MECHANICAL CHARACTERIZATION OF GLASS/EPOXY COMPOSITE MATERIAL WITH NANOCLAYS

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Abstract. *The numerical simulation of composite structures under impact loads hinges on the knowledge of the material response to dynamic loads. In this work, a detailed quasi-static and dynamic material characterization is presented. The material is a S2-glass/epoxy laminate composite, where montmorillonite nanoclays, with concentration of 0%, 5% and 10% in weight, were added. The quasi-static tests were run in a standard tensile test machine, according to ASTM standards. The dynamic compression tests used the split pressure Hopkinson bar (SPHB), so that a wide range of strain rates, between* $10^{-2}s^{-1}$ *and* $10^{-3}s^{-1}$*, was achieved. It was noticed that the mechanical behavior of the composite is sensitive to the strain rate, but not to the concentration of nanoclays or fraction of fibers.*

Keywords*: composites, nanocomposites, impact, characterization, SPHB.*

1. INTRODUCTION

The use of composite materials has grown significantly in civil construction and aerospace, naval and automotive industry, substituting traditional materials, as steel and aluminum. In many situations, this type of material is subject to impact loads, as boat hooves impacted against the ground, shipyards and rocks; or aircraft's leading edge, subjected to the impact of birds or small debris launched off the ground during landings and takes-off. Thus, the study of the behavior of this type of material under low (10^{-4} s^{-1} to 10^{-1} s^{-1}) and high strain rates (10^{1} s^{-1} to 10^{3} s^{-1}) is of fundamental importance.

Studies devoted to high strain rate loadings in metallic materials have been carried through and published in the literature, however similar studies with composites, such as carbon or fiber glass, are comparatively rarer. The Split Pressure Hopkinson bar (SPHB) has made possible the dynamic characterization of materials, being capable to generate strain rates at compression, tensile, torsion and shear loading states. However, only in the last past decade the first works regarding the use of this equipment for the ceramics and laminate composites characterization had been presented, but many presented results are not coincident or even divergent.

The work of Honsur *et al.* (2001) presented the characterization of a carbon fiber/epoxy resin under compressive loads and moderate strain rates using the SPHB. According to the authors, the material is strain rate sensitive and different behavior of the matrix was observed at different strain rates. In Navarro *et al.* (2005), this same dependence was also verified when considering tensile loadings. Waas *et al.* (1997) carried out compressive dynamic studies in fiberglass/epoxy resin laminates and observed that the maximum stress under dynamic loads is about 1.7 times greater than the corresponding quasi-static regime. Also, using SPHB, Sun and Tsai (2002) performed diverse dynamic and quasi-static tests, with two different specimen configurations, aiming to develop a constituent law for a fiberglass/epoxy resin laminates. The main conclusions of this work were the rate sensitive character of the material and the influence of specimen configuration on the results. Sun and Weeks (1998) presented a fiber glass characterization through a modified SHPB to allow tensile tests, having observed a linear material behavior when the loading is aligned with the fibers and a non linear behavior when off axis loading is employed, such difference being due to matrix effects.

In the great majority of the works, the type of fiber configuration used is unidirectional, that is, it has only one main direction in each layer. In the present work, however, a dynamic characterization of a composite laminate with epoxy resin will be carried out in a bidirectional fiber glass of balanced plain wave type, presenting the same fiber fraction in two perpendicular directions in the same layer of the laminate. Moreover, the available material for study contains two different fiber concentrations, 65% and 52%, and addition of montmorillonite nanoclays (10%, 5% and 0% in weight of the matrix). Manufacturing aspects of this kind of composite can be found in the works of Haque e*t al.* (2003) and Yasmin *et al.* (2003). The characterization of modified epoxy/montimorillonite nanocomposites was presented in the work of Yilmazer *et al.* (2003). Ávila *et al.* (2005) presented low-velocity impact tests with the same kind of material, concluding that impact resistance capacity is increased as nanoclays concentration increases until the limit of 5%, from which a reduction of that property is verified.

This work presents the mechanical characterization of the described glass/epoxy composite under the quasi-static and dynamic regime by using the SPHB, aiming to analyze the changes in its behavior with variations of the strain rate, fiber fraction and nanoclays concentration, thus getting parameters of the material behavior for future numerical simulations of phenomenon of impact against such materials.

2. COMPOSITE DESCRIPTION

A S2-glass/epoxy composite laminates with added nanoclays, manufactured and furnished by the Mechanics of Composites Laboratory of Federal University of Minas Gerais (UFMG), is studied. The initial material presentation was rectangular plates of size 350 x 350 mm^2 and a thickness of 4 mm.

The studied fiber configuration is a plain weave balanced type, ie formed by equal fractions of fibers in the longitudinal and transversal directions at each layer. Its superficial density is 200 g/m^2, and was it manufactured by TEXGLASS. The epoxy resin is constituted by ARALDITE M® and HY 956 hardener, with specific mass of 1,1g/m^3.

The added nanoclays are composed by montmorillonite from NANOCOR I 30E manufacturer with 0%, 5% and 10% fraction in weight of matrix. The composite laminates were assembled with 24 and 32 layers, corresponding to fiber fractions of 52% and 65% in weight, respectively. Table 1, shown below, summarizes the different kinds of laminates employed in this study:

Table 1: Amount of laminates available for the tests.

	0% of nanoclays	5% of nanoclays	10% of nanoclays
24 layers (52% in weight)	3	3	3
32 layers (65% in weight)	1	1	1

The following nomenclature will be adopted in this study to identify the specimens: a label “Fg”, denoting the fiber glass composite type, followed by two numbers referring to nanoclays concentration. Thus, 00, 05 and 10 correspond, respectively, to 0%, 5% e 10% of nanoclays addition. If necessary, two additional digits were used to distinguish the number of layers of the laminate, Tab. 2. Due to reduced number of specimens and difficulties to obtain a greater quantity of samples, in some tests it was not possible to carry out a statistical analysis or normalize the experiments.

Table 2: Material identification nomenclature.

Identification	Material	
	Nanoclays concentration	Number of layers
Fg00	0%	-
Fg05	5%	-
Fg10	10%	-
Fg00-32	0%	32
Fg05-32	5%	32
Fg10-32	10%	32
Fg00-24	0%	24
Fg05-24	5%	24
Fg10-24	10%	24

The plates are resulting of a second generation process of composite manufacture with nanoclays, which does not present the stage of heating of the material. The process of manufacture of these plates, detailed in the work of Ávila *et al.* (2005), comprises the stages to follow:

1. The nanoclay is mixed to acetone;
2. The solution is added to the hardener ou resin, forming a homogeneous emulsion;

3. This emulsion is degassing for two hours, to eliminate de bubbles and the acetone;
4. The hardener with only the nanoclays is added to the other part of mixture, formin de epoxy resin;
5. The resin impregnate to the fiber glass;
6. The layers are joined, building the laminate;
7. Is made the first cure at the vacuun and ambient temperature;
8. The pos-cure is made at temperature of 60° along 4 hours.

A visual inspection is made through the back-light technique to verify the existence of voids. Previous works show that the vacuum cure technique leads to small voids formation. To manufacture the coupons, a water-cut machine and diamonded tools were used. Such a procedure prevents the heating of the material that would harm the cohesion between the layers of the laminate.

3. CHARACTERIZATION METHODS

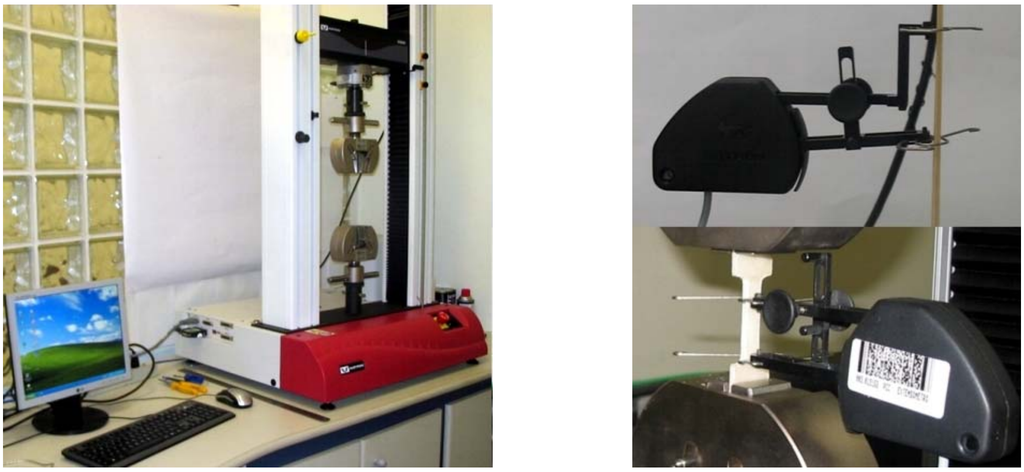
Two sets of material mechanical characterization tests were executed:

- Compression and tension quasi-static tests;
- High strain-rate compression tests by using a modified SPHB.

The methodology used for the experiments specified above is now presented.

3.1 Quasi-static tests

These tests were carried out by using a Instron machine, model 3369, with static strain-gages models 2630-106, (25 mm in length) or model 2630-112 (50 mm in length), as presented in Fig. 1.

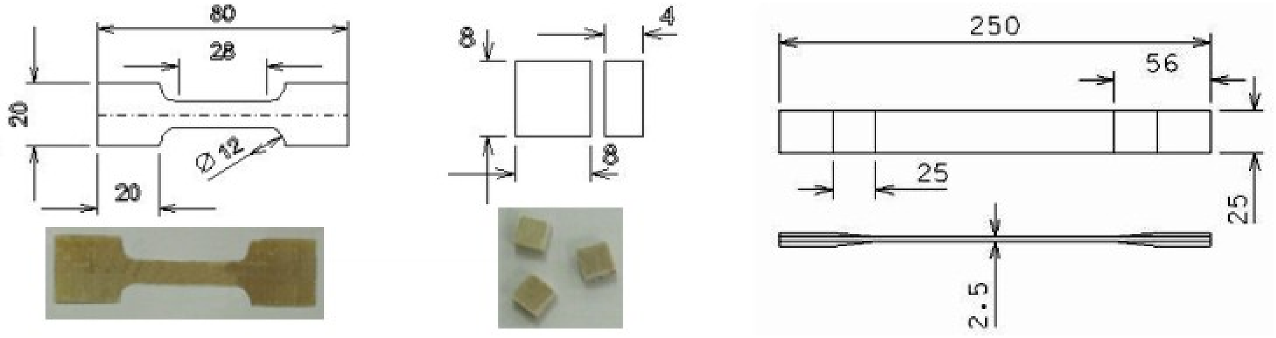


(a) (b)

Figure 1: (a) Instron machine for quasi-static characterization; (b) Detailed view of strain-gages.

The specimen dimensions for the tension tests of the 32-layers laminates were based in the work of Sun and Tsai (2002), adopting the shape usually employed to metallic material tests, namely the dog-bone, with a thickness reduction of 8 mm, 4 mm in thickness and 80 mm in length. Note that the length was reduced due the amount of available material to the study. In this work such geometry will be called ‘Configuration A’. For each sample the manufacture of 2 types of test specimens for tension was possible: namely [0°, 90] and [- 45°, +45°] configurations. The procedure used for tests was based on the standard ASTM-D3039 and the geometry of such prismatic specimens was 250 mm in length, 25 mm in width and 2.5 mm in thickness. Tabs for setting of test machine mordents were added and the resulting state loading was [0°, 90°] referenced to fiber direction; this geometry will be called ‘Configuration B’. The strain rate range to these tests was 10^{-3} s^{-1} to 10^{-1} s^{-1}, justifying the quasi-static situation hypothesis.

The compression test specimens were prismatic (8x8x4 mm^3), with compression loading being applied perpendicularly to laminas. In Fig. 2 geometrical definitions of ‘Configuration A’, ‘Configuration B’ type and compression specimens are presented, whereas Tab. 3 summarizes the amount of specimens employed in the different tests.



(a) (b) (c)

Figure 2: Geometry description of test specimens: (a) ‘Configuration A’, (b) compression tests prism and (c) ‘Configuration B’. Dimensions are given in mm.

Table 3: Amount of diferent test specimens.

Configuration	Amount								
	Fg00-32	Fg05-32	Fg10-32	Fg00-24	Fg05-24	Fg10-24	Fg00[1]	Fg05[1]	Fg10[1]
Tension – ‘Configuration A’	4	4	4	0	0	0	0	0	0
Tension – ‘Configuration B’	0	0	0	0	0	0	5	5	5
Compression	30	30	30	30	30	30	0	0	0

(1): In accordance with ASTM-D3039.

3.2 Dynamic tests

The dynamic tests were carried through the modified Split Pressure Hopkinson Bar (SPHB) developed by the Group of Solid Mechanics and Structural Impact of University of São Paulo, dicribed in Gallina *et al.* (2003) and Micheli *et al.* (2004). The experimental apparatus is formed by two rigid bars, a striker and the specimen to be tested, placed between the bars, as depicted in Fig. 3. An elastic compression pulse is generated in the input bar by the shock of the striker, pneumatically accelerated, and travels along this bar. When the pulse arrives at specimen-bar interface part of it is transmitted through the specimen, whereas another part is reflected. The material characteristics is obtained from the superposition of incident and reflected pulses.

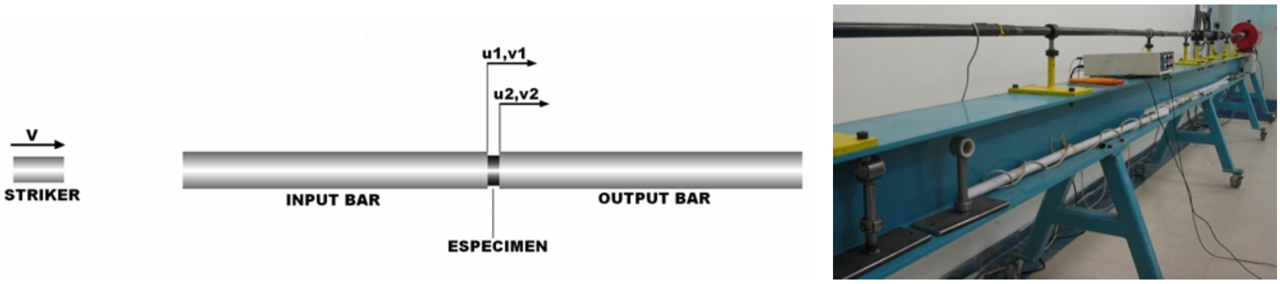


Figure 3: SPHB operation scheme and a perspective view of the equipment.

The basic assumptions on a SPHB test are:

- None of the bars undergoes plastic deformations during the test;
- Only unidirectional elastic pulses exist in the bars;
- The time interval a pulse takes to cross the specimen is very small compared to the total time of the test;
- The specimen deformation process is uniform;
- There is no dispersion of waves throughout the bars and the specimen.

The incident pulse, ε_i, the transmitted pulse, ε_t, and the reflected one, ε_r, are measured by using strain-gages attached to the external surfaces of the bars, allowing to obtain the stress-strain curve and the strain rate of a specimen with length L_s and cross section area A_s, as indicated in the expressions below:

$$\varepsilon_s = \frac{c_0}{L_s}\int_0^t (\varepsilon_i - \varepsilon_r - \varepsilon_t)dt \tag{1}$$

$$\sigma_s = E\frac{A}{2A_s}(\varepsilon_i + \varepsilon_r + \varepsilon_t) \tag{2}$$

$$\dot{\varepsilon}_s = \frac{c_0}{L_s}(\varepsilon_i - \varepsilon_r - \varepsilon_t) \tag{3}$$

where $c_0 = \sqrt{E/\rho}$ is the speed of the elastic wave, E is the Young´s modulus, ρ is the density and A is the cross section area of he bars.

The specimen geometry adopted here follows the dimensions proposed by Honsur *at al.* (2001) and Sun and Tsai (2002), Fig. 2. It is worthwhile to note that the same geometry was adopted to quasi-static and dynamic compression tests, since one of the main aims of this work is to investigate solely the influence of strain rate on the mechanical response. Due to the equipment technical limitations it was possible to perform the dynamic material characterization in the range of 500 s^{-1} to 2500 s^{-1}.

4. EXPERIMENTAL RESULTS AND DISCUSSION

This section presents the results obtained in the composite mechanical characterization through the methodology described in previous sections. It must be mentioned that all the curves were transformed to true stress, σ, and true strain, ε, values, since impact phenomena are usually related to finite strains and distortions.

4.1 Quasi-static characterization

In this section it will be presented the stress-strain curves, under compression and tension loadings, of composites with 0%, 5% and 10% added nanoclays. In the sequence it will be depicted the tension test results of the material described as ‘32 layers (65% in weight of fibers)’ by using ‘Configuration A’ to the specimens. The curves shown in Fig. 4 represent, respectively, the material behavior under tension at [0°,90°] and [-45°,+45°] directions.

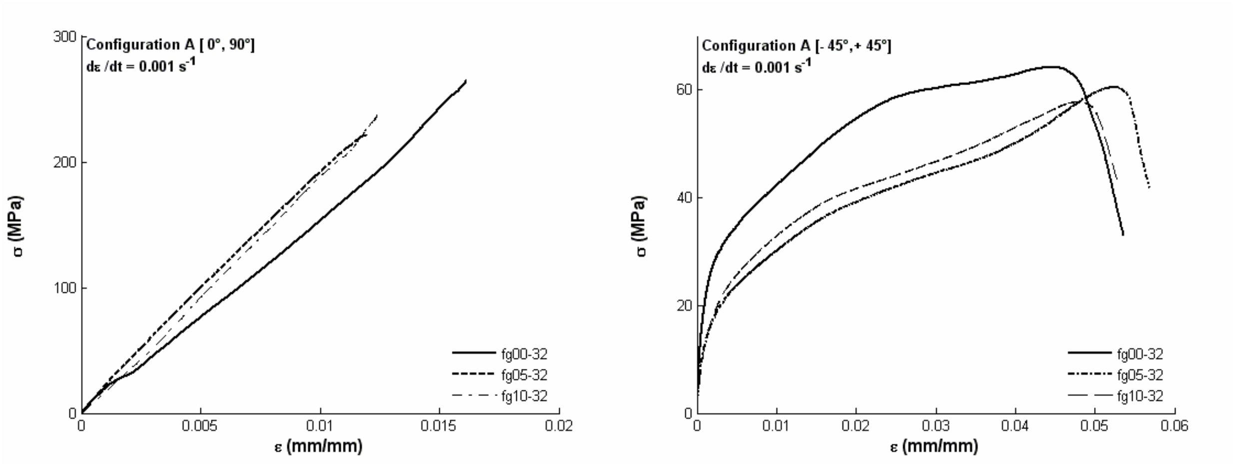


Figure 4: Stress-strain curve to the 32-layers composite laminate under tension states [0°,90°] and [-45°,+45°], when using specimen ‘Configuration A’ .

From the results shown in Fig. 5 one can clearly observe the material anisotropy, since the material response varies as the applied load direction changes. This differentiation may be observed mainly in the maximum stress values linear constitutive equations are adequate to model the material behavior when the loading and fiber direction coincide, but when off-axis situations are considered higher order constitutive relationships must be used, as was also observed by Sun and Tsai (2002), among others. This discrepancy can be explained as follows: in the first case the material under tension is the fiber, and being a ceramic like material has a brittle behavior (linear up to failure); in the second case, the matrix influence adds viscoelastic characteristics to the material behavior as a whole. The same conclusions are valid to 24-layers composite laminates, since the only difference to the previous specimen is exactly the number of layers.

Keeping fixed the load direction, no significant differences of material behavior for different nanoclays concentration were observed. It must be noted that the specimen geometry allowed a correct control of load application and the totality of the tests presented failure within the strain gage valid range of operation.

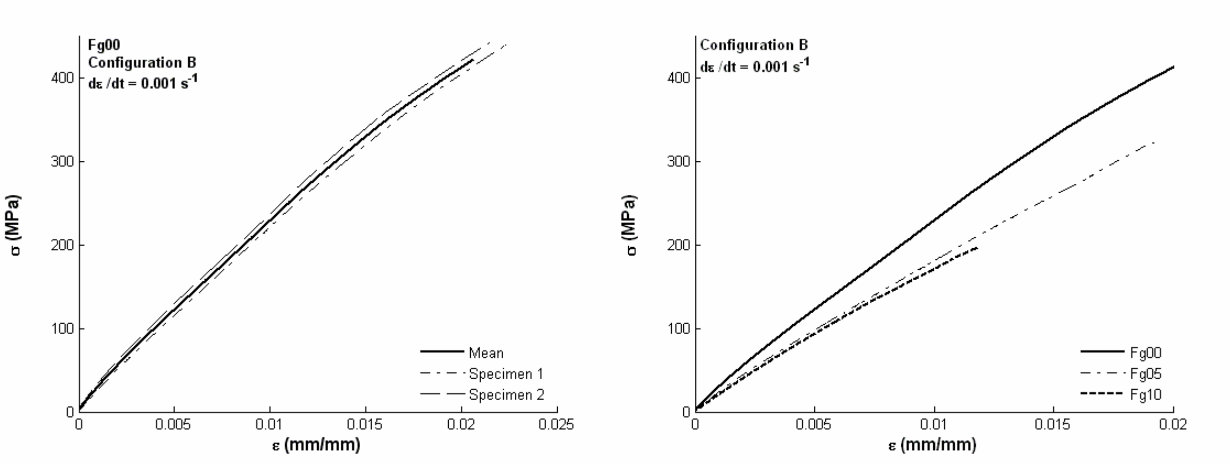


Figure 5: Stress-strain curves to tension test accordingly the ASTM-D3039 standard.

The test results, obtained through ASTM-D3039 standard and adopting ‘Configuration B’ are presented in Fig. 5. In these tests, a better mechanical behavior was observed in the material without nanoclays addition, although a great similarity in the results of the different samples continues being verified. The adopted configuration did not allow total control of applied load, since in 90% of the tests executed accordingly the ASTM-D3039 standard, the failure occurred outside the strain gage region, but to the 45° in the region of the mordents. In Fig. 6, the stress-strain curves obtained for Fg05 and Fg10 specimens indicate an increase of stiffness as the strain rate of the test is increased. The failure mechanisms of the different specimens were not the same so the higher value of the limit stress, observed for the Fg05 specimen, cannot be considered.

The results of compression quasi-static tests will be presented in section 4.2 together with the dynamic results, in order to facilitate the comparison between these two sets of results.

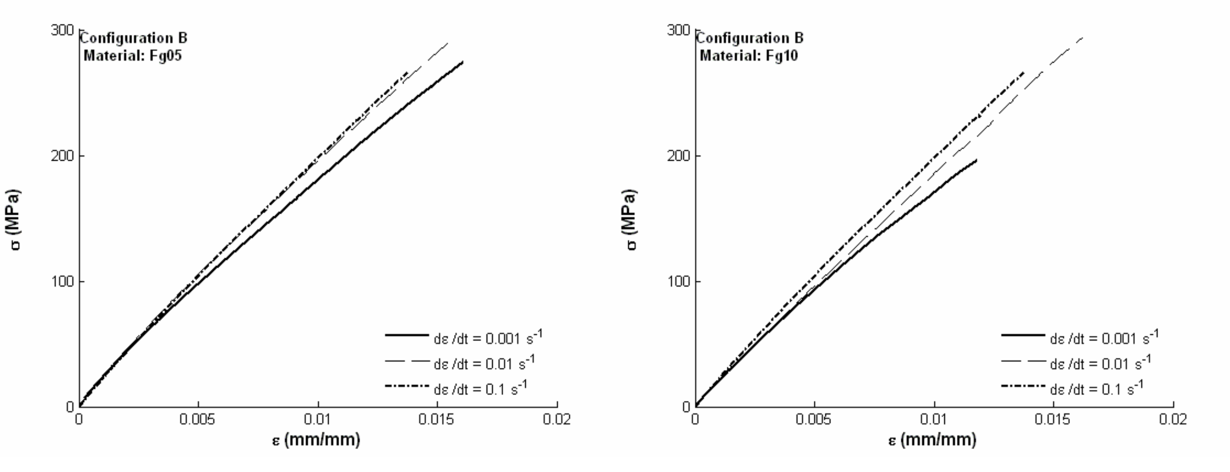


Figure 6: Stress-strain curves to Fg05 and Fg10 under tension at various strain rates.

4.2 Dynamic characterization

In the sequence it will be presented the dynamic compression stress-strain results of 0%, 5% e 10% nanoclays added materials with 24 and 32 layers. In Fig. 7, the obtained stress-strain curves, including quasi-static results, are depicted.

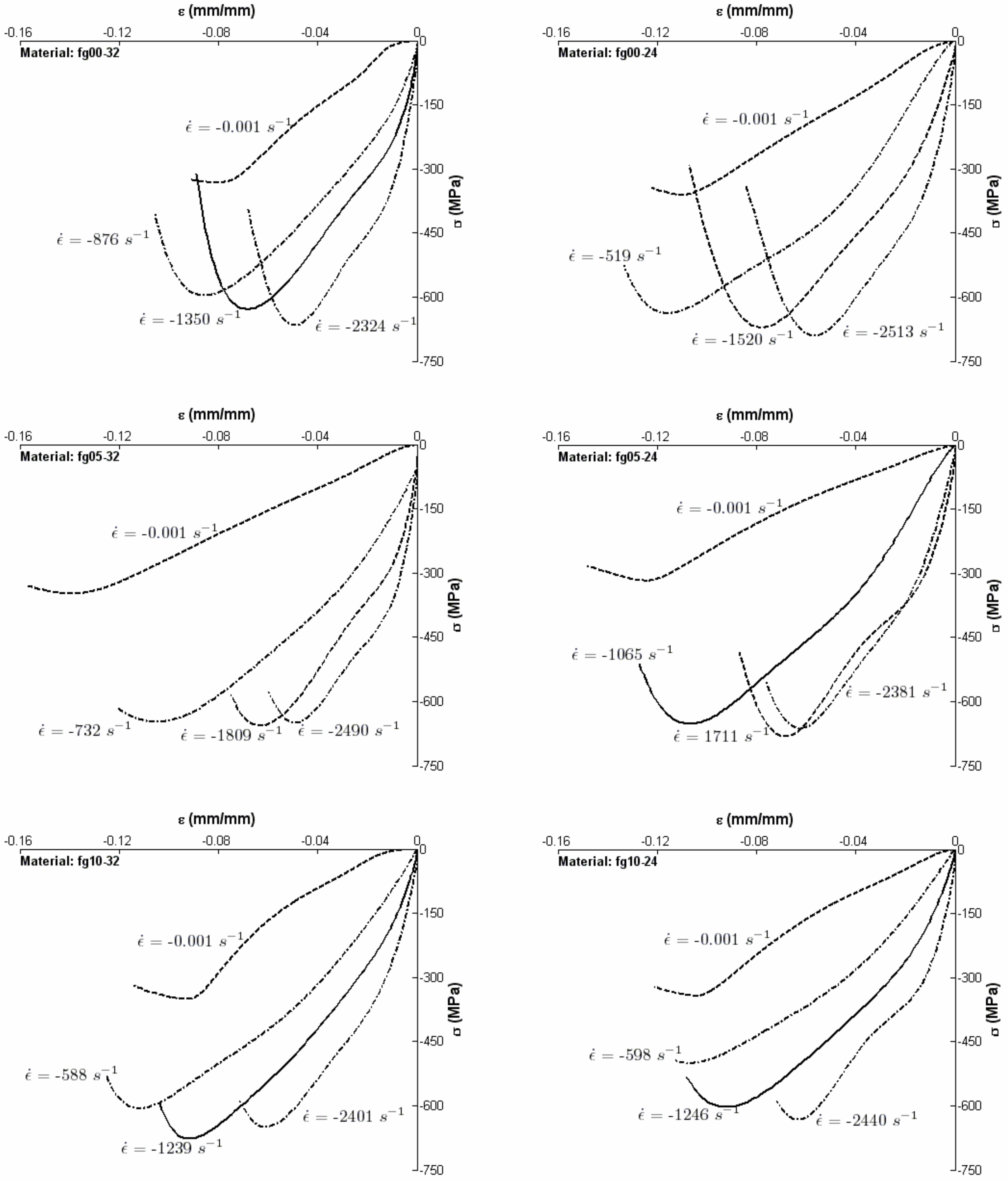


Figure 7: Stress-strain curves to Fg 00, Fg05 and Fg10 specimens under compression, at high strain rates (quasi-static results shown for comparison purposes).

From the analysis of the graphs one can conclude that all the configurations of the material in study are strain rate sensitive, showing behavior differentiated of the ones presented in the quasi-static tests. Among the differences in the quasi-static and dynamic material behavior it can be cited the maximum stress, being on average 90% to 120% greater than the quasi-static one. Another remarkable feature refers to resilience modulus of the material, i.e. the energy absorbed by it until the collapse. One can observe that this characteristic increases significantly when the load is dynamically applied. Thus, it can be concluded that this material presents characteristics that make possible its use in elements subjected to impact, as presented in other works, since both the maximum tension and absorbed energy increased when the load is of a dynamic nature. Finally, amongst the tests performed for the characterization of the material, it was not observed an important influence of the fraction of fibers and nanoclays concentration on the material strength.

5. CONCLUSIONS

A experimental framework to both quasi-static and dynamic material characterization of fiber-glass reinforced composed with additives (montmorillonite nanoclays) has been described. The results indicate that:

- It was found that the material in study has an anisotropic behavior, showing distinct behavior when different load directions are considered;
- For the quasi-static tension tests it was observed differentiation in the behavior of the material when different configurations of test body are used. The ‘Configuration A’ made possible a better control of the applied load, generating a failure pattern within the region measured by the strain-gage in all the valid tests; however this configuration showed a less resistant material. Tests with ‘Configuration B’, following ASTM-D3039 standard, presented a more resistant material however it did not make possible the failure control. It seems that such phenomenon occurs due to a greater sensitiveness of configuration;
- One can conclude that the material in study is sensible to strain rate to both tension and compression actions, as proven, respectively, in the quasi-static and SHPB dynamic tests.
- The main differences in the behavior of the material under compression in quasi-static and dynamic regimen are the increase of the maximum tension supported and energy absorbed until the collapse. The maximum tension is about 90% to 120% greater of what the maximum tension in quasi-static regimen. The energy absorbed by the material also increases significantly;
- In the tests performed for the characterization of the material, it was not observed an important influence of the fraction of fibers and nanoclays concentration on the material strength.

6. ACKNOWLEDGEMENTS

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