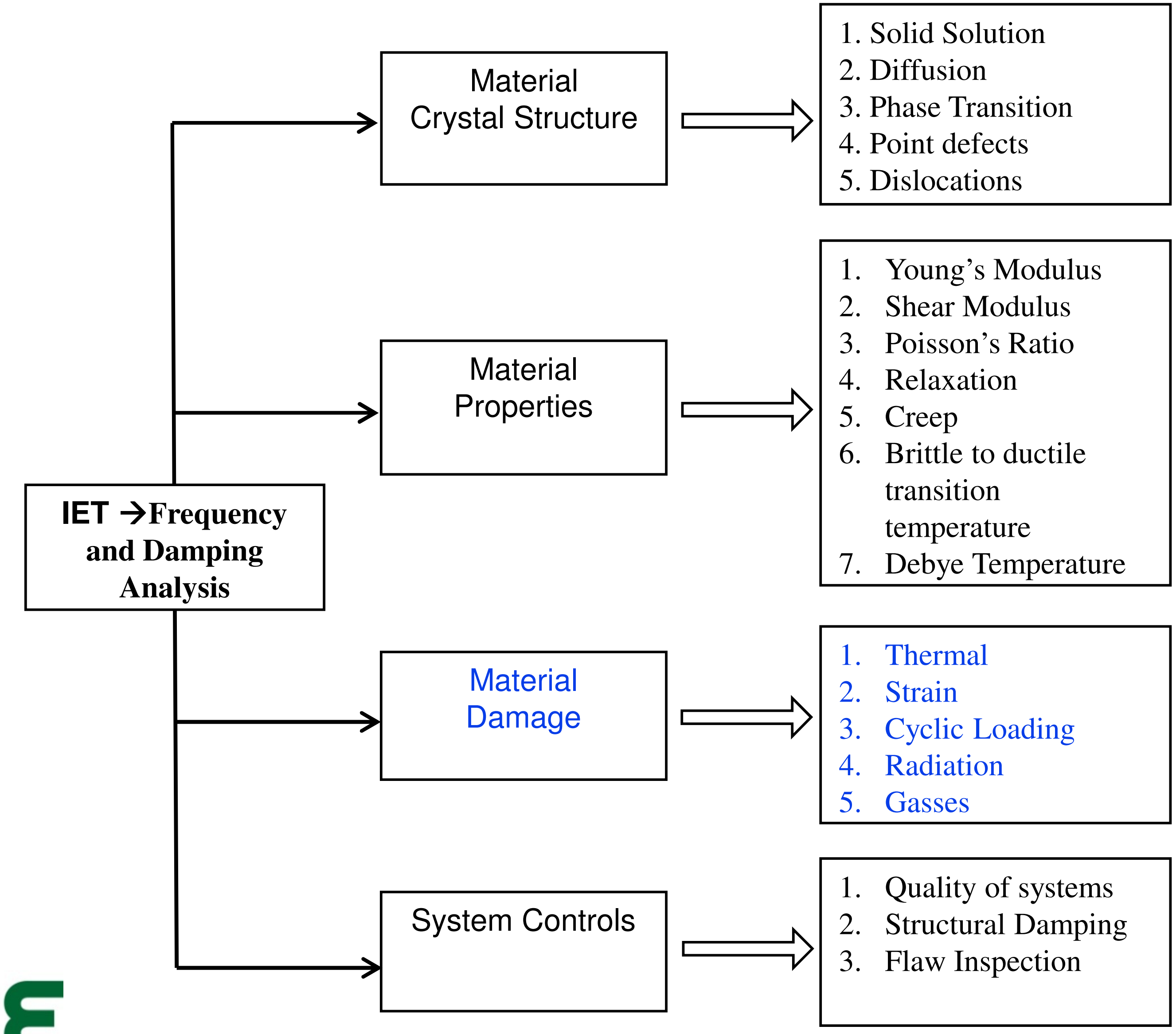


Data of SiC around-coated on graphite substrate, $H = 3$ mm, $B = 4$ mm, $E_s = 10$ GPa, $L = 30$ mm

No.	h (mm)	R	E_c (GPa)	E_f (GPa)
1	0.18	0.060	120	327
2	0.18	0.060	115	313
3	0.19	0.063	128	336
4	0.18	0.060	117	318
5	0.20	0.067	130	329
Average			122 ± 7	325 ± 9

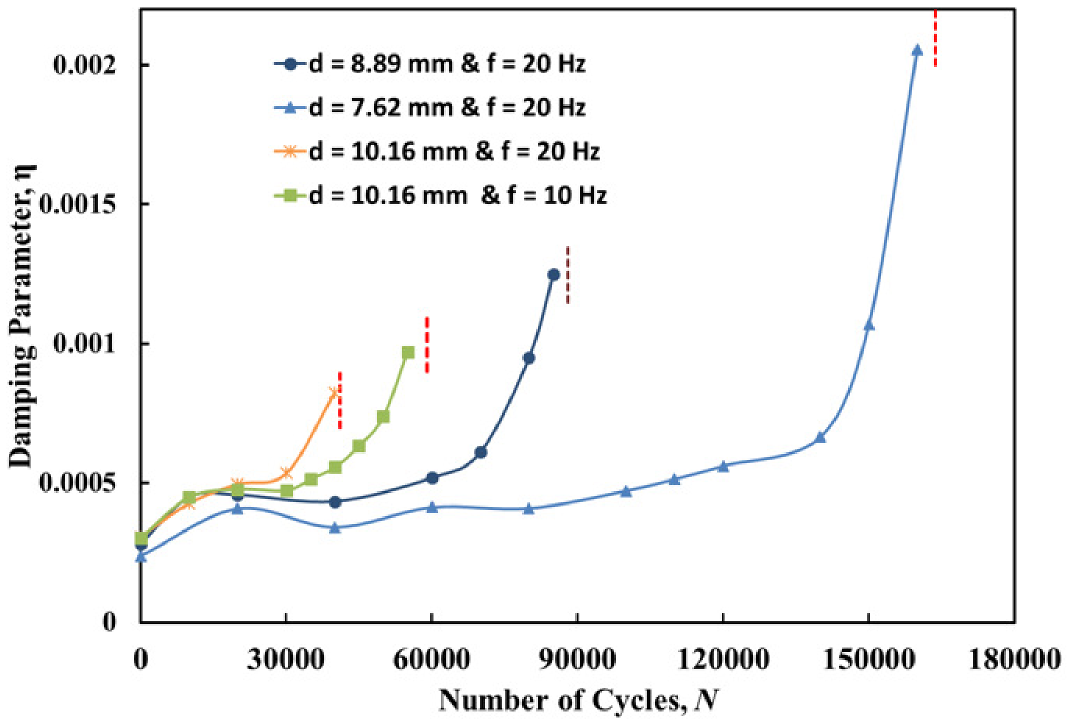
Evaluating elastic modulus and strength of hard coatings by relative method, Y.W. Bao *et al.*, Mat. Sci. Eng. A 458 (2007) 268

IET: Cases
Application domains and results



IET: Cases

Fatigue analysis of metals



Carbon steel 1018 sample during fatigue test

Measured Damping parameters versus number of cycles during fatigue tests for different stress levels, LCF

First order relative changes damping => characteristic for crack formation

Fatigue analysis of metals using damping parameter, V. Mortezavi *et al.*, Int. J. Fatigue 91 (2016) 124

IET: Cases

Material damage

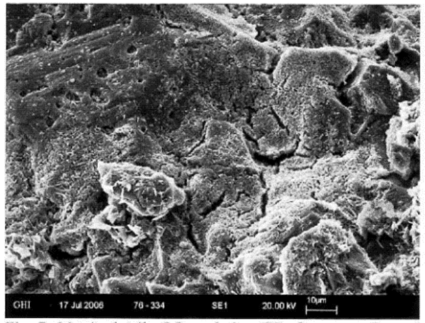
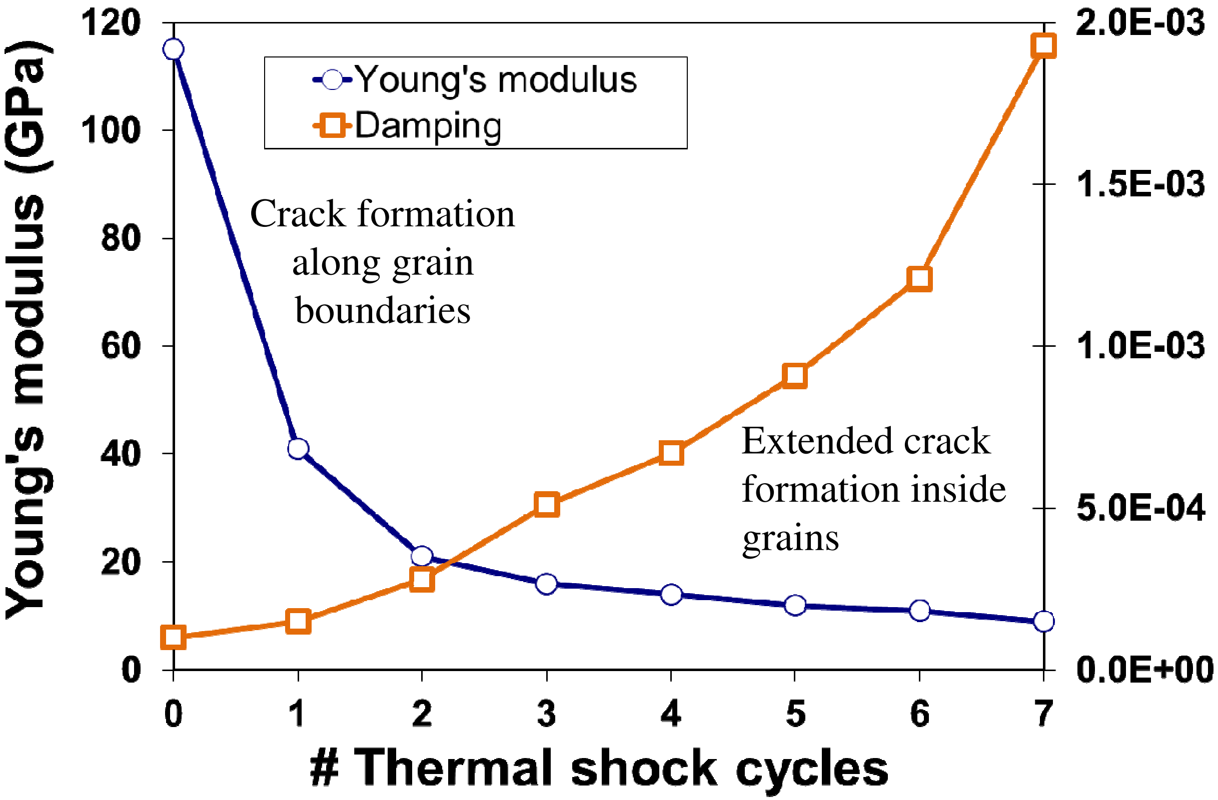


Fig. 7: Matrix detail of formulation *CR* after seven thermal shock cycles obtained by SEM

Resonant Frequency and Damping Analysis vs. Thermal shock cycles



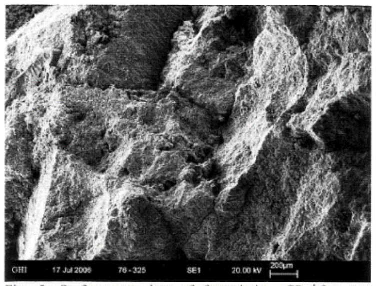
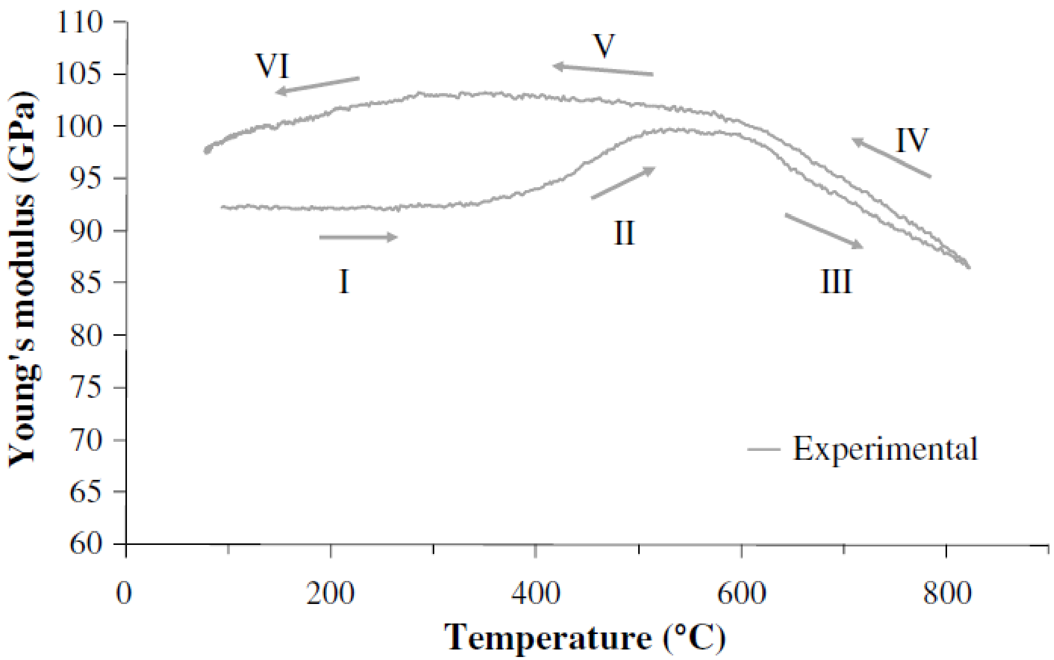


Fig. 5: Surface overview of formulation *CR* after two thermal shock cycles obtained by SEM

T. Tonnesen, R. Telle,"Evaluation of Thermal Shock Damage in Castables by a Resonant Frequency and Damping Method" Proc. 49. Int. Feuerfest-kolloquium, pp. 133-136, 2006

IET: Cases
Material damage

Young's modulus variations vs temperature for a multi inclusions material containing 30% of spherical inclusions



E. Yeugo Fogain, High temperature characterization of the elastic properties of fused –cast refractories and refractory castables, Thesis, University of Limoges, France 2006

- Stage I: E decreases lightly because of lowering of the atomic bonding rigidity
- Stage II: E increases because closure of debondings due to the influence of CTE mismatches between the phases. Thermal expansion of the inclusion part is greater than the one of the matrix part => damage reduces
- Stage III: E decreases because of formation of phases of low viscosity. At the end of this stage the material is partially or fully cured and can be considered free of stress
- Stage IV: E increases due to the solidification of the low viscosity phases
- Stage V: E increases while the interatomic bondings are rigidified and stresses develop in the material but do not reach the strength value
- Stage VI: E decreases due to internal stresses due to CTE mismatches and increase of the internal damages of the material

IET: Cases
Refractory research

Study of damage of high zirconia fused-cast refractories by measurement of Young's modulus

A. Sibil[a,*], J.P. Erauw[b], F. Cambier[b], M. R'Mili[a], N. Godin[a], G. Fantozzi[a]

[a] MATEIS, INSA Lyon, Bât. Blaise Pascal, 7 av. Jean Capelle F-69621 Villeurbanne, France
[b] BCRC, av. Gouverneur Cornez 4 B-7000 Mons, Belgium

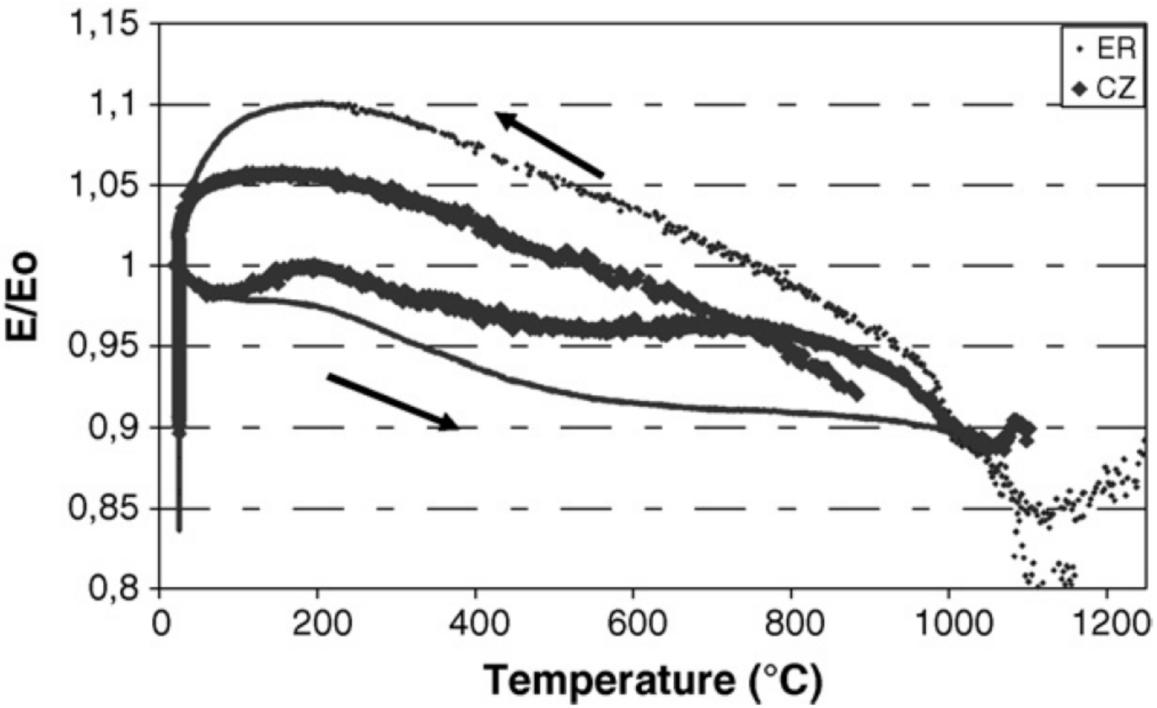
Materials Science and Engineering A 521–522 (2009) 221–223

2 materials wit different composition of their glassy phase were studied:

ER: ER1195 sodic glassy phase

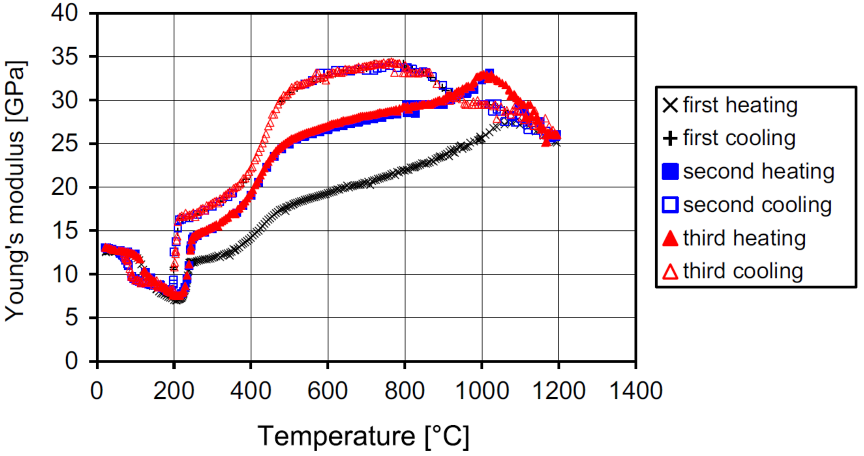
CZ: SCIMOZ glassy phase with boron

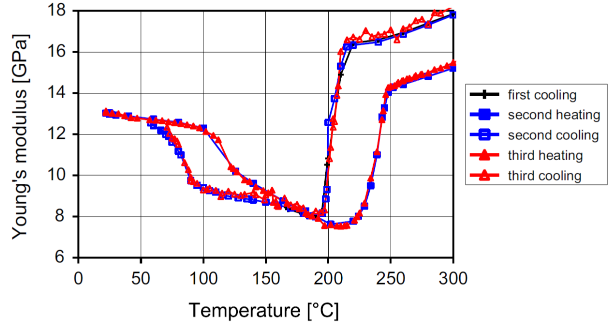
The smaller hysteresis loop of the CZ material indicates a smaller thermal expansion mismatch between the glassy phase and zirconia



IET: Cases
Refractory research / Silica brick

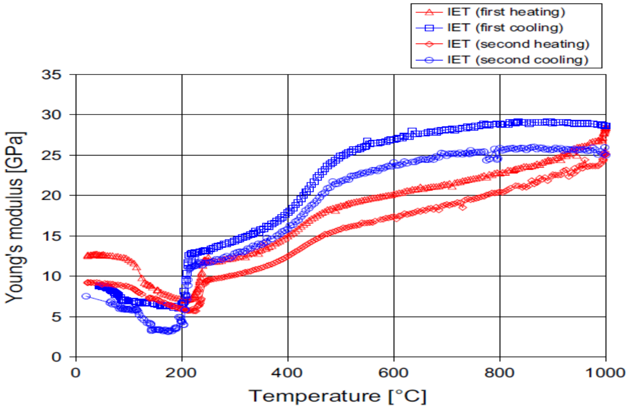
Temperature decency of E of a silica brick material with 19.4% porosity measured up to 1200 C





Detailed view up to 300 C showing a precisely reproducible hysterisis loop

Temperature decency of E of a silica brick material with 18.6% porosity measured up to 1000 C



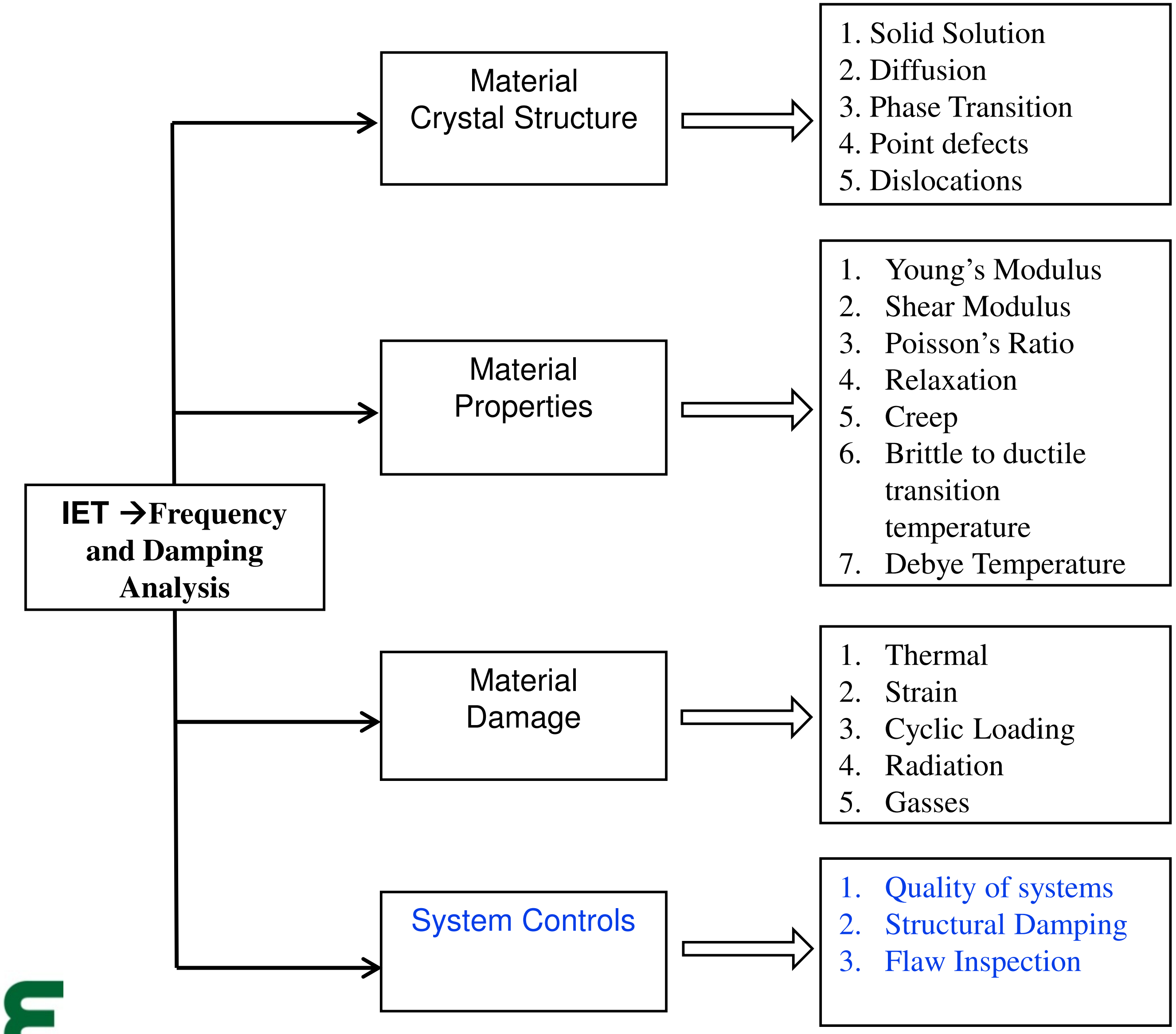
Elastic anomalies:

- Stiffness decrease at low temperature: 50 – 150 C
- Broad transition region at low temperature: 150-250 C
- Sharp transition starting from 210-220 C
- Max. E at 800 C (during cooling)

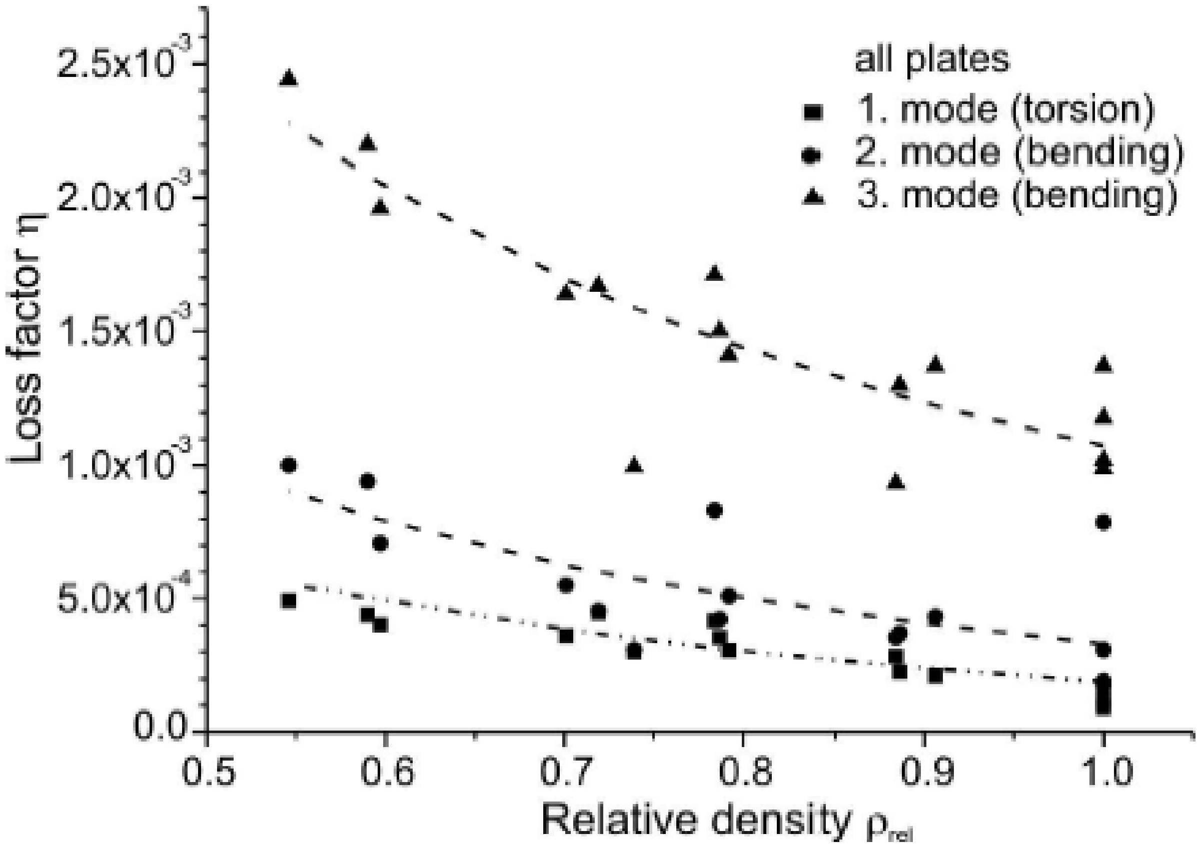
- E(T) on the max. temperature reached, this due to the damage accumulation. This damage accumulation is more severe when bricks are heated to lower temperature. This damage accumulation must be attributed to additional microcracking

W. Pabst, E. Gregorová, J. Kutzendörfer, Elatic anomalies in tridymite- and cristobalite-based silica materials, Ceramics International 40 (2014) 4207-4211

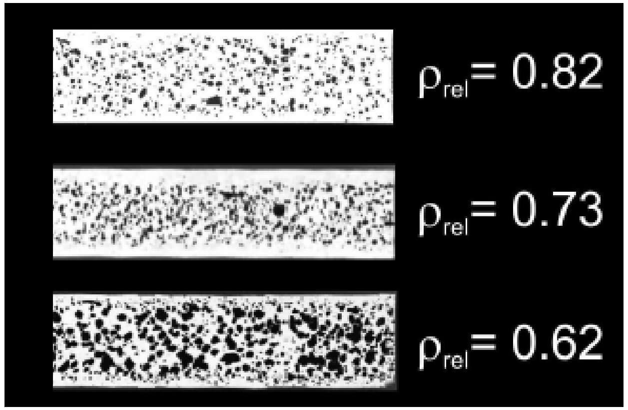
IET: Cases
Application domains and results



IET: Cases
System analysis



Loss factor as a function of relative density for the first three modes of foam materials



Aluminium foam material

- ❑ Study of damping behavior for structural components in lightweight structures
- ❑ ….

A.Trepper, C. Körner, R.Singer, "Structural Damping of Integral Foam Componets", Porous Metals and Metallic Foams, Procedings of the 5th Intl Conf on Porous Metals and Metallic Foams, Sept. 5-7, 2007, Montreal, pp. 43-46, 2008

Conclusions

The Resonant Frequency and Damping Analyser (RFDA) is basically non-destructive and can be applied for :

- Accurate determination of resonant frequencies and damping.
- Accurate quantification of elastic moduli (Young’s Modulus, Shear Modulus, coefficient of Poisson according to ASTM E-1876, ISO 12680-1, ENV 843-2).
- Tests as a function of temperature, also on components.
- Non-destructive quality-control of components before use and (fatigue) cracks or diffuse damage after use or testing.
- In-situ monitoring of micro-structural developments during processing.

Thank you for your attention

Ing. Bart Bollen

IMCE NV

Slingerweg 52
B-3600 Genk
Belgium

Tel:	+32.89.41.00.70
Fax:	+32.89.41.00.79
E-mail:	bart.bollen@imce.net
Web:	http://www.imce.net