ФИЗИКА

THE APPARATUS FOR MEASUREMENT OF YOUNG'S MODULU OF CERAMICS AT ELEVATED TEMPERATURES

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Abstract: The construction of a laboratory-made fully automated resonant apparatus for the modulated force thermomechanical analysis (mf-TMA) is described. The measurement is based on the impulse excitation technique (IET). The apparatus is able to provide the measurements of the resonant frequency, which is a base for determination of Young's modulus, sound velocity and internal damping during heating up to 1200 °C. An expanded relative uncertainty of the Young's modulus determination at elevated temperatures is 1.1 %.
Keywords: resonant measurement, Young's modulus, impulse excitation technique

1 Introduction

Young's modulus is one of the most frequently measured mechanical parameters of materials. Young's modulus depends on the structure of the material as well as external conditions. Therefore, it can be used for the indirect measurement and investigation of porosity, texture, intergranular phases, composition, the creation of cracks, phase changes, sintering, hardness, apparent density, compressive strength, flexural strength, and others.

A great number of experimental techniques have been developed to determine the elastic properties of ceramic materials. Young's modulus can be measured using static or dynamic methods. The disadvantage of the static methods is the impossibility of continuous measurement; therefore these methods are not suitable for thermomechanical analysis (TMA). Methods, which meet the requirements of TMA use resonant frequency as a base for determining Young's modulus. These methods are most often based on the flexural vibration of the sample, which can be easily excited, has large amplitude of deflection and are reliable at high temperatures. Samples have simple geometry (a cylinder/prism with uniform cross-section or circular plate).

The sample can be made to vibrate at the resonant frequency either by a driver that continuously varies the frequency of the output signal or by a mechanical impact. Both methods utilize the same formula for the calculation of Young's modulus. The measured quantities are the dimensions and mass of the sample and the resonant frequency [1-6]. Two techniques serve to acquire the resonant frequency.

The first technique, so called sonic resonance method (SRM), is based on the forced vibration of the sample with the known frequency. The equipment is relatively simple. It contains tunable RC oscillator with an amplifier connected to an exciter as a source of the forced vibration. Sample vibrations are registered with a sensor connected to a preamplifier and its output signal is observed on AC mV-meter (or oscilloscope). The frequency, at which the mV-meter signal reaches the maximum value, is the resonant frequency. The resonant frequency can be found manually and checked with some methods if the frequency belongs to the fundamental mode of the flexural vibration [7]. This method can be automated if the RC oscillator works in a sweeping regime [8, 9].

The second technique, so called impulse excitation technique (IET), is based on the free vibrations of the sample after being exposed to a mechanical impulse [4, 10]. The free vibrations are registered by the sensor, stored and then analyzed using the fast Fourier transformation (FFT). The result of FFT is a frequency spectrum of the sample vibration, where the resonant frequency of the fundamental mode of the flexural vibrations can be found. The free vibrations are naturally dampened by internal processes in the sample. Therefore, a coefficient of the internal damping (internal friction) can be also determined. The mechanical impulse can be realized by the steel or ceramic projectiles which fall down on the sample, or by the electromagnetic impulse tool (which is probably most often used) or by the exciter fed by the impulse signal. The IET was significantly developed after progress in computer technology and today is more used than SRM.

To compare SRM with IET, both methods give the same resonant frequency, i.e. their accuracy is identical. But IET can be designed as non-contact while SRM needs two thin wire suspensions located in the antinodal points of the sample. These suspensions are weak points of SRM – they are often a source of false resonances and their strength is limited at high temperatures. Consequently, IET is more reliable at high temperatures. In addition to that, IET allows a simultaneous measurement of the resonant frequency and internal damping of the sample.

The aim of the paper is a description of the apparatuses used in the Thermophysical Laboratory of the Constantine the Philosopher University in Nitra.

2 The apparatus for SRM

We constructed two apparatuses for SRM – one for a room-temperature measurement and the other for the high-temperature measurement. A principal scheme of the room-temperature apparatus is depicted in Fig. 1.

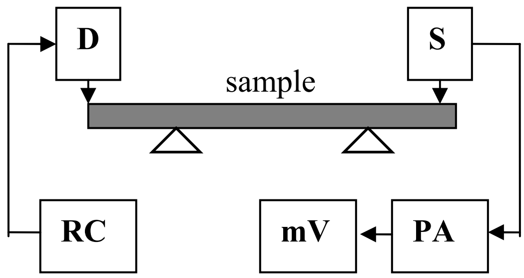

Fig. 1. The scheme of the room-temperature apparatus for SRM. D – driver, S – sensor, RC – RC oscillator, mV- AC mV-meter, PA – preamplifier.

The sample is put on soft supports located 0.224 l from the both ends of the sample, i.e. in the nodal points of the fundamental mode of the flexural vibration. The sensor is movable, so we can measure a sample deflection in different points and unambiguously determine the vibrating mode. The RC oscillator can work in a sweeping regime or can be tuned manually. The other variant of the apparatus is that it has a non-contact exciter which is a speaker under the middle of the sample. The sensor is a gramophone piezoelectric cartridge.

The high-temperature apparatus has the same principal scheme, but the sample is suspended on PtRh10 wires with a diameter of 0.15 mm as visible in Fig. 2. A furnace is connected to a temperature programmer Classic Clare 4.0 to obtain linear heating with a rate of 1 – 10 °C/min or isothermal heating at selected temperatures up to 1250 °C. The high-temperature apparatus uses a piezoelectric sensor and electrodynamical driver. The furnace is built from porous alumina bricks and alumina fiber pads. The working space (20×10×10 cm) is heated with four silicon carbide rods.

A description of this apparatus is in [8], where an uncertainty analysis of the measurement of Young's modulus is also given. We successfully used this apparatus in our research that is documented in a number of articles, e.g. [11-15]. The relative expanded uncertainty 1.1 % of the Young's modulus at the elevated temperatures evaluated in [8] is also valid for the apparatus pictured in Fig. 3. We empirically confirmed that resonant frequencies determined by the IET and SRM are identical. The same is known from literature [16].

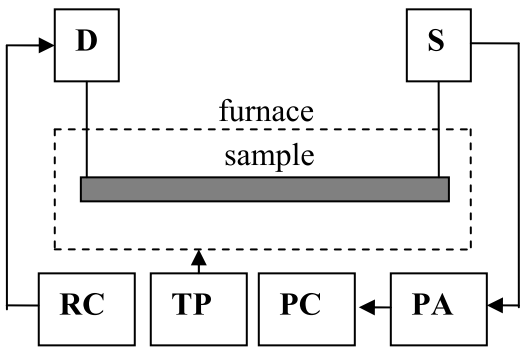

Fig. 2. The scheme of the high-temperature apparatus for SRM.
D – driver, S – sensor, RC – RC oscillator,
PC computer, PA – preamplifier, TP – temperature programmer.

3 The apparatus for IET

In spite of the long history of experience with SRM, we decided to construct a new apparatus on a base of IET because of its advantages that are mainly higher reliability at high temperatures and simultaneous measurement of the resonant frequency and internal damping of the sample. The scheme of this apparatus is depicted in Fig. 3.

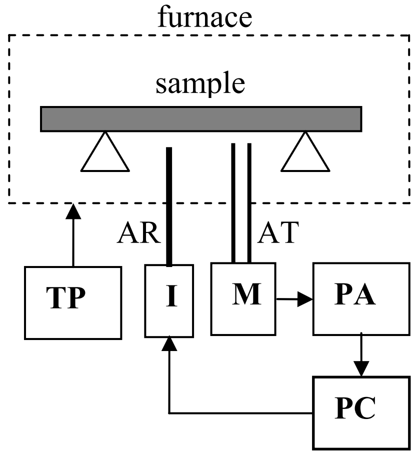

Fig. 3. The scheme of the high-temperature apparatus for IET.
I – impactor, AR – alumina rod, M – microphone, AT – alumina tube,
PA – preamplifier, PC - computer, TP – temperature programmer.

3.1 Impactor

To excite vibrations in the sample, the sample must be hit with an impactor (impulse tool). It can be a little hammer moved manually in the case of the room-temperature apparatus, or an electromagnetic hammer in the case of the high-temperature apparatus. The hammer must hit the sample in the area between nodal points as pictured in Fig. 3. We used the impactor depicted in Fig. 4, where a control circuit is also visible. The impactor is made from alumina rod Ø3 mm that is moved in a vertical direction by an electromagnet.

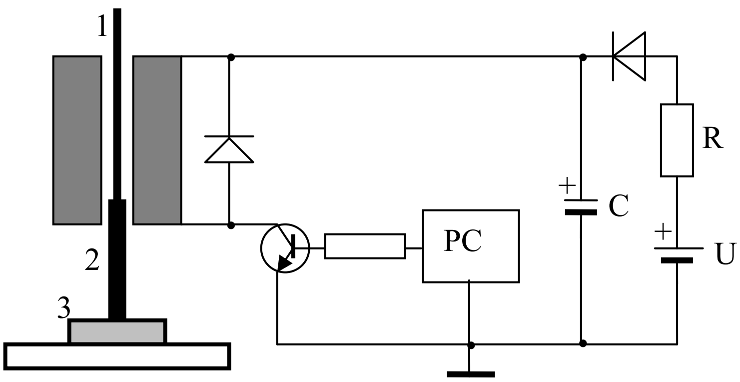

Fig. 4. Impactor. 1 – alumina rod, 2 – iron core in a coil, 3 – rubber pad,
C = 0.1 F (25 V), R = 6.5 Ω, U = 5 – 8 V,
PC – impulse generator in PC, two diodes 45V/2A, transistor KD602.

The impactor is controlled by a computer program and executed automatically every t seconds. The period t is chosen by the user. Every t seconds the main program calls a subprogram which sends the signal to the impactor, records the response of the sample and processes the recorded signal. The period t must be long enough to charge the capacitor ($t > 4$ s). After every t seconds, the opening signal is sent from the computer via the Advantech PCI-1710HG card interface to the transistor base. The transistor is open, and the capacitor is discharged through the coil which attracts an iron core up and hits the sample. This signal from the computer is adjustable, and its power is proportional to the collector current of the transistor, i.e. to the intensity of the impactor hit. The program adjusts the impulse voltage automatically in order to reach the lowest power of impact to detect the vibrations. When the output of the preamplifier is under the selected threshold, the impulse voltage is slightly increased. After the hit, the current gradually decreases and the core moves down softly. For the measurement period t we usually chose 15 or 30 s.

3.2 Sample holder

It is desirable to have the sample supported in its nodal points of the fundamental mode of the flexural vibrations. Because the mechanical impulse is applied vertically, as visible in Fig. 3, the sample must be reliably fixed with wires. We used kanthal wire Ø0.3 mm. The sample, 130 mm long, is put in a horizontal position on the wires in the two nodal points and two other wires fix the sample. Since the ceramic sample shrinks during heating, two weights maintain sufficient forces for fixing the sample (Fig. 5). A similar technique is also used in [17, 18].

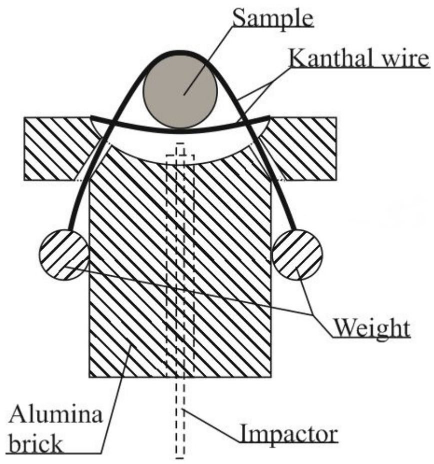

Fig. 5. The sample holder.

3.3. Furnace and temperature control.

The furnace is built from refractory porous alumina bricks and alumina fiber pads. A working space is 120×120×250 mm where the holder with the sample is located in a homogenous temperature field. The heating elements are four silicon carbide rods connected to a temperature programmer Clasic Clare 4.0. The temperature is measured with Pt-PtRh10 thermocouple in the close vicinity of the sample.

The measurements can be run in different temperature regimes including heating and cooling with a rate from 1 °C/min to 10 °C/min or isothermal heating at selected temperatures up to 1200 °C.

4 Measurement of the sound response

4.1 Sensor and preamplifier

We use an electret microphone to catch the sound response of the sample on the impulse excitation. Since the microphone must be located outside the furnace, an alumina tube with an inner diameter of 5.6 mm and a length of 230 mm was used as a sound guide between the microphone and the sample. There was a small gap of ~2 mm between the alumina tube and the sample, so the measurement of the sample vibrations was contact-free.

We located the impactor and sensor under the furnace as visible in Fig. 3. It lowers the vertical flow of the hot air in the furnace (so called a chimney effect), so the temperature field in the furnace is more homogenous. A similar arrangement is applied in [18] where both, impactor and microphone, are located under the furnace. In the apparatus designed in [17], the impactor is under the furnace and the microphone is above it.

A voltage signal from the microphone is weak, for that reason the microphone was connected to an input of the preamplifier CanaKit UK 151 designed around the LM386 amplifier integrated circuit. The output of the preamplifier was connected to the headphone input of the computer.

4.2 Processing of the sound response

The operating software is responsible for four processes: a) controlling the repeating frequency of the measurement and a power of the impactor, b) recording the sample's sound response to the excitation, c) processing the data, and d) displaying the results. The software is developed under the environment of Matlab.

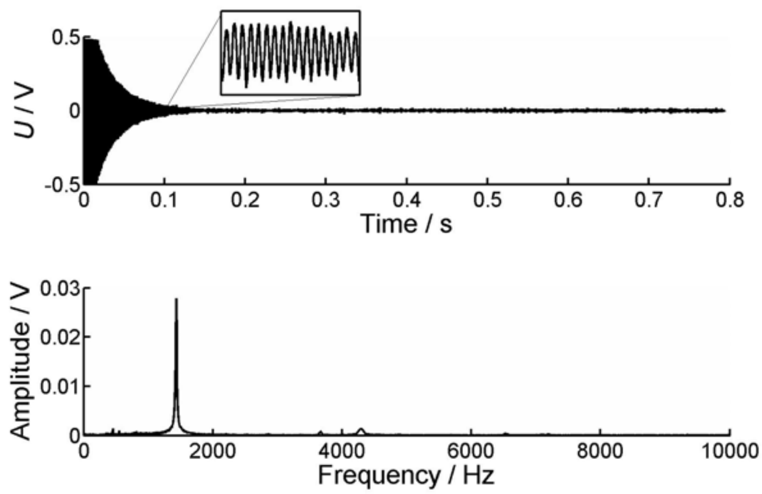

Fig. 6. A typical response of the sample to the mechanical impulse, where the output voltage of the microphone is visible in the upper picture and its frequency spectrum in the lower picture.

Simultaneously with the mechanical impulse, the program begins to record the sound response. We used a computer sound card for the data recording. It is possible to reach the sampling rate 40 kHz, which is sufficient for determination of the resonant frequency which varies from 700 Hz to 7000 Hz. The measured signal is stored in a time domain, displayed and in the next step is transformed into a frequency domain using fast Fourier transformation (FFT). The typical response of the sample to the mechanical impulse is shown in Fig. 6, where a frequency spectrum of the sound response is also shown. The fast Fourier algorithm is implemented in the Matlab as a function 'fft'. A logarithmic decrement, which characterizes internal damping, is found from the half-height width of the resonant peak [19].

5 The measurement example

The apparatus described above is used for dynamic thermomechanical analysis of ceramic samples. To present the capability of the apparatus to measure elastic and damping quantities of solid materials, we show one example in Fig. 7. Here are results obtained for the unfired ceramic sample (60 mass% of illite and 40 mass% of quartz) in a temperature regime 20 °C → 1100 °C → 50 °C, in which the heating and cooling rates were 1 °C/min. The measured results reflect the main processes in the sample: releasing the physically bound water, $\alpha \rightarrow \beta$ and $\beta \rightarrow \alpha$ transformation of quartz and sintering.

This example shows the ability of the apparatus to provide frequency and damping measurements in one experiment during heating/cooling. It can be seen, that damping measurement is accompanied with a scattering. Such a scattering is also often present in a commercial apparatus [17, 18]. The reason for this is in the high sensitivity of the internal damping measurement to the sample quality (inhomogeneities, cracks and pores).

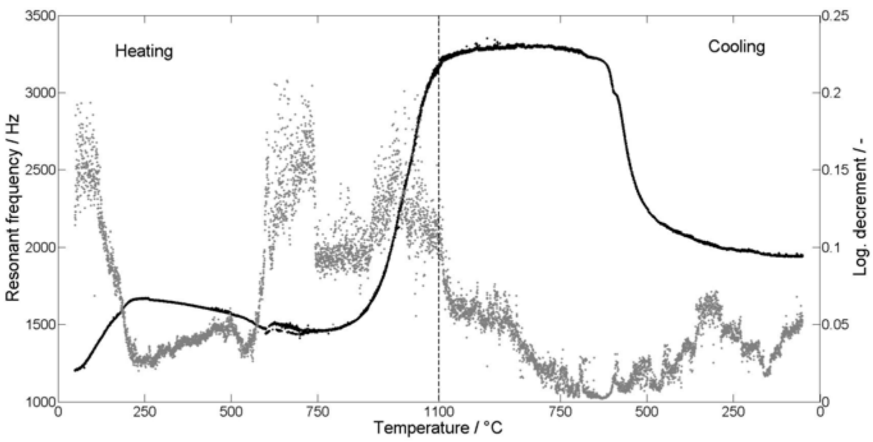

Fig. 7. The resonant frequency (black points) and logarithmic decrement (gray points).

6 Conclusions

A laboratory-made apparatus for the measurement of the resonant frequency and internal damping that exploits an impulse excitation technique (IET) is described. The apparatus is capable of measuring under a dynamic heating program or isothermal heating up to a temperature of 1200 °C. The expanded relative uncertainty of Young's modulus at elevated temperatures is 1.1 %. As an example of the measurements shows, the measurement of the resonant frequency is reliable. The internal damping measurement can be accompanied with quite a high scattering due to its strong dependence on defects in the sample.

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АППАРАТУРА ДЛЯ ИЗМЕРЕНИЯ МОДУЛЯ ЮНГА КЕРАМИКИ ПРИ ПОВЫШЕННЫХ ТЕМПЕРАТУРАХ

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Аннотация: В статье описана конструкция автоматизированной установки для динамического термомеханического анализа. Измерение основано на бесконтактном снимании микрофоном свободных изгибательных колебаний (цилиндрического или призматического) образца. Возбуждение колебаний проводится механическим импульсом от электромагнитного импульсора работающем в периодическом режиме с частотой 1 импульс в 15 или 30 с. Снимается зависимость амплитуды свободных колебаний образца от времени, которая переведётся с помощью трансформации Фурье на зависимость амплитуды колебаний от частоты, где определится резонансная частота и также коэффициент затухания по полуширине резонансного пика. Резонансная частота служит для расчёта модуля Юнга или скорости звука. Измерять эти величины могут в режиме линейного нагрева и охлаждения (скорость до 10 °С/мин) или в режиме изотермического нагрева. Максимальная рабочая температура установки 1200 °С. Расширенная относительная неопределённость при повышенных температурах 1,1 %.

Ключевые слова: резонансные измерения, модуль Юнга, импульсное возбуждение колебаний