UNIVERSITA' DEGLI STUDI DI TRENTO
Department of Industrial Engineering

Laboratory activity – Properties and Characterisation of materials (module 1)

Mechanical properties evaluation of ceramic and polymeric materials in flexural and tensile configuration and quasi static condition.

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A.Y. 2020-2021

Contents

1 Introduction

In this laboratory important mechanical properties of both ceramic and polymeric materials were evaluated.
In the first part ceramic samples were considered, in particular alumina produced through sintering and slip casting at various temperatures and zirconia reinforced alumina also in this case sintered at different temperatures. The material is known to be have a brittle behaviour, so flexural tests were performed in order to obtain the flexural modulus and the stress and strain at break for the different samples.
In the second part bone cement was analyzed. This material today is widely used in the orthopedic field to fix prosthesis elements, like acetabular cups of hip replacements, tho the bone tissue. Also this material is known to be brittle since the main component are Poly(methyl methacrylate) (PMMA) and some filler materials, flexural test was performed and flexural modulus, stress and strain at break were evaluated.
In the third part a polymer was studied, Polypropylene. In this case the material behaviour is a ductile one, so it was possible to perform tensile tests at different cross-head speeds. The properties obtained are the elastic modulus, the stress and strain at yield. Moreover, through the Eyring model, the activation energy and volume were calculated.

2 Materials and methods

2.1 Flexural test

2.1.1 Description of the test

Flexural test is used in engineering mechanics to characterise the behaviour of a slender structural element subjected to an external load applied perpendicularly to a longitudinal axis of the element. This test is used to characterise polymers and brittle materials such as ceramics.
In the test, a specimen with rectangular cross-section is placed on two parallel supporting pins. The loading force is applied in the middle by means of loading pin. A flexure test produces tensile stress in the convex side of the specimen and compression stress in the concave side. This creates an area of shear stress along the midline. The flexural test can be carried out in 3 points bending configuration or 4 points bending configuration. A scheme of a 3 points bending configuration is shown in Figure 2.1.

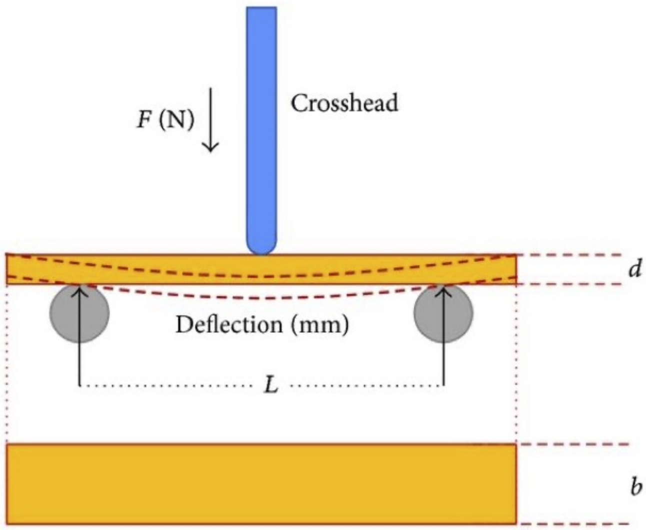

Figure 2.1: 3 points bending test scheme.

The 3 points bending test provides values for the modulus of elasticity in bending, flexural stress, flexural strain and the the flexural strain-stress response of the material. One of the most important advantage is the ease of the specimen preparation and testing, while a disadvantage is that the results of the testing method are sensitive to specimen and loading geometry and strain rate.
Electro-mechanical testing machine (Instron 5969) was used with a load cell of 50 kN.

2.1.2 Parameters of the test

The standard used in this laboratory activity for the flexural test is the ASTM D790[1]. According to the standard the rate of crosshead motion $[mm/min]$ can be calculated as:

$$R = \frac{Z\,L^2}{6d}, \tag{2.1}$$

where L is the support span, d is the depth of the beam and Z is the rate of straining of the outer fibre (it shall be equal to 0.01).
After the experiment, the collected data (that are related to extensive properties of the specimen) can be converted into intensive properties that are related to the material itself.
Always referring to the standard, it is possible to calculate the flexural strain $[mm/mm]$ (of the outer surface) as:

$$\varepsilon_f = \frac{6Dd}{L^2}, \tag{2.2}$$

where D is the maximum deflection of the center of the beam, L is the support span and d is the depth of the beam tested.
For what concerns the flexural stress in the outer fibres at midpoint $[MPa]$ it can be calculated as:

$$\sigma_f = \frac{3PL}{2bd^2}, \tag{2.3}$$

where P is the load at a given point on the load-deflection curve, L is the support span, b is the width of the beam tested and d is the depth of the beam tested.
Other two important parameters for the characterisation of the material are the tangent modulus of elasticity and the chord modulus. The first one can be calculated as:

$$E_B = \frac{L^3 m}{4bd^3}, \tag{2.4}$$

where L is the support span, b is the width of the beam tested, d is the the depth of the beam tested and m is the slope of the tangent to the initial straight-line portion of the load-deflection curve.
The chord modulus can be calculated when the curve is not properly linear. In this case a specific interval is selected in the strain axis and the slope of the curve is then calculated as:

$$E_f = (\sigma_{f2} - \sigma_{f1})/(\varepsilon_{f2} - \varepsilon_{f2}) \tag{2.5}$$

Of course, the besto choice of the interval $(\varepsilon_{f2} - \varepsilon_{f2})$ is where the curve is as linear as possible.

2.1.3 Toe compensation

Making reference to Figure 2.2, it is possible to observe that in a typical stress-strain curve there is a toe region (AC) that does not represent a property of the material. It is an artifact caused by a takeup of slack and alignment or seating of the specimen. In order to give the corrected zero point on the strain or extension axis this effect must be compensated according to the annex 1 of the standard ASTM D790[1].

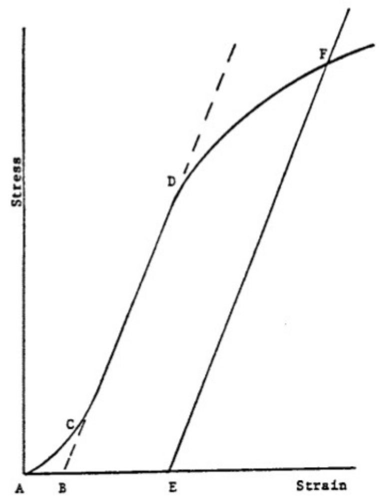

Figure 2.2: Material with Hookean region.

If the material exhibits a region of Hookean behaviour, like in Figure 2.2, a continuation of the linear region of the curve is constructed through the zero-stress axis and the interception is the corrected zero strain point from which all extensions or strains must be measured.
In the case of a material that does not exhibit any linear region, like in Figure 2.3, the same kind of toe correction of the zero-point can be made by constructing a tangent to the maximum slope at the inflection point.

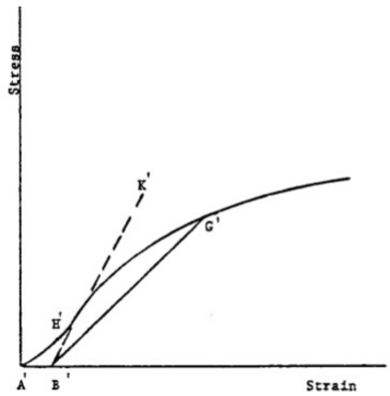

Figure 2.3: Material with no Hookean region.

2.2 Ceramic specimens

Alumina (or aluminum oxide) is the most well-known and most commonly used fine ceramic material. It has the same sintered crystal body as sapphire and ruby. It has been used for decades in electrical components for its high electrical insulation, and is widely used in mechanical parts for its high strength, and corrosion and wear resistance[2].
Zirconia can be added to alumina in order to improve its properties. Zirconia toughened alumina ceramics (also called ZTA ceramics) are special combination of aluminum oxide and $10 - 30\%$ of zirconium oxide; they provide advantages in terms of strength (higher than alumina), cost (lower cost than zirconia), fracture toughness, corrosion and erosion resistance[3]. All the information related to the groups and the samples are shown in Table 2.1.

Group	Material	Sintering temperature [°C]
1	Sintered Al_2O_3	1250 °C (specimen A, B, C)
1	Sintered Al_2O_3	1550 °C (specimen F, G, H)
2	Sintered $Al_2O_3 + ZrO_2$	1400 °C (specimen 1, 2, 3)
2	Sintered $Al_2O_3 + ZrO_2$	1600 °C (specimen 8, 9, 10)
3	Slip casted Al_2O_3	1500 °C

Table 2.1: Information related to material and sintering temperature of the specimens studied by each group.

The dimensions of of the cross-section of all the specimens (rectangular bars) were measured. Tables from 2.2 to 2.4 show the average dimensions obtained. The span length (L) was always 40 *mm* (out of standards).

Specimen	Width (W) [*mm*]	Thickness (d) [*mm*]
A	18.977 ± 0.002	5.07 ± 0.04
B	19.01 ± 0.01	5.10 ± 0.01
C	18.946 ± 0.007	5.06 ± 0.02
F	17.10 ± 0.05	4.60 ± 0.06
G	17.10 ± 0.03	4.71 ± 0.06
H	17.1 ± 0.1	4.9 ± 0.2

Table 2.2: Measurements of the specimens of group 1.

Specimen	Width (W) [*mm*]	Thickness (d) [*mm*]
1	17.06 ± 0.09	4.37 ± 0.08
2	17.0 ± 0.2	4.4 ± 0.1
3	16.9 ± 0.2	4.41 ± 0.07
8	16.73 ± 0.06	4.3 ± 0.1
9	16.7 ± 0.1	4.32 ± 0.04
10	16.74 ± 0.08	4.3 ± 0.1

Table 2.3: Measurements of the specimens of group 2.

Specimen	Width (W) [*mm*]	Thickness (d) [*mm*]
1	25.27 ± 0.09	9.37 ± 0.04
2	26.97 ± 0.08	8.3 ± 0.2
3	23.2 ± 0.3	9.6 ± 0.1

Table 2.4: Measurements of the specimens of group 3.

The rates of crosshead motion for the different groups are reported in Table 2.5 .

Group	R [mm/min]
1	0.5
2	0.5
3	0.3

Table 2.5: Rates of crosshead motion for each group.

2.3 Bone cement (PMMA) specimens

Bone cement is typically made of PMMA with some filler materials. PMMA is an amorphous thermoplastic polymer. It presents a glass transition temperature around 105 °C. Acrylic bone cement based on Poly-Methyl Methacrylate (PMMA) is used in orthopedics to fix both the femoral and acetabular components in a total hip replacement. Today, in most total joint replacements including the hip, knee, and ankle, acrylic bone cement is used as a means of fixation of the prosthesis to the bone. Bone cement is also often used in the fixation of pathological fractures and in the repair of bone defects[4]. Tested bone cement was Cemex®-Isoplastic provided by Tecres S.p.a.

For this part of the laboratory activity the span length was always 40 mm and the rate of crosshead movement was 0.5 mm/min.

Dimensions of the cross section of rectangular bars were measured, results are shown in Table 2.6.

Specimen	Width (W) [mm]	Thickness (d) [mm]
1	10.11 ± 0.02	4.1 ± 0.2
2	10.19 ± 0.28	4.45 ± 0.07
3	10.30 ± 0.07	4.5 ± 0.1

Table 2.6: Measurements of the specimens bone cement.

2.4 Tensile test

2.4.1 Description of the test

Tensile test is a form of tension testing and it is a destructive engineering and material science test whereby controlled tension is applied to a sample until it fully fails. It is one of the most common mechanical testing techniques and it is used to find out how strong a material is and also how much it can be stretched before it breaks. This test method is used to determine yield strength, ultimate tensile strength (UTS), ductility, strain hardening characteristics, Young's modulus and Poisson's ratio. An example of tensile testing configuration is shown in Figure 2.4.

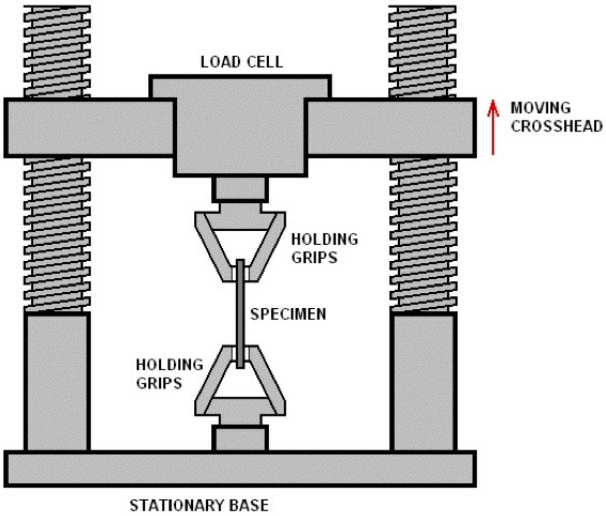

Figure 2.4: Tensile test scheme.

Tensile testing provides details of the tensile mechanical properties of the material. These properties can be plotted on a graph as a stress/strain curve to show details such as the point at which the material failed as well as providing details on properties like the elastic modulus, strain and yield strength.

For the test, specimens are prepared in a variety of ways depending on the test specifications. Dumbbell specimens obtained from injection moulding with a ISO527-1B shape were used, like that shown in Figure 2.5.

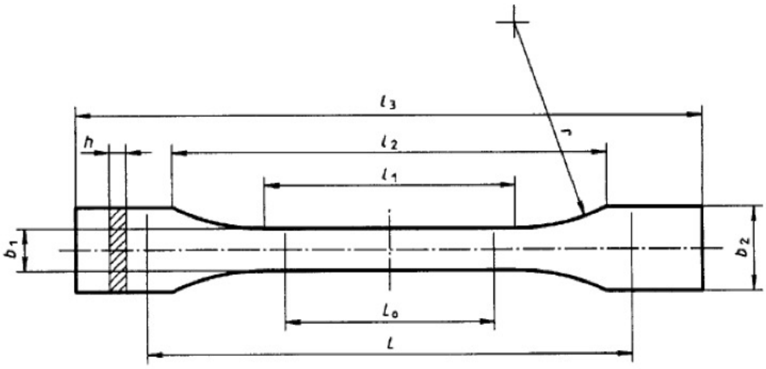

Figure 2.5: Dumbbell specimen scheme.

2.4.2 Parameters of the test

Calculation of the most important parameters of the tensile test was made according to the standard ISO 527[5].

For the calculation of the elastic modulus the rate of the cross-head motion was of 1 mm/min and a contact extensometer Instron 2620 with gauge length of 12.5 mm was used. In particular the modulus of elasticity can be defined as:

$$E = \frac{\sigma_2 - \sigma_1}{\varepsilon_2 - \varepsilon_1}, \tag{2.6}$$

where σ_1 is the stress in megapascal (MPa) measured at the strain value of $\varepsilon_1 = 0.0005$ and σ_2 is the stress in megapascal (MPa) measured at the strain value of $\varepsilon_2 = 0.0025$.

For what concerns the construction of the stress/strain curves, three different speeds of the crosshead were used. In particular they were 2.20 and 200 mm/min.
The stress and the strain were calculated respectively as:

$$\sigma = \frac{F}{A} \tag{2.7}$$

$$\varepsilon = \frac{\Delta L_0}{L_0}, \tag{2.8}$$

where F is the measured force concerned, in Newton, A is the initial cross-sectional area of the specimen, expressed in square millimetres [mm^2], L_0 is the gauge length of the test specimen, expressed in millimetres and ΔL_0 is the increase in the specimen length between the gauge marks, expressed in millimetres.
Only the first specimen was analysed with the aid of the extensometer, while the others were analysed through non-instrumented tensile test.

2.4.3 Eyring model

Polymers viscous flow activation energy is the minimum required energy for the flow unit (chain segment) to overcome the barrier and transit from the in-situ position to the nearby hole in the flow process and it is a sign of the sensitivity for the apparent shear viscosity to temperature. This parameter can be used to evaluate the difficulty on the material flow and its processing performances.
The Eyring model is an equation used in chemical kinetics to describe changes in the rate of a chemical reaction against temperature. In particular, the equation is:

$$\frac{\sigma_y}{T} = \frac{E_y^*}{V^*\,T} + \frac{k}{V^*}\,ln(\dot{\varepsilon}^*/\dot{\varepsilon}_0^*), \tag{2.9}$$

where k is the Boltzmann's constant, V^* is the activation volume and E_y^* the activation energy.

2.5 Polypropylene (PP) specimens

Polypropylene (PP) is a linear hydrocarbon thermoplastic polymer. PP, like polyethylene (HDPE, L/LLDPE) and polybutene (PB), is a polyolefin or saturated polymer. Polypropylene is one of those most versatile polymers available with applications, both as a plastic and as a fibre, in virtually all of the plastics end-use markets[6].
Dimensions of the cross section of the specimens were measured and the results are shown in Table 2.7.

Specimen	Width (W) [mm]	Thickness (d) [mm]
1	12.53 ± 0.02	3.22 ± 0.01
2	12.66 ± 0.04	3.267 ± 0.006
3	12.68 ± 0.04	3.267 ± 0.006
4	12.50 ± 0.05	3.24 ± 0.01

Table 2.7: Measurements of the specimens of PP.

3 Results and discussion

3.1 Flexural test on ceramics

Alumina is a ceramic material that presents a brittle behaviour not depending on the production method as it's possible to observe in Figure 3.1, Figure 3.2, Figure 3.3 where the stress-strain curves of sintered Al_2O_3 at 1250°C, 1550°C and slip casted Al_2O_3 at 1500°C respectively are reported. Moreover, in Table 3.1 and Table 3.2 the flexural moduli, the strains and stresses at break as well as the value average are presented. To evaluate the strain at break the toe compensation method was used.

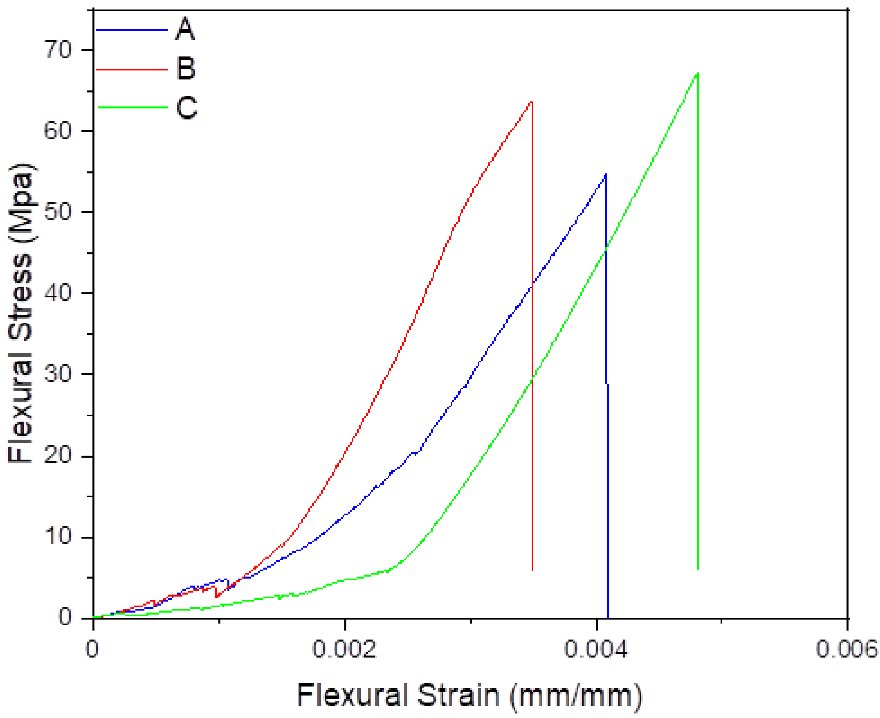

Figure 3.1: Stress-strain curves for 1250°C sintered AL_2O_3 specimens.

Specimen	E [MPa]	ε_U [mm/mm]	σ_U [MPa]
A	22715 ± 18	0.0024	54.77
B	32304 ± 19	0.0021	63.69
C	27498 ± 10	0.0024	67.14
Average	27505 ± 4794	0.0023 ± 0.0002	61.87 ± 6.39

Table 3.1: Flexural modulus, strain and stress at break for 1250°C sintered AL_2O_3 and calculated average value.

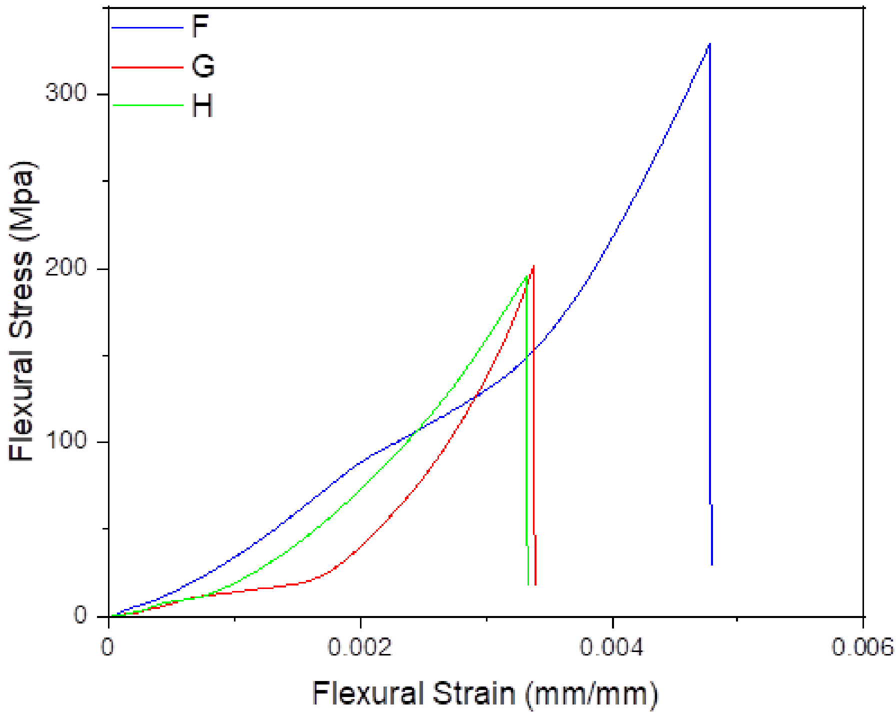

Figure 3.2: Stress-strain curves for 1550°C sintered AL_2O_3 specimens.

Specimen	E $[MPa]$	ε_U $[mm/mm]$	σ_U $[MPa]$
F	139030 ± 169	0.0024	329.00
G	142345 ± 304	0.0013	202.12
H	104108 ± 198	0.0018	195.27
Average	128494 ± 21184	0.0019 ± 0.0005	242.13 ± 75.30

Table 3.2: Flexural modulus, strain and stress at break for 1550°C sintered AL_2O_3 and calculated average value.

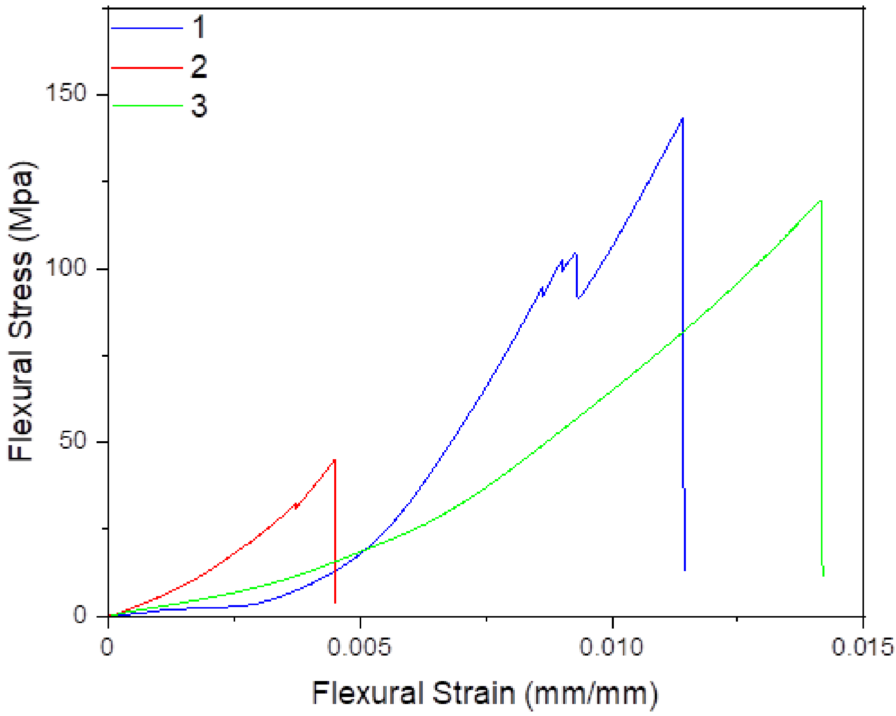

Figure 3.3: Stress-strain curves for 1500°C slip casted AL_2O_3 specimens.

Specimen	E $[MPa]$	ε_U $[mm/mm]$	σ_U $[MPa]$
1	25930 ± 18	0.0054	143.22
2	18015 ± 19	0.0025	45.05
3	12490 ± 10	0.0094	119.86
Average	18811 ± 6755	0.0057 ± 0.0034	102.70 ± 51.28

Table 3.3: Flexural modulus, strain and stress at break for 1500°C slip casted AL_2O_3 and calculated average value.

Just like the previous samples also the zirconia reinforced alumina is a brittle material as it's possible to observe Figure 3.4, Figure 3.5 where are presented the stress-strain curves sintered at 1400°C and 1600°C respectively. Reported in Table 3.4 and Table 3.5 are the Flexural moduli, strains and stresses at break for the different ceramic samples. Also in this case the toe compensation method was applied to obtain the strain at break.

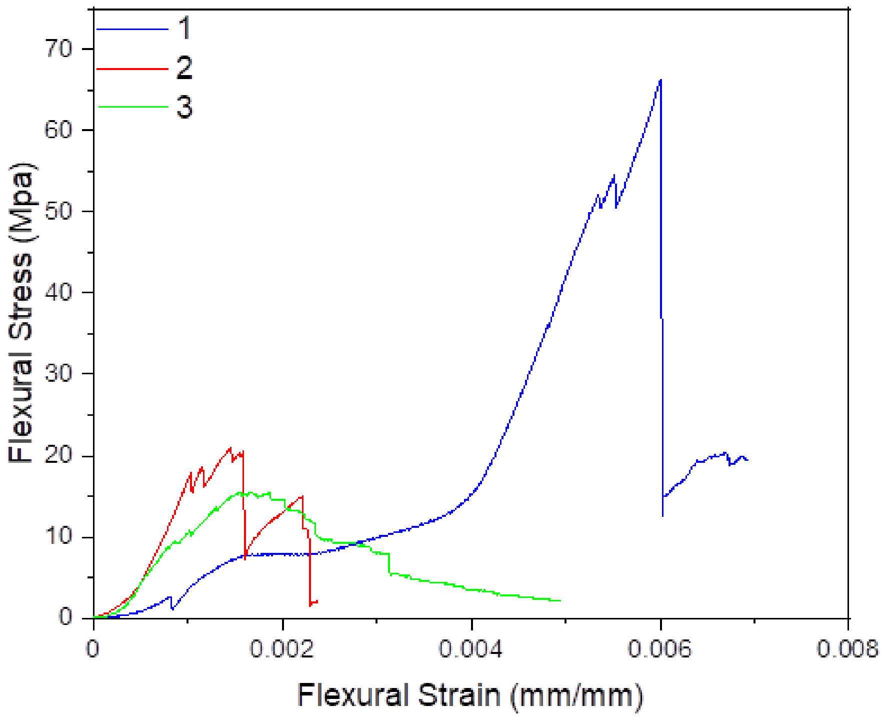

Figure 3.4: Stress-strain curves for 1400°C sintered ZrO_2 reinforced AL_2O_3 specimens.

Specimen	E $[MPa]$	ε_U $[mm/mm]$	σ_U $[MPa]$
1	29603 ± 39	0.0024	66.23
2	25746 ± 62	0.0011	21.04
3	12486 ± 56	0.0013	15.65
Average	22612 ± 8979	0.0016 ± 0.0007	34.30 ± 27.78

Table 3.4: Flexural modulus, strain and stress at break for 1400°C sintered ZrO_2 reinforced AL_2O_3 specimens and calculated average value.

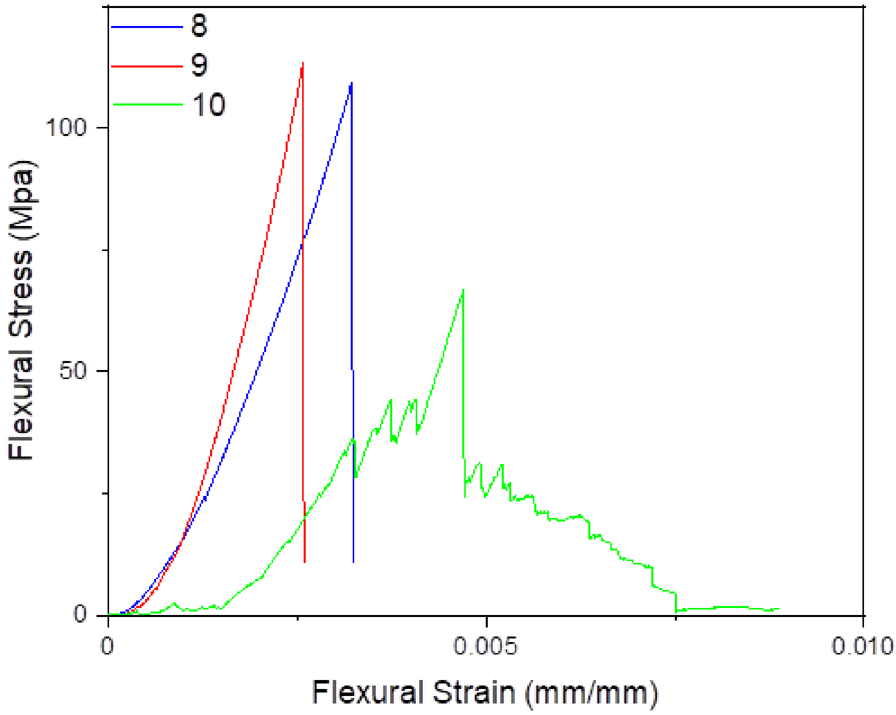

Figure 3.5: Stress-strain curves for 1600°C sintered ZrO_2 reinforced AL_2O_3 specimens.

Specimen	E $[MPa]$	ε_U $[mm/mm]$	σ_U $[MPa]$
8	44154 ± 3959	0.0024	109.54
9	65298 ± 94	0.0017	113.50
10	49588 ± 62	0.0014	66.94
Average	53013 ± 10980	0.0018 ± 0.0005	96.66 ± 25.82

Table 3.5: Flexural modulus, strain and stress at break for 1600°C sintered ZrO_2 reinforced AL_2O_3 specimens and calculated average value.

In Figure 3.6 are shown the average flexural moduli for the samples considered, while in Figure 3.7 and Figure 3.8 it's possible to observe the different average strains at break and stresses at break respectively.

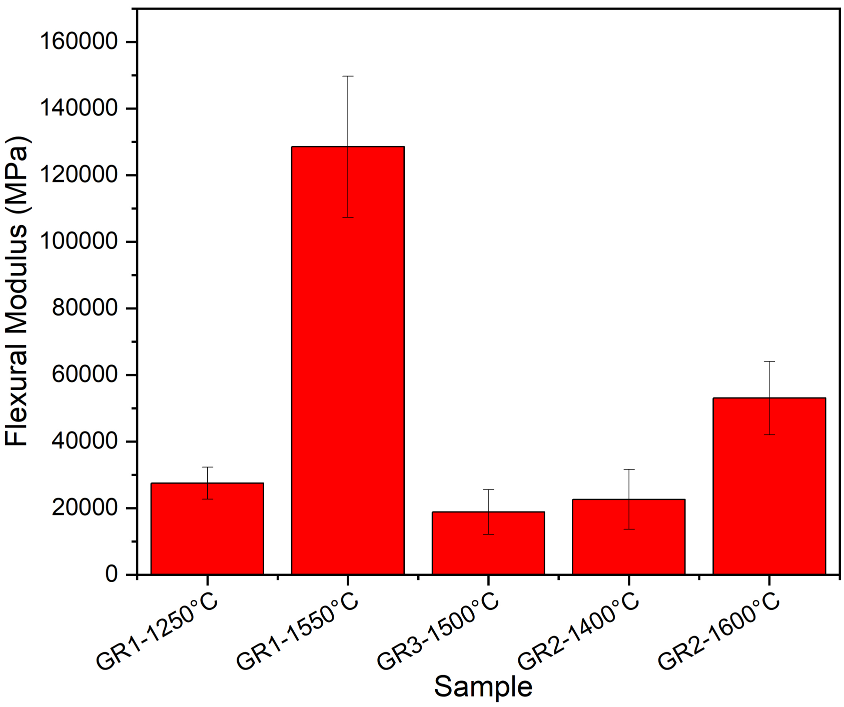

Figure 3.6: Flexural modulus for the different samples.

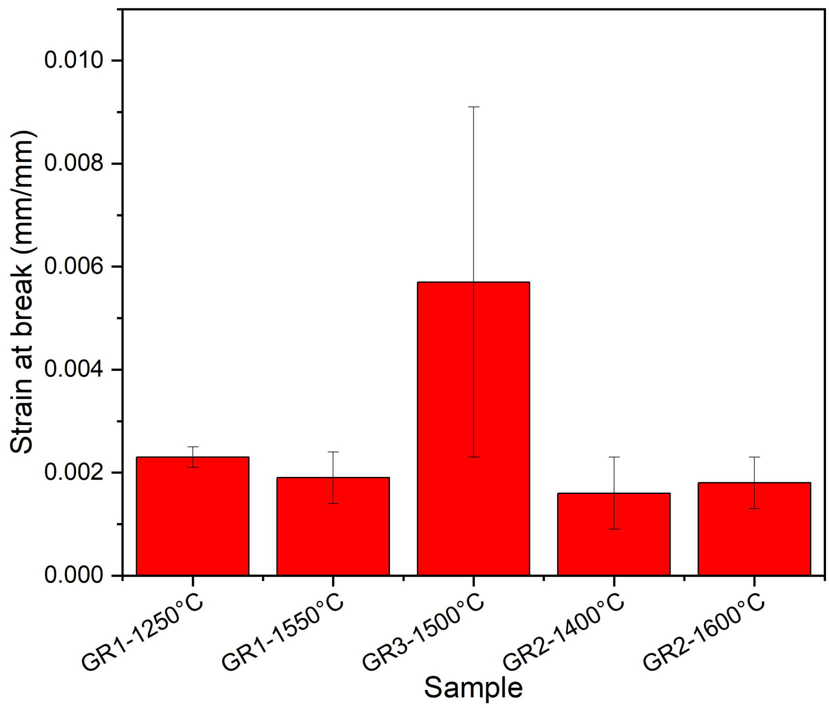

Figure 3.7: Strain at break for the different samples.

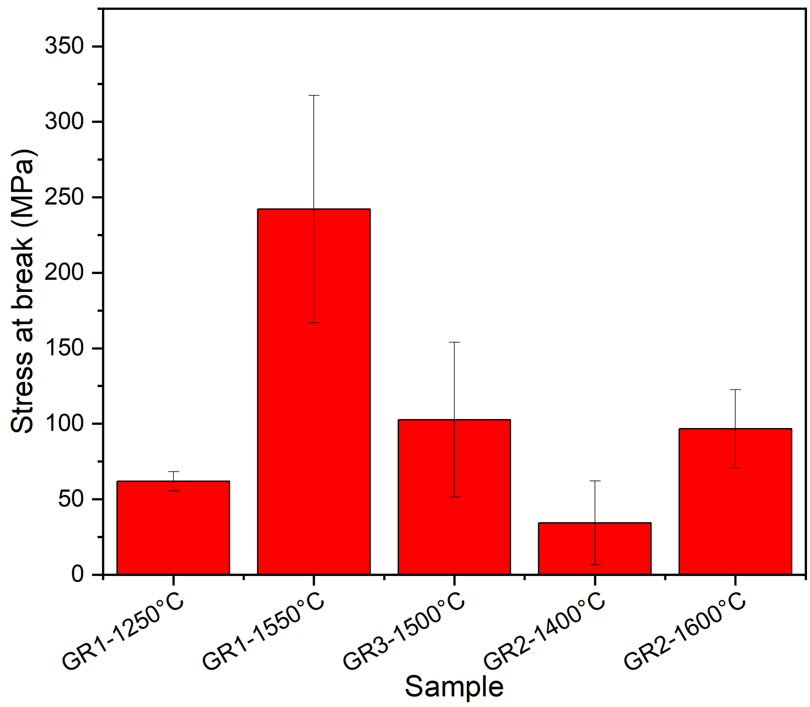

Figure 3.8: Stress at break for the different samples.

It's possible to observe how both the flexural modulus and the stress at break increase with the sintering temperature, whereas for the strain at break this pattern is not present as strongly as in the previous properties and this might be due to the toe compensation method applied to the evaluation on the strain. Also with the insert of reinforcements of ZrO_2 in the alumina it's possible to see a change in the tested properties although it was not expected such a difference in the flexural modulus. This discrepancy might be caused by different sintering temperatures and by the difficulties in the definition of a clearly linear region of the stress-strain curves for the evaluation of the flexural modulus.

3.2 Flexural test on Poly(methyl methacrylate) (PMMA)

Poly(methyl methacrylate) that mainly compose bone cement present a brittle behaviour, as it's possible to notice looking at the stress-strain curve of Figure 3.9, obtained through flexural tests. Indeed, after the elastic portion of the curve the sample undergo fracture without a plastic deformation.

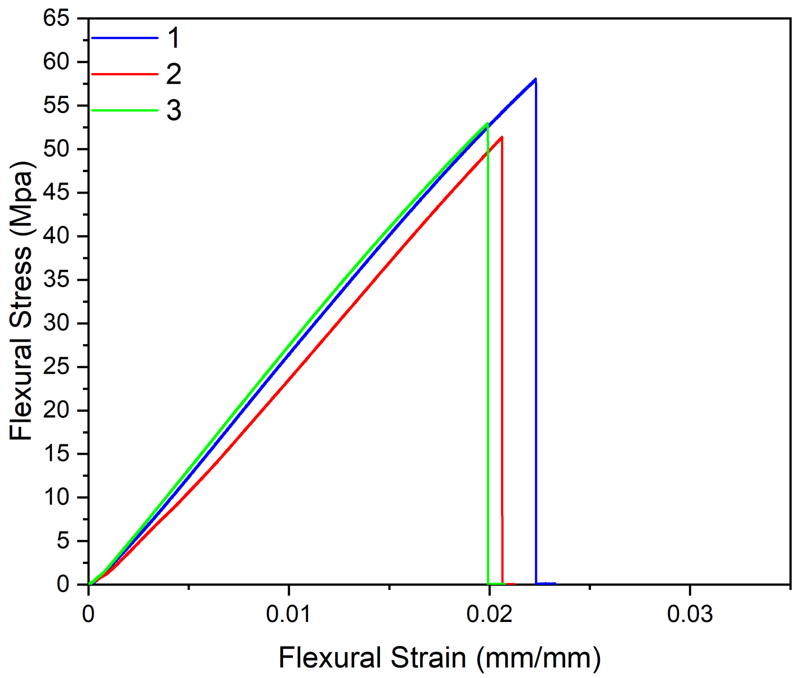

Figure 3.9: Flexural stress-strain curves on PMMA specimens.

From the stress-strain curves it was possible to evaluate the flexural modulus, the strains and stresses at break of the different specimens, values that are reported, also with the average in Table 3.6.

Specimen	E $[MPa]$	ε_U $[mm/mm]$	σ_U $[MPa]$
1	2773.3 ± 0.4	0.022	58.01
2	2672.7 ± 0.3	0.021	51.44
3	2778.1 ± 0.5	0.020	53.01
Average	2741.3 ± 59.5	0.021 ± 0.001	54.15 ± 3.43

Table 3.6: Flexural modulus, strain and stress at break of PMMA specimens and calculated average value.

In Figure 3.10 is presented a visual representation of the obtained flexural modulus with the average.

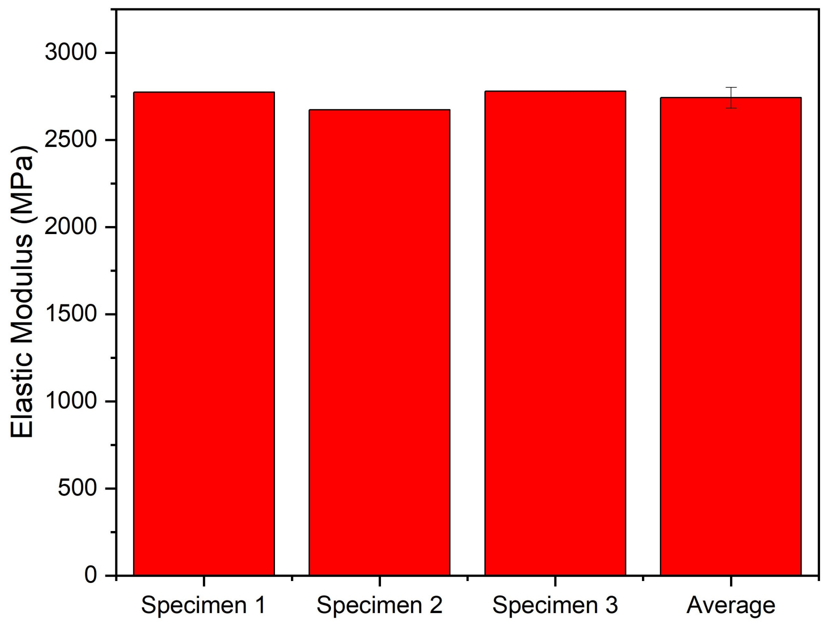

Figure 3.10: Flexural modulus of different specimens of PMMA and average.

As previously explained the bone cement is, for now, the main way to fix orthopedic prosthesis to the bone in order to avoid the unwanted movement of the device that cause pain in the patient. However the high flexural stress can cause the phenomenon of stress shielding, leading to the resorption of the bone making the tissue more fragile.

3.3 Tensile test on polypropylene (PP)

As previously described Polypropylene is a thermoplastic polymer that, contrarily to the materials since here considered, shows a ductile behaviour. This type of behaviour is reflected on the stress-strain curves obtained through three tensile tests that present different cross-head speeds, $\dot{\varepsilon}$, presented in Figure 3.11.

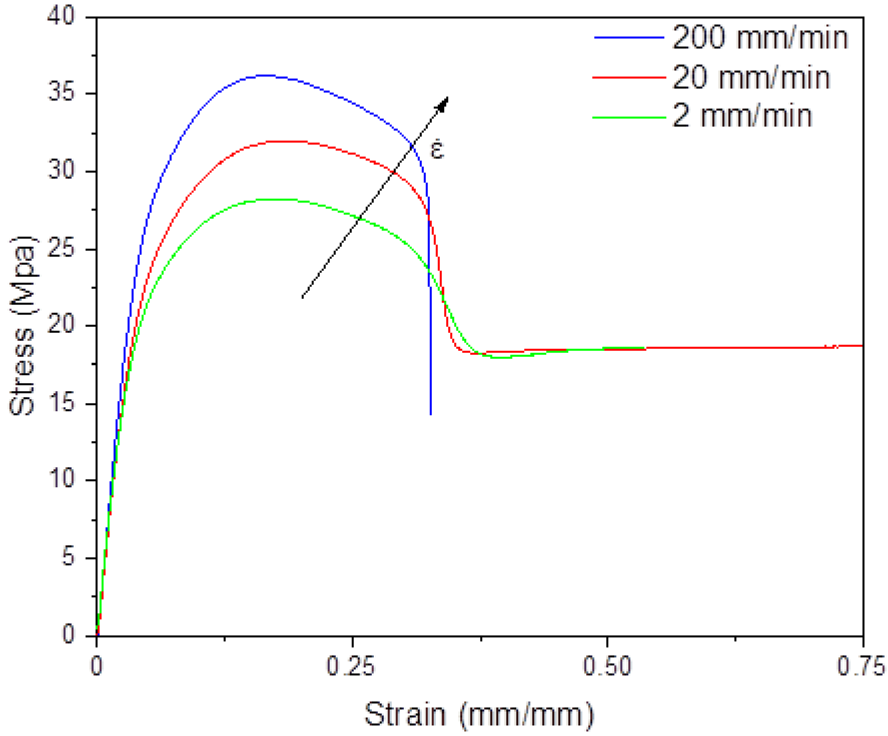

Figure 3.11: Tensile stress-strain curves on PP specimens with different cross-head speeds.

Following standard ISO 527[5] the values of σ_1 and σ_2 were calculated and are reported in Table 3.7. It was possible to evaluate the elastic modulus for the different considered cases whose values are shown in Figure 3.12. Also in this case it's possible to observe how, as the cross-head speed increments, also the elastic modulus increases.

Cross-head speed $[mm/s]$	ε_1 $[mm/mm]$	σ_1 $[MPa]$	ε_2 $[mm/mm]$	σ_2 $[MPa]$
200	0.0005406	0.1976	0.002469	1.312322
20	0.000532	0.452093	0.0025884	1.0025884
2	0.0005036	0.822824	0.0025012	1.597999

Table 3.7: Strains and Stresses for the evaluation of the elastic modulus of PP.

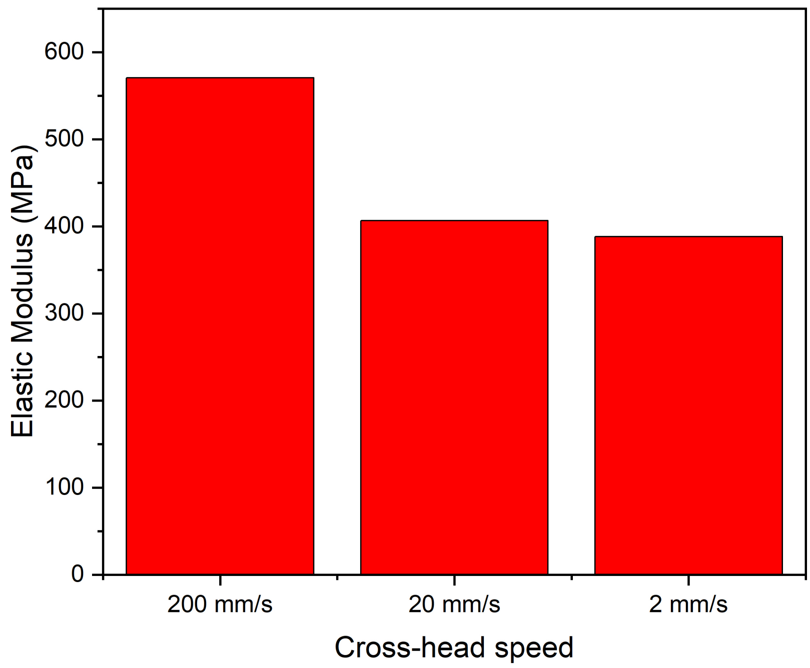

Figure 3.12: Elastic modulus of PP specimens with different cross-head speeds.

In Table 3.8 the yielding strain and stresses are reported, values that were obtained from the stress-strain curves of Figure 3.11 .

Cross-head speed $[mm/s]$	ε_Y $[mm/mm]$	σ_Y $[MPa]$
200	0.1639	36.20
20	0.1849	32.00
2	0.1756	28.23

Table 3.8: PP yielding strain and stresses.

Known the yielding stress and strain values the activation energy and the activation volume of Polypropylene were calculated and values are shown in Table 3.9. Values similar to the obtained ones can be found in the literature for Polypropylene at temperature lower that $110°C$[7]. To obtain the activation energy and volume the Eyring model was used and the line obtained through the linear fitting of the experimental values is reported in Figure 3.13.

Activation Energy $[KJ/mol]$	Activation Volume $[m^3/mol]$
$1.42 \cdot 10^{-3}$	47.21

Table 3.9: PP activation energy and activation volume.

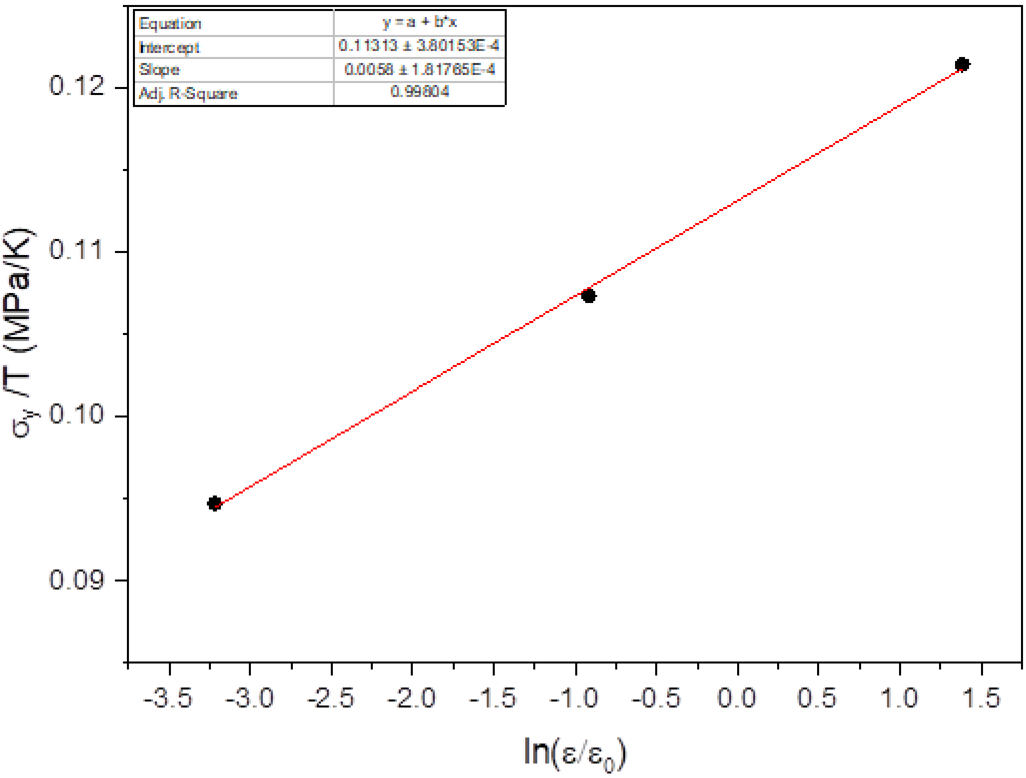

Figure 3.13: PP Eyring model.

4 Conclusions

The aim of this laboratory activity was to evaluate fundamental mechanical properties of ceramic and polymeric samples through flexural and tensile tests according to the known behaviour of the material.

In the first part samples of alumina were analyzed whose differences were the production method, the sintering temperature and, for the reinforced alumina, also the chemical composition. What was observed was an increase of the considered mechanical properties with the increase of the sintering temperature, but this trend was not well displayed for the strain at break probably due to the toe compensation applied to estimate this values.

In the second part of the laboratory bone cement was considered. In this case it was seen the the average flexural modulus was 2741.3 ± 59.5 MPa and this, for a patient with a prosthesis anchored to the bone, can lead in the long run to bone resorption and so fragility of the tissue.

In the third part another polymer was considered, Polypropylene. This material, differently from the others considered is a ductile material, so it was possible to evaluate the strain and stress at yield and so the elastic modulus was evaluated. It was possible to see how increasing the cross-head speed there's also an increment of the elastic modulus and of the yielding stress but not of the yielding strain were the highest value of 0.1848 mm/mm is reached at 20 $\mathrm{mm/s}$. Furthermore the activation energy and the activation volume were calculated of $1.42 \cdot 10^{-3}$ $\mathrm{KJ/mol}$ and 47.21 $\mathrm{m^3/mol}$ respectively.

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