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ELASTIC AND VISCOELASTIC PROPERTIES OF SYNTHETIC RUBBER (POLYCHLOROPRENE): DYNAMIC AND STATIC CHARACTERIZATION

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The evaluation of macroscopic elastic and viscoelastic properties of rubber-like polymer materials may be carried out experimentally by using accurate static and dynamic measurement techniques. However, the viscoelastic behavior of rubber-like polymeric materials involves different elastic and damping responses, as a function of particular mechanical stress applied during an experimental test. In this work experimental results of the elastic modulus and damping coefficients measured with static and dynamic methods are compared. The measurement techniques, performed in controlled temperature conditions, and the experimental data are accurately described.
Keywords: Elasticity, viscoelasticity, damping, rubber

1. Introduction

A detailed characterization of polymer materials is of importance in order to accurately address engineering applications and related performances. As it is well known, viscoelastic properties of polymeric materials, could influence the elastic response, not only as a function of temperature, but also as a function of large or fast deformations, due to different kind of applied stress. Mechanical properties, in terms of elastic and viscoelastic behavior, of two samples of polychloroprene rubber (neoprene), with different density, are investigated by means of four experimental techniques. Compressive and tensile behavior is investigated, in static and quasi static conditions and the elastic response to dynamic load is achieved, as well as the indentation modulus. Measurements are performed for comparative purpose only. Cited reference standards are used as a general guideline.

Neoprene rubber shows a typical viscoelastic response and has negligible plastic deformation at low strains; it is also resistant to abrasion, temperature fluctuations and burning (burn point is around 260°C) and glass transition temperature is -36 °C. Neoprene is widely used in engineering and industrial applications, as a gasket or corrosion-resistant coatings, since it is a versatile and resistant materials also in extreme environmental and working conditions.

2. Experimental methods

Static, quasi-static and dynamic measurements are performed to characterize the elastic and viscoelastic properties and the mechanical response of polychloroprene rubber samples stressed in different working conditions. The experiments are performed at the macroscale level by means of normal compression, uniaxial tensile test and dynamic test. Moreover, indentation modulus is evaluated on the basis of shore-A hardness test. In this paper the elastic responses of polychloroprene rubber samples, as a function of different experimental method, are compared.

2.1 Indentation modulus

A shore-A hardness durometer is used to measure the hardness of the rubber samples, as shown in Figure 1. Shore-A hardness values can be converted to elastic indentation modulus E_H using the following formula suggested by Kunz and Studer [1], for polymeric elastomers [2]:

$$E_H = \frac{1-\nu^2}{2r_d} \cdot \frac{0.549 + 0.07516 \cdot Sh_A}{0.025(100 - Sh_A)} \cdot (2.6 - 0.02 Sh_A). \tag{1}$$

where Sh_A is the Shore-A hardness, r_d is the tip radius of the durometer (0.395 mm) and it is assumed Poisson ratio ν=0.475.

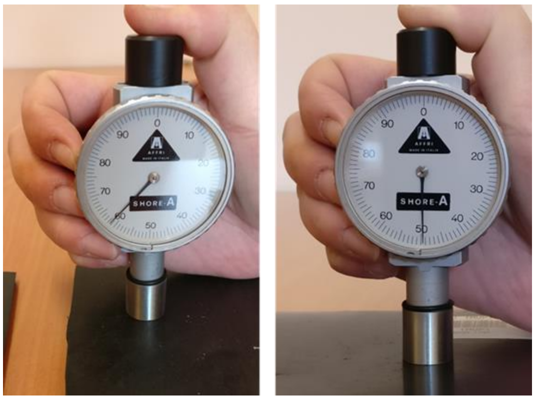

Figure 1: Measurement of shore-A hardness, by using a calibrated tester.

2.2 Pure Static Young's modulus

Following suggestions of ASTM D395-03 standard [3], the samples are subject to a static-loading force acting perpendicularly on the surface, as shown in Figure 2. As in typical Hooke's experiment, a series of static load are applied on the sample surface area and the corresponding thickness decreasing is measured. Thickness is measured by a digital comparator (resolution 0.5 μm) as a function of 7 increasing loads from 4 kPa up to 16 kPa. Compressive test allows to evaluate the pure static modulus from the fit of experimental data, on the basis of the following relation:

$$E_s = \frac{mg}{A} \cdot \frac{L_0}{dl}. \tag{2}$$

where m is the loading mass (kg), g is the gravity acceleration (m/s^2), A is the surface area (m^2) on which force is applied, L_0 is the initial thickness of the sample (m) dl is the static deflection (m).

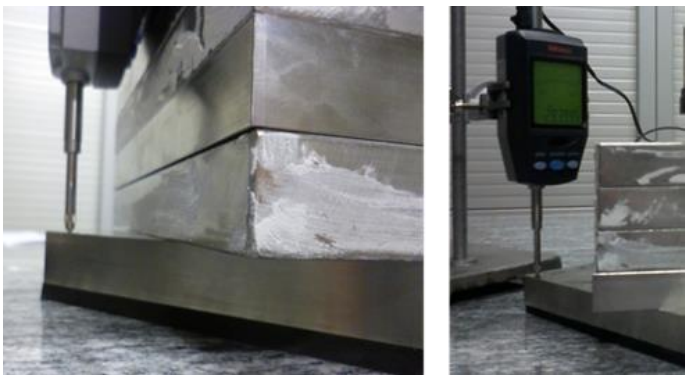

Figure 2: Measurement of thickness decreasing, as a function of static load.

2.3 Quasi-static Young's modulus

Stress-strain, from uniaxial tensile tests according to the ISO 527-1, ISO 527-2 and ASTM D 412 standard [4-6], on the rubber specimens are performed using a custom-made setup, as shown in Figure 3. In this setup, "bone-shaped" samples are pulled and force response is measured downstream by a load cell (resolution 5 mN). Samples are pulled from 0 to 3.5 mm (0 to 18% normal tensile strain) at 0.03 mms^{-1} of deformation rate ($4.3 \cdot 10^{-4}$ s^{-1} of strain rates). Occurring deformation is measured by a linear encoder (resolution 0.1 μm). As the material is viscoelastic, the strain-rate may affect the force response. Stress-strain test allows to evaluate the elastic tensile modulus by the best fit of experimental data in the linear region, on the basis of following relation:

$$E_{q-s} = \frac{F}{A} \cdot \frac{L_0}{dl} . \tag{3}$$

where F è is the measured tensile force (N), A is the surface area (m^2) on which F is applied, L_0 is the initial thickness of the sample (m) dl is the occurring deformation length (m).

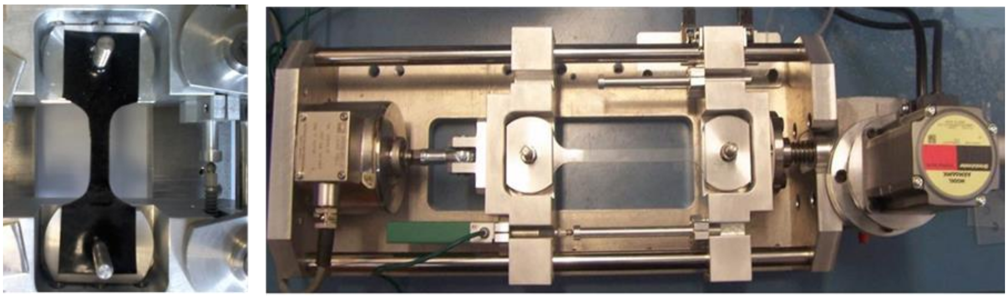

Figure 3: "Bone-shaped" sample and the measurement device.

Viscoelastic behavior is evaluated from relaxation time processes after deformation [7]. Relaxation time decay is calculated on the basis of the empirical Kohlrausch-Williams-Watts (KWW) time decay function:

$$\Phi(t) = \exp\left(-\frac{t}{\tau_0}\right)^{\gamma} . \tag{4}$$

where t is the time, τ_0 is the relaxation time at which $\Phi(t)$ decays to the value $1/e$ and the exponent γ describes the breadth of the distribution in the limits of $0 \le \gamma \le 1$. The relaxation time is directly related to the material damping properties. In general terms, as τ_0 decreases, the internal damping tends to increase, and *vice versa*. As a result, a greater aptitude to dissipate or absorbing energy of mechanical stress occurs.

2.4 Dynamic Young's modulus

Dynamic Young's modulus is determined on the basis of resonant frequency response of the loading mass-sample (mass-spring) system, subject to a vertical vibration, in Figure 4 the measuring set-up is shown. Reference Standard are ISO 9052-1 [8] and ISO 10846-3 [9], and literature [10, 11]. Samples are placed between an inertial base and the loading masses (from 16 kg up to 64 kg). The resonance frequency is determined by measuring the maximum acceleration level on the loading mass, at constant excitation force of about 50 mN. From angular resonance frequency ω_0 and the loading mass m on the surface are A it is possible to evaluate the dynamic Young's modulus from the following relation:

$$E_d = \frac{\omega_0^{\,2} \cdot m}{A \cdot \left(1 - \zeta^2\right)} \cdot \left(L_0 - dl\right) . \tag{5}$$

where ζ is the damping ratio, L_0 is the initial thickness of the material and dl is the static deflection due to different load applied. In particular damping ratio is determined from the width of the resonance peak, on the basis of half-power method:

$$\zeta = \frac{\omega_2 - \omega_1}{2\omega_0}. \tag{6}$$

where ω_1 and ω_2 are the values of frequency at -3dB (or √2 in amplitude) from the value of the resonance peak. From damping ratio ζ it is possible to evaluate the damping properties of the materials, since $\zeta = c/c_{crit}$, and, by definition, critical damping is $c_{crit} = \sqrt{mk} = \omega_0 \cdot m$.

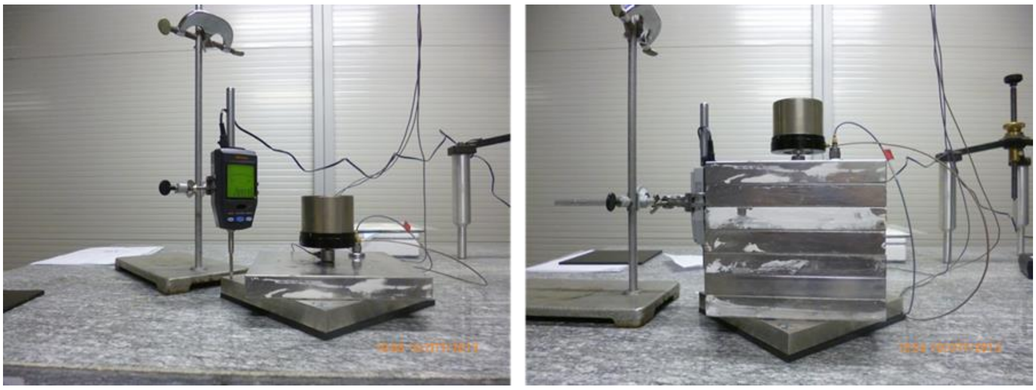

Figure 4: Measurement of dynamic Young's modulus, at 4 kPa and at 16 kPa.

3. Experimental results

Experimental tests are carried out in laboratory controlled environmental conditions, temperature is constant, 21.0±0.5 ℃ and fluctuation of relative humidity is within 10%, at 40% R.H.

3.1 Shore-A hardness test

A shore-A hardness is measured both on the core and on the surface of the samples, since rubber on the surface is lightly stiffer (or tough) than inside, due to working processes. Samples are provided in large sheet (30×30)cm and nominal thickness of 6.5 mm. In the pictures of Figure 5 it is possible to observe difference between surface and core of the samples.

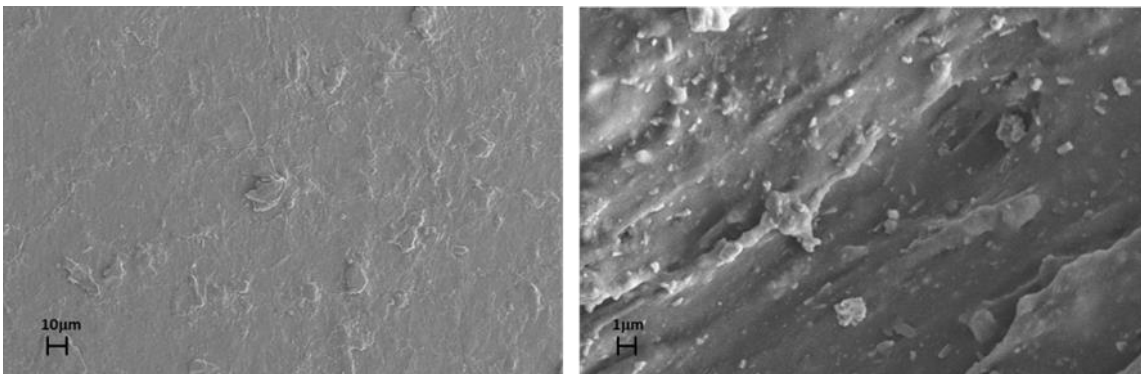

Figure 5: Neoprene sample surface and sample core.

Shore-A value measured on the surface and on the core are within the measurement uncertainty. Shore-A of sample A is 50±1, and shore-A of sample B is 66±1. On the basis of relation (1), the indentation modulus E_H of the rubber, is calculated, in MPa.

Sample A: E_H= 5.4 MPa

Sample B: E_H= 8.2 MPa

3.2 Uniaxial compression test

Samples are compressed under 7 static loads from 4 kPa up to 16 kPa (2 kPa steps). Thickness is measured on a rigid reference surface by mean a digital comparator. Samples are subject to a dead load of 2 kPa and the initial thickness L_0 is measured in this condition. The samples surface area is 400 cm^2. Each load is applied step by step and occurring thickness decreasing is measured after 2 hours, in order to avoid possible short-time creep effects [12]. In the graphs of Figure 6 the determination of static Young's modulus is shown.

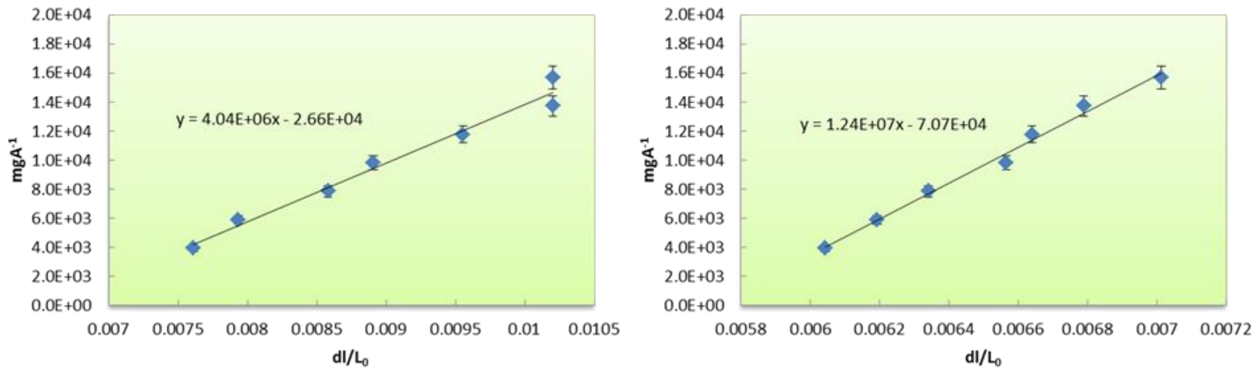

Figure 6: Experimental results of static Young's modulus on sample A and sample B.

Uniaxial compressive test allows to evaluate the elastic response of the samples as a function of static load and occurring deformations. The static elastic modulus E_s is calculated from relation (2).

Sample A: E_s= 4.0 MPa

Sample B: E_s= 12.4 MPa

3.3 Uniaxial tensile test

Samples are subjected to uniaxial tensile test. Initial thickness L_0 of both sample is 70 mm. Geometrical dimensions of sample A: 6,65 mm thickness, 9,70 mm width (64.50 mm^2 area); sample B: 6,43 mm thickness, 9,96 mm width (64.04 mm^2 area). The samples are fixed to the clamping system by means of two pins and fixed with a screw. It is important to underline that neoprene rubber is a strain-rate dependent material, due to viscoelasticity, as a consequence elastic response is stiffer for high strain-rate values and *vice versa*. In this work measurement are performed at constant strain rate of $4.3 \cdot 10^{-4}$ s^{-1} in order to evaluate quasi-static modulus at very low deformation rate. In the graphs of Figure 7 the experimental data of stress-strain, in the elastic (linear) region are shown, red line is the best-fit of experimental data. In the graph of Figure 8 the stress relaxation is depicted, dotted line is the KWW best fit.

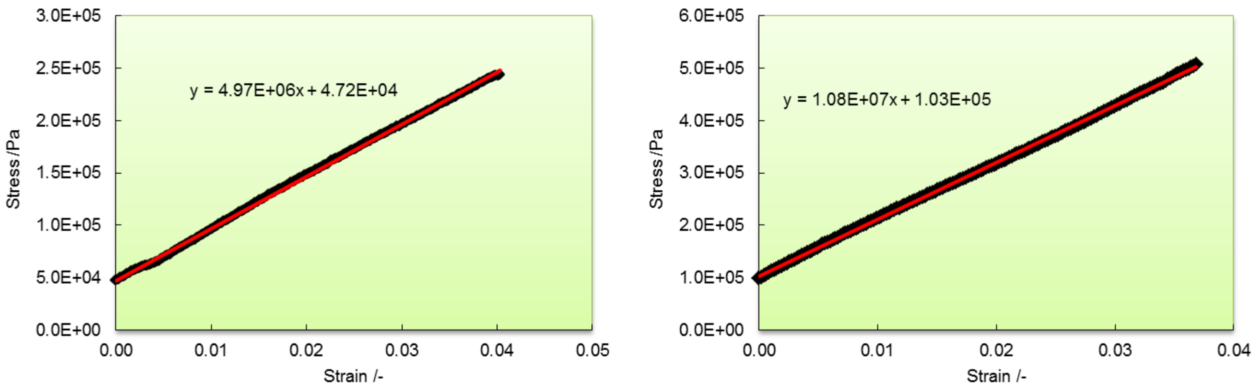

Figure 7: Experimental results of quasi-static Young's modulus on sample A and sample B.

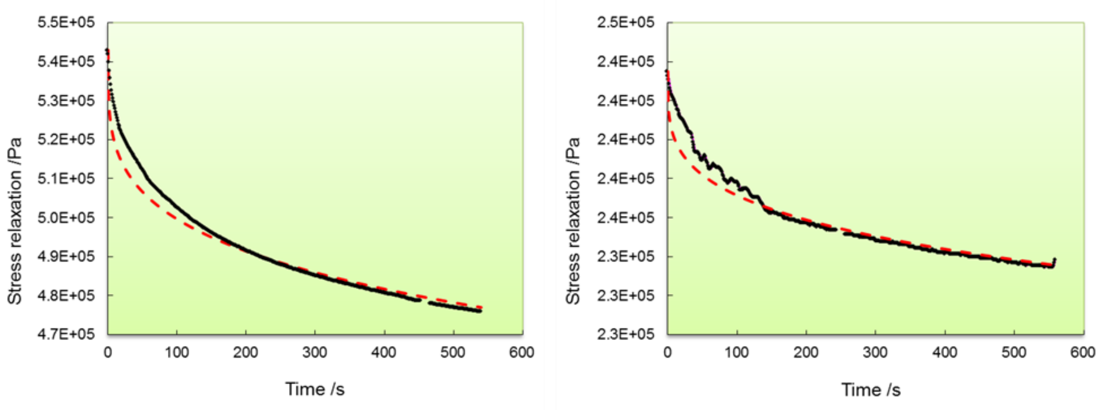

Figure 8: Stress-relaxation behavior on sample A and sample B.

Uniaxial tensile test allows to evaluate the elastic response of the samples as a function of quasi static applied tension and occurring deformations. Tension is applied at very low deformation rate. The quasi-static elastic modulus $E_{q\text{-}s}$ is calculated from relation (3). Viscoelastic response is determined from the relaxation time, measured from relation (4) from the best fit of experimental time-decay curve, as shown in Figure 8.

Sample A: $E_{q\text{-}s} = 4.9$ MPa, $\tau_0 = 9.6 \cdot 10^7$ s (with γ=0.26)

Sample B: $E_{q\text{-}s} = 10.8$ MPa, $\tau_0 = 1.3 \cdot 10^6$ s (with γ=0.26)

3.4 Resonant method

The resonant frequency response of the loading mass-sample (mass-spring) system is measured at 7 different load, from 4 kPa up to 16 kPa. Dynamic Young's modulus is determined from relation (5) and from the resonant peaks width the damping ratio is calculated from relation (6). In the graphs of Figure 9, the resonance peak and the dynamic Young's modulus, as a function of different load are shown.

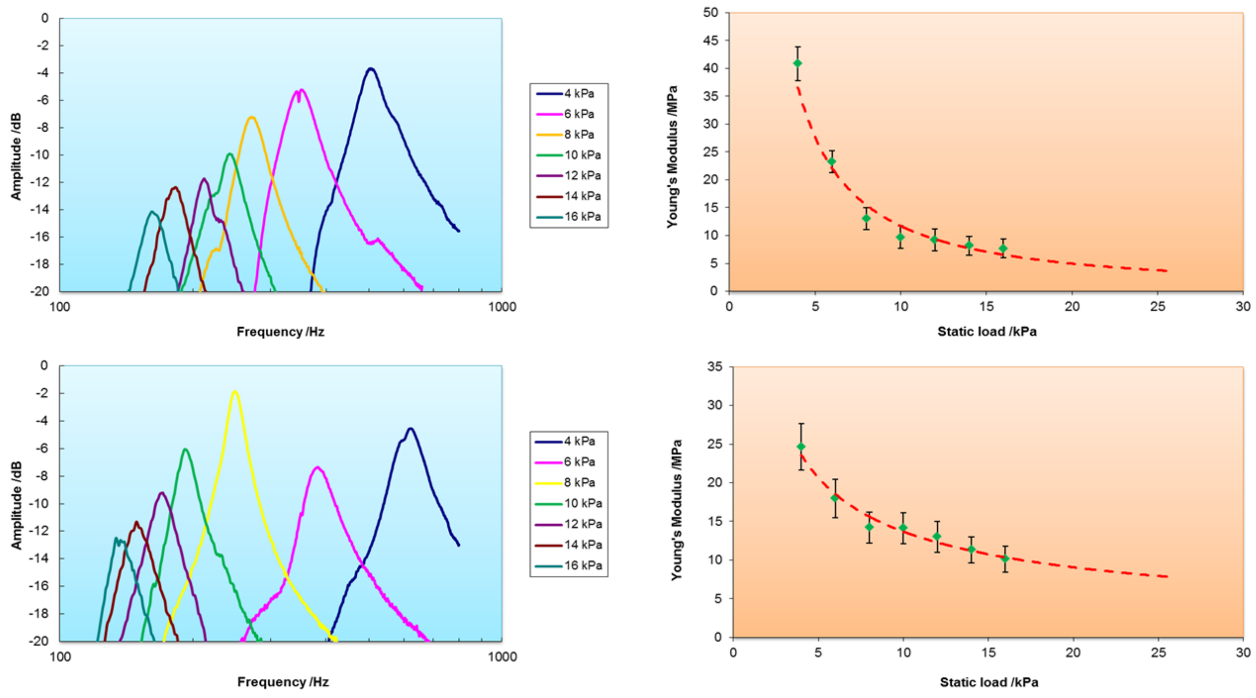

Figure 9: Neoprene sample surface and sample core.

As it is possible to notice, dynamic Young's modulus show a stress-dependence. In particular, at low stress, elastic response is stiffer, than at higher stress. As a matter of facts, as also observed in tensile test, below 10 kPa, the slope of the stress-strain curve is variable. From this measurements it is possible to verify that dynamic Young's modulus tends to be quite constant after 10 kPa, as a consequence dynamic modulus E_d is estimated from data interpolation. Viscoelastic response, in terms of damping ratio, critical damping and damping coefficient, is determined on the basis of relation (6).

Sample A: $E_d = 6$ MPa, $\zeta = 0.14$, $c_{crit} = 27.0 \cdot 10^5$ kg·s^{-1}, $c = 1.84 \cdot 10^5$ kg·s^{-1}

Sample B: $E_d = 12$ MPa, $\zeta = 0.15$, $c_{crit} = 29.5 \cdot 10^5$ kg·s^{-1}, $c = 2.18 \cdot 10^5$ kg·s^{-1}

4. Conclusions

In this paper the elastic response, in terms of static, quasi-static and dynamic measurements are performed to characterize the elastic and viscoelastic properties of polychloroprene rubber samples. The experiments are performed at the macroscale level by means of uniaxial compression test, uniaxial tensile test and dynamic test. Moreover the indentation modulus is evaluated on the basis of shore-A hardness test. In the following graph of Figure 10 the comparison of Young's modulus, determined on the basis of the above described techniques, is shown.

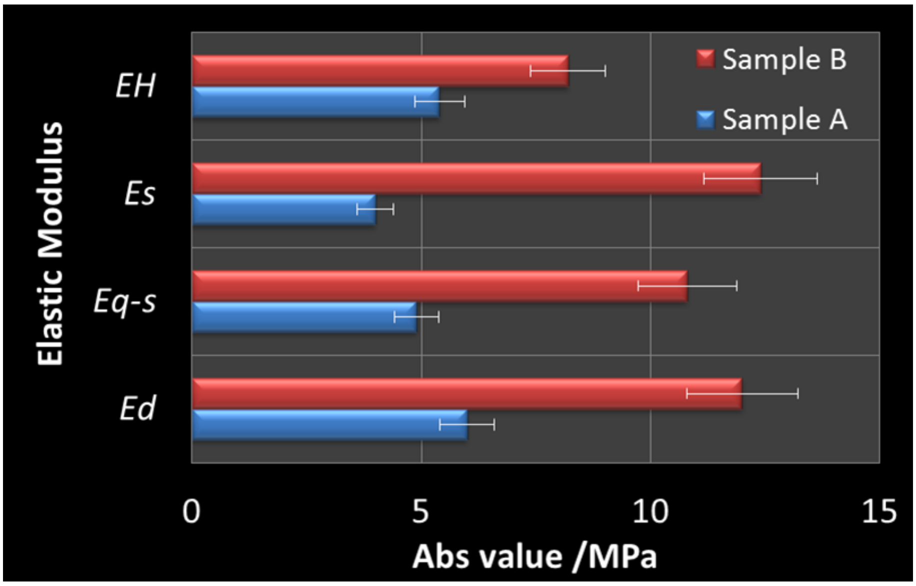

Figure 10: Comparison of Young's modulus measured through different experimental techniques.

As it is possible to notice, Young's modulus measured by using 4 different experimental techniques, gives compatible values. Data are expressed with a precautionary uncertainty of 10%. Some differences can be achieved for indentation modulus E_H, since it is properly the elastic response of the rubber samples when subjected to the action of a concentrated load in a single point. On the contrary, in both static (compressive and tensile) and dynamic experimental methods, the samples are subjected to the action of a distributed load on a surface area.

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