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!?
Ein neues Meßverfahren zur Bestimmung des Elastizitäts- moduls und der Dämpfung

Von Fritz Förster in Stuttgart

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

Commonly used vibration modes for a prismatic beam

Fundamental flexure mode

Fundamental torsion mode
**IET: principle**

Relation resonance frequency - stiffness

Transverse vibration of homogenous isotropic prismatic bars

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

- **Euler-Bernoulli**
  - Deformation energy
  - Energy of motion
  - Rotatory inertia

- **Rayleigh**
  - Shear deformation contribution

- **Timoshenko**
  - Shear modulus
  - Density
  - Cross-section shape factor


Integrated material control engineering
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 the 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
- $\rho$ = density
- $L$ = length, $t$ = thickness
- $T_1$ = correction factor
  = $f(t/L, \text{Poisson’s ratio } \nu)$

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

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

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**

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**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 $Q^{-1}$.

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

$f = 1/T = \omega/2\pi$: resonant frequency

$k$: exponential damping

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

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**Digital Signal Processing**
**IET: principle**
Simultaneous multiple frequencies and damping analysis

- Computer supported IET measurement systems

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

Example: vibration signal lasts 0.5 s \(\Rightarrow\) \(\frac{44100}{22050} = 2 \text{ Hz}\)

vibration signal lasts 0.1 s \(\Rightarrow\) \(\frac{44100}{4410} = 10 \text{ Hz}\)

- Enhanced algorithms (RFDA) \(\Rightarrow\) \(\frac{\Delta f}{f} < 10^{-4}\)
Damping or Internal friction \((Q^{-1})\) is energy dissipation in a material due to the movement of microstructural features.
**IET: principle**

Measuring results

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

### RFDA Professional
- 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 restrictions 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 °C (optionally to 1050 °C)
- Heating rate 1-5 °C/min
- Air atmosphere (optionally with gas flow)

RFDA HT1600
- Up to 1600 °C or 1750 °C
- Heating rate 1-5 °C/min
- Air atmosphere (optionally with gas flow)

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

RFDA HTVP1750C
- Heating rate 1-5 °C/min
- HTVP1750C: inert or vacuum

RFDA LTVP 800
- From -50 °C to 800 °C
- Heating rate 1-60 °C/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
Example: High alumina castable material
**IET: Cases**

Application domains and results

- IET → Frequency and Damping Analysis
  - Material Crystal Structure
    - 1. Young’s Modulus
    - 2. Shear Modulus
    - 3. Poisson’s Ratio
    - 4. Relaxation
    - 5. Creep
    - 6. Brittle to ductile transition temperature
    - 7. Debye Temperature
  - Material Properties
    - 1. Solid Solution
    - 2. Diffusion
    - 3. Phase Transition
    - 4. Point defects
    - 5. Dislocations
  - Material Damage
    - 1. Thermal
    - 2. Strain
    - 3. Cyclic Loading
    - 4. Radiation
    - 5. Gasses
  - System Controls
    - 1. Quality of systems
    - 2. Structural Damping
    - 3. Flaw Inspection

IMCE

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

ZrO$_2$ partially stabilized with 2.7% MgO

Phase transformation tetragonal to monoclinic

Phase transformation monoclinic to tetragonal

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

Study:
- Dislocations
- Relaxation
- Interstitials
- ......

**IET: Cases**

**Application domains and results**

- **Material**
  - Crystal Structure
  - Properties

  - Young’s Modulus
  - Shear Modulus
  - Poisson’s Ratio
  - Relaxation
  - Creep
  - Brittle to ductile transition temperature
  - Debye Temperature

- **IET → Frequency and Damping Analysis**
  - Material
  - Damage

  - Thermal
  - Strain
  - Cyclic Loading
  - Radiation
  - Gasses

- **System Controls**
  - Quality of systems
  - Structural Damping
  - Flaw Inspection
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

Comparison of the dynamic and static Young’s modulus

<table>
<thead>
<tr>
<th></th>
<th>Temperature [°C]</th>
<th>E_{STAT} [GPa]</th>
<th>E_{DYN} [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alumina</td>
<td>25</td>
<td>400.3</td>
<td>400.8</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td>374.3</td>
<td>375.7</td>
</tr>
<tr>
<td></td>
<td>1000</td>
<td>345.9</td>
<td>348.1</td>
</tr>
<tr>
<td>Zirconia</td>
<td>25</td>
<td>209.2</td>
<td>209.6</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td>186.9</td>
<td>187.8</td>
</tr>
<tr>
<td></td>
<td>1000</td>
<td>139.6</td>
<td>140.5</td>
</tr>
</tbody>
</table>

Difference < precision of measurements

Linear dependency between porosity and E

\[ E = E_0 (1 - 2\phi) \]

Porosity dependency can be decoupled from the temperature dependency of E

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 surrounding 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
**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
Young’s modulus of coating: comparison between the elastic properties of the uncoated and coated sample

![Image showing a sample with 1mm scale]

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

<table>
<thead>
<tr>
<th>No.</th>
<th>$h$ (mm)</th>
<th>$R$</th>
<th>$E_c$ (GPa)</th>
<th>$E_f$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.18</td>
<td>0.060</td>
<td>120</td>
<td>327</td>
</tr>
<tr>
<td>2</td>
<td>0.18</td>
<td>0.060</td>
<td>115</td>
<td>313</td>
</tr>
<tr>
<td>3</td>
<td>0.19</td>
<td>0.063</td>
<td>128</td>
<td>336</td>
</tr>
<tr>
<td>4</td>
<td>0.18</td>
<td>0.060</td>
<td>117</td>
<td>318</td>
</tr>
<tr>
<td>5</td>
<td>0.20</td>
<td>0.067</td>
<td>130</td>
<td>329</td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td>122 ± 7</td>
<td>325 ± 9</td>
</tr>
</tbody>
</table>

**IET: Cases**

Application domains and results

**Material Crystal Structure**
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**Material Properties**
- 1. Thermal
- 2. Strain
- 3. Cyclic Loading
- 4. Radiation
- 5. Gasses

**IET → Frequency and Damping Analysis**
- 1. Solid Solution
- 2. Diffusion
- 3. Phase Transition
- 4. Point defects
- 5. Dislocations

**Material Damage**
- 1. Quality of systems
- 2. Structural Damping
- 3. Flaw Inspection

**System Controls**
- 1. Thermal
- 2. Strain
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Fatigue analysis of metals using damping parameter, V. Mortezavi et al., Int. J. Fatigue 91 (2016) 124
**IET: Cases**

**Material damage**

Resonant Frequency and Damping Analysis

vs. Thermal shock cycles

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

- 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

E. Yeugo Fogain, High temperature characterization of the elastic properties of fused –cast refractories and refractory castables, Thesis, University of Limoges, France 2006
Study of damage of high zirconia fused-cast refractories by measurement of Young's modulus

A. Sibil, J.P. Etauw, F. Cambier, M. R’Mili, N. Godin, G. Fantozzi

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BCIC, av. Gouverneur Cornex 4 B-7000 Mons, Belgium


2 materials with 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

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

IET: Cases
Application domains and results

IET → Frequency and Damping Analysis

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integrated material control engineering
Aluminium foam material

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


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

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