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INFLUENCE OF TEMPERATURE AND STRAIN RATE ON THE TENSILE BEHAVIOR OF EPOXY REINFORCED WITH CARBON NANOTUBES

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Abstract. *The purpose of this work is study the behavior of an adhesive composite diglycidyl ether of bisphenol A (DGEBA) reinforced with carbon nanotubes. Tensile tests were performed under three temperatures (25ºC, 50ºC and 75ºC), varying the strain rate (0.5mm/min, 5.0mm/min and 50mm/min). It was shown the effect of temperature and strain rate on the behavior of the composite. Stiffness and ultimate tensile strength decrease with temperature while they are directly proportional with strain rate. The experimental results are analyzed to illustrate this. In further work, an analytical expressions based on the curves obtained on this work would be desire to propose a modeling for ultimate strength and Young's modulus as function of temperature and strain rate*
Keywords: *epoxy, temperature, tensile test, strain rate, modeling*

1. INTRODUCTION

There is a growing use of polymers in different fields of industry such as aerospace, automotive and petrochemical (Reis, *et al.*, 2012), because of its low cost and weight and facility of manufacture and processing (Reis, *et al.*, 2015). Maintenance and repair of ducts, pipes and others structures represent great cost on industry (Amorim, 2015). The role of polymer adhesives is getting essential on structural applications, protective coatings and reparability, especially on petroleum industry, where the welded structures have shown unwilling, since the weld process causes metallurgical transformations and concentration of residual stresses. Besides, from time to time the weld joints show crack, due to the aggressive and corrosive environment they are exposed (da Silva, 2007).

In order to override this method, petrochemical industries have used adhesive polymers and their composites. Currently, the epoxy resin is widely used in adhesives and as matrix composites, such as Bisphenol A epoxy, due to its easiness of shaping and its excellent adhesive, mechanical and thermal properties (Gonzalez-Garcia, *et al.*, 2005). Since mechanical properties of carbon fiber are engineered, being the most resistant reinforcing fiber, and at room temperature it is not affected by humidity or aggressive means like acids (Callister, 2008), its usage as fiber phase on composites has increased and the necessity to know better its interaction with resin matrix is needed (Reis, *et al.*, 2013). Mechanical properties of composites is determined by both fiber and matrix phases. Not only the properties of it single material have great influence, but also the size and disposition of the fiber, i.e. the interface fiber/matrix affects the properties of the final composite (Dilsiz and Wightman, 1999).

Yet those properties are sensitive to temperature and strain rate at which the composites are loaded (Reis, *et al.*, 2012). Until today there are few studies and information available about the effects of strain rate and temperature on adhesives composites. The stiffness and strength on composites materials normally drops with temperature rise, and decrease even more when temperature exceeds the glass transition temperature (Bai, *et al.*, 2008). It is reported (Harding, 1992) that as the strain rate goes higher, the ultimate strength increase.

In this present paper, the effect of temperature and strain rate on the behavior of DGEBA adhesive reinforced with carbon nanotubes is analyzed through tensile tests. The experimental results are presented to show the influence of temperature and strain rate on the strength and Young Moduli of the material. Next analytical expressions are proposed for those parameters as functions of strain rate and temperature. The results obtained with the analytical expressions are compared with the experimental ones.

2. MATERIALS AND METHODS

2.1 Materials

The adhesive composite has a matrix phase of diglycidyl ether of Bisphenol A (DER 331, EEW=190) from *Dow Química S.A.,* and fiber phase of single-wall carbon nanotubes (Baytubes C-150 CP) from Bayer, with diameter varying from 0.4-3.0 nm, and a concentration of 5%. The merger process was: dispersion at high speed with grinding mill. A curing agent of polyamides was used with a mix ration 100:43. The dispersion of nanotubes in the epoxy matrix was observed on a SEM is shown on "Fig. 1". It was dd a voltage of 5kV with a zoom varying from x8000 to x15000.

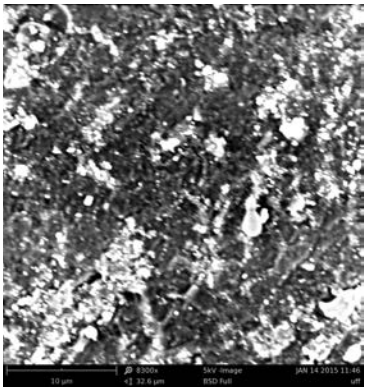


Figure 1. Example of picture taken by SEM, the white reflection represents the nanotubes.

From the *Dow Química* technical report, DER 331 is the most used liquid epoxy resin, since it applications are wide: adhesives, automotive coatings, casting, composite etc. It is also used as a standard for composites and others variations. According with Morell *et al.* (2009), DGEBA Young's modulus and glass transition temperature are 3.8 GPa and 116ºC, respectively; more properties are presented on "Tab. 1" according to manufacturer. The Baytubes are used to increase Young Moduli and hardness on composite. According to the manufacturer, the dispersion of Baytubes on a polymer matrix will reduce the length of crack and its propagation, which enhance the durability of the epoxy composite.

Table 1. Properties of DGEBA (DER 331).

Viscosity at 25ºC (mPa•s)	11000-14000
Density at 25ºC (g/ml)	1.16
Heat Deflection Temperature (ºC)	111
Flexural Strength (N/mm²)	96
Flexural Modulus (kN/mm²)	3.0
Tensile Strength (N/mm²)	79
Maximum elongation (%)	4.4

The specimens used on the tensile test were manufactured with shape and size specified by ASTM D-638-10, 2010 (Type I). For a thickness equals 7 or under, the distance between grips *d* is 115 mm, the gage length *g* is 50 mm and the initial cross section is 65 mm² ($w \times t$, where $w = 13$ mm is a standard dimension and $t = 5$ mm is the average thickness of the specimens manufactured) "Fig. 2". The length overall *l* of the specimens is 165 mm. The DGEBA and carbon nanotubes were mixed and disposed in steel mold with the standard specimen size and cured at room temperature.

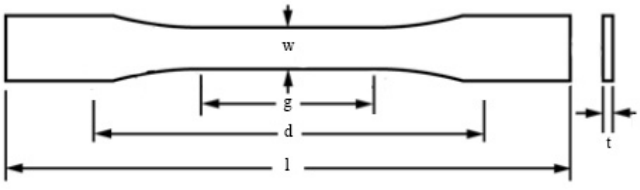


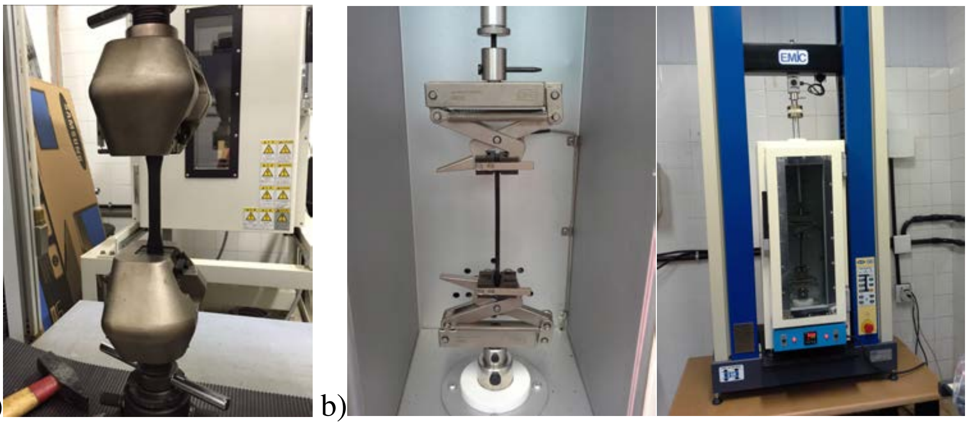
Figure 2. Standard tensile test specimen dimensions according to ASTM-D638-10

2.2 Methods

Three temperatures were pre defined: room temperature 25ºC, 50ºC and 75ºC. For each temperature three strain rate were determined: 5 mm/min, according to ASTM-D638-10, 50 mm/min and 0,5 mm/min. Those strain rates were determined as 10% more and 10% less respectively than the standard strain rate (Reis, *et al.*, 2012).

The tensile test at 25ºC were performed on a Shimadzu® AG-X universal testing machine "Fig. 3a". For the others two temperatures an EMIC 5kN universal machine with a thermostatic chamber was used "Fig. 3b". Five specimen were performed at a given strain rate for each temperature, total of 45, as a standard procedure (ASTM D-638-10, 2010).

After the performances, it was calculated the mean curve for each strain rate at each temperature and determined the modulus of elasticity and strength.



a) b)

Figure 3. a) Tensile test at 25ºC performed on a Shimadzu® AG-X; b) EMIC machine used to performed tensile test for 50ºC and 75ºC.

3. RESULTS AND DISCUSSION

The mean curves for each test are illustrated in "Fig. 4"; they were defined until the elastic limit since for the tests at 50ºC and 75ºC, after a while inside the oven, the material changed its linearity and the curves were too complex to analyze. Then the ultimate strength is not represented on these curves, but it is shown in "Tab. 2".

At room temperature the curve is linear for any strain rate. As the temperature increase, the curves become unstable. At 75ºC the tests at 5 mm/min resulted on a mean curve slightly lower than the tests at 0.5 mm/min. But the ultimate strength increase with higher strain rate as it was expected ("Tab. 2").

Analyzing "Tab. 2" and "Tab. 3", it is observed that the ultimate strength and Young's modulus decrease with temperature and increase with strain rate, which confirms the dependence of the behavior of the composite with temperature and strain rate (Banea, *et al.*, 2011).

Table 2. Ultimate strength in MPa

	25ºC	50ºC	75ºC
0.5 mm/min	28.25	8,03	1,03
5.0 mm/min	29.28	17,97	1,82
50,0 mm/min	31.05	18,37	2,19

Table 3. Young's Modulus in GPa

	25ºC	50ºC	75ºC
0.5 mm/min	2.4	1.3	0.070
5.0 mm/min	2.6	1.5	0.056
50,0 mm/min	2.8	2.1	0.269

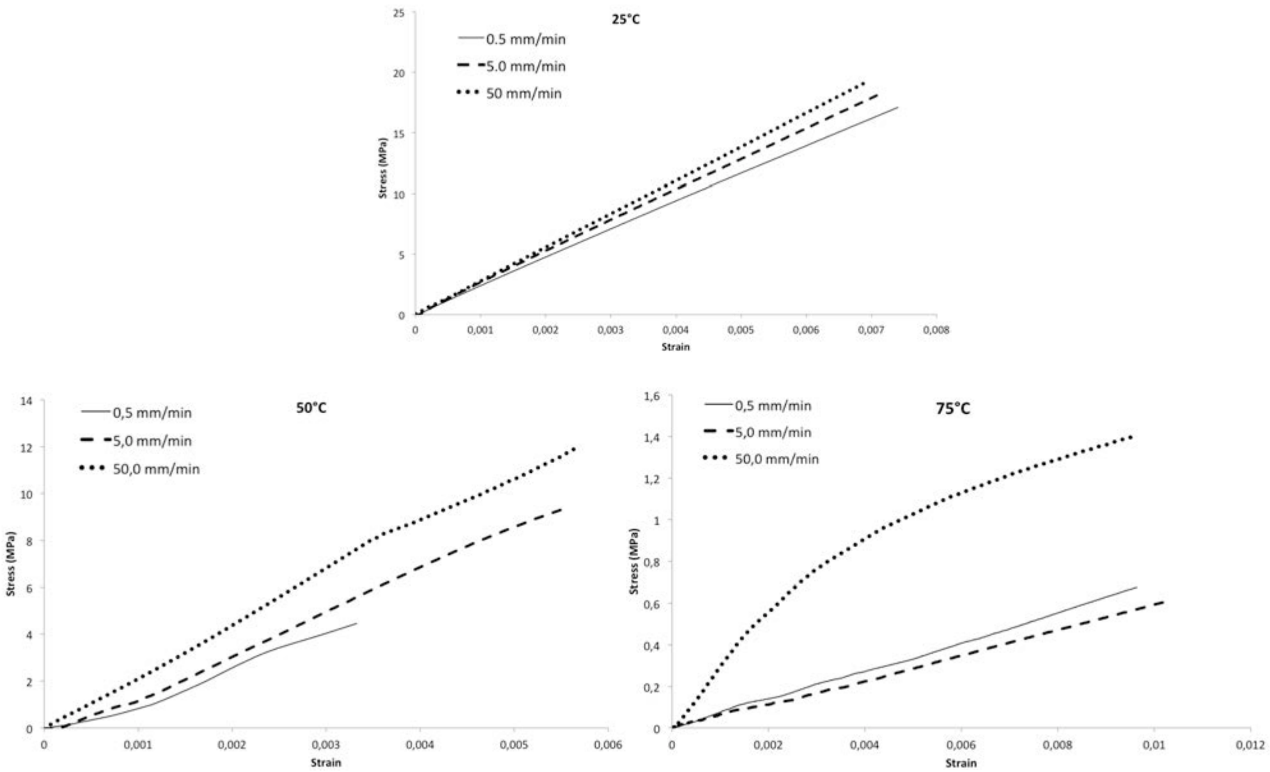


Figure 4. Top: mean curves of tests at 25ºC for 0.5 mm/min, 5.0 mm/min and 50 mm/min; bottom left: mean curves of tests at 50ºC for each strain rate; bottom right: mean curves of tests at 75ºC for each strain rate.

The stress-strain curve showed on “Fig. 5” illustrates how this composite is influenced by temperature. When grouping all the curves above in a single chart, at 75ºC the modulus of elasticity is so low that the strength could be mistaken by a constant. In fact, during the tensile tests the material was starting to melt, especially in low strain rate (0.5 mm/min), because the specimen stayed longer inside the oven, then the shape of the specimen had a slightly bent after rupture as in “Fig.6”. The difference on stiffness between the curves of 50ºC and 25ºC is lower than the curves of 75ºC compared with 50ºC, which suggest that the composite has non-linear behavior as a function of temperature. An analytical modeling would be appropriate to verify this dependence.

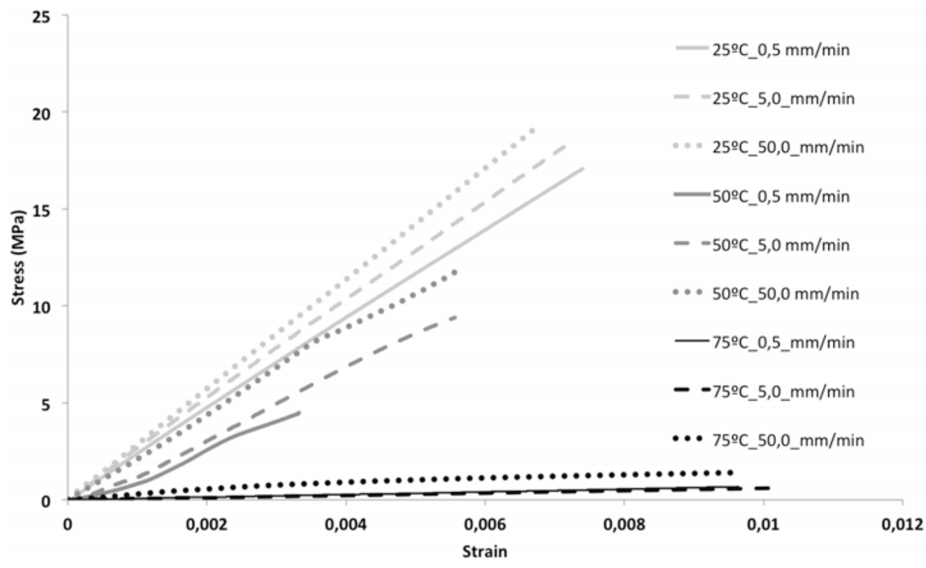


Figure 5. Mean curves of the tensile test.

Figure 6. Curved specimen after a test.

4. CONCLUSION

The effect of temperature and strain rate on the behavior of mechanical properties of the DGEBA adhesive reinforced with carbon nanotubes was observed. The response to a range of temperature (25ºC, 50ºC and 75ºC) and strain rate (0.5, 5.0, 50.0 mm/min) was experimentally measured during tensile tests, showing a decrease of Young's modulus and ultimate strength with temperature, but an increase of this properties with strain rate. The results show that the mechanical properties of the composite are affect by the strain rate and strongly by the temperature, suggesting that this dependence is not linear. It is desirable to develop analytical expressions in future work to observe this dependence and compare the experimental results with predicted ones.

5. REFERENCES

Amorim, F.C., 2015, *Infuência da Temperatura no comportamento mecânico do polímero DGEBA (Diglicidil éter bisfenol A*, M.Sc thesis, Universidade Federal Fluminense, Niterói, Brazil.

ASTM D638-10. Standard test method for tensile properties of plastics; 2010.

Bai, Y., Keller, T., Vallée, T., 2008. "Modeling of stiffness of FRP composites under elevated and high temperatures". *Composites Science and Technology* 68 (2008) 3099–3106

Banea, M.D., de Sousa, F.S.M, da Silva, L.F.M., Campilho, R.D.S.G., de Bastos Pereira, A.M., 2011. "Effects of Temperature and Loading Rate on the Mechanical Properties of a High Temperature Epoxy Adhesive". *Journal of Adhesion Science and Technology*, Vol.25 (2011) 2461–2474

Callister, W.D. , 2007. *Ciência e engenharia de materiais uma introdução*. 7th edition.

Da Silva, A.H.M.F.T., 2007, *Proposta de um critério de resistência para juntas metálicas soldadas*, M.Sc thesis, Universidade Federal Fluminense, Niterói, Brazil.

Dilsiz, N., Wightman, J.P., 1998. "Surface analysis of unsized and sized carbon fibers". *Carbon 37* (1999) 1105-1114

DOW, "Product Information". 1 Jul. 2015 <http://msdssearch.dow.com/PublishedLiteratureDOWCOM/dh_01e8/0901b803801e8ef7.pdf?filepath=epoxy/pdfs/noreg/296-01408.pdf&fromPage=GetDoc>

González-Garcia F., Minguez E., Soares, B.G, 2005. "Caracterização do sistema éter diglicidílico do bisfenol A / poliaminas alifáticas". *Polímeros: Ciência e Tecnologia*, vol. 15, n° 4, p. 261-267, 2005

Harding, J., 1993. "Effect of strain rate and specimen geometry on the compressive strength of woven glass-reiforced epoxy laminates", *Composites*, Vol. 24, number 4, p. 323

Morell, M., Ramis, X., Ferrando, F., Yu, Y., Serra, A., 2009 "New improved thermosets obtained from DGEBA and a hyperbranched poly(ester-amide)", *Polymer*, Vol. 50 (2009) 5374-5383

Reis, J.M.L., Amorim, F.C., da Silva, A.H.M.F.T., da Costa Mattos, H.S., 2015. "Influence of temperature on the behavior of DGEBA (bisphenol A diglycidyl ether) epoxy adhesive". *Compos* Part B 2012.43:2041.

Reis, J.M.L., Chaves, F.L., da Costa Mattos, H.S., 2013. "Tensile behaviour of glass fibre reinforced polyurethane at different strain rates", *Materials Design*, 49 (2013) 192-196, 2013.

Reis, J.M.L., Coelho, J.L.V., Monteiro, A.H., da Costa Mattos, H.S., 2012. "Tensile behavior of glass/epoxy laminates at varying strain rates and temperatures". *Compos* Part B 2012.43:2041.

6. RESPONSIBILITY NOTICE

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