# Mechanical properties of thermoset-metal composite prepared under different process conditions

Gean Vitor Salmoria[1*], Felix Yañez-Villamizar[2], Aurelio Sabino-Netto[3]

CIMJECT, Department of Mechanical Engineering, Federal University of Santa Catarina, 88040-900 Florianopolis, Santa Catarina, Brazil

***Abstract*—** *This paper reports a study on the morphology and mechanical behavior of a thermoset-metal composite used in prototype molds as a function of the process conditions. The investigation of the mechanical properties of epoxy-aluminum specimens post-cured using different routines showed that they are related to the self-controlled diffusion characteristic of thermoset polymeric systems. A high-temperature post-cure routine resulted in higher values for the modulus, stiffness and glass transition temperature, Tg, for the specimens. The fracture surfaces analysis showed the presence of defects, which appeared as empty spaces in the epoxy matrix, due to the mixing and casting process. The defect size and the specimen strength showed a direct correlation. The Weibull modulus was 7.95 for the epoxy specimens characterizing low toughness and the presence of defects in the material, as revealed by fractography. The probability of failure increased rapidly to 50% for applied stress greater than 38 MPa.*

***Keywords*— *Thermoset-metal, mechanical properties, process conditions.***

## I. INTRODUCTION

Thermoset resin curing permits the rapid manufacturing of parts and objects by casting. The main applications of thermoset curing are the production of prototypes and parts used in the automobile, electrical, biomedical and aerospace industries. Thermoset curing is also applied in the manufacture of functional prototypes, such as prototype tools [1-7]. The thermal and mechanical properties are of great importance for prototype tools such as molds for the injection of thermoplastics, since these tools are subjected to particular work conditions, with temperature variations and mechanical requirements [7-12].

The mechanical properties of parts or tools manufactured by thermoset curing are dependent on the degree of cure of the resin. Manufacturing parameters, such as mixing, curing, post-curing and resin characteristics (monomers, oligomers and curing agent composition), maximum density of cross-linking, photosensitivity, rate and degree of cure, and post-cure method are very important in relation to the structure and properties of the part or tool [10-17]. Knowledge of the relationship between the thermoset resin structure and properties is useful in the manufacturing process, application and quality control of tools. Prototype molds with molding blocks produced by thermoset polymer resins have low mechanical properties compared to conventional steel tools. Prototype molds produced by casting of a thermoset-metal composite are commonly used to produce a small series of polymeric parts [5, 7 12, 14-17]. During injection molding, the mold is subjected to several types of loading, either static or dynamic. This means that the composite in the molding blocks needs to have good mechanical performance to avoid premature failure of the prototype mold. This paper reports a study on the morphology and mechanical behavior of an epoxy-aluminum composite used in prototype molds as a function of the post-cure process conditions.

## II. MATERIALS AND METHODS

### 2.1 Materials

An epoxy polymer containing 30% aluminum by weight, supplied by Hunstman as RenCast 436, was mixed with the appropriate curing agent, Ren HY 150, obtained from the same company. The material is an epoxy system base in bisphenol A and phenol novolac derivatives.

### 2.2 Mixture Procedure

In order to determine the effect of the post-cure process on the mechanical properties of the epoxy-aluminum composite, the mixture of epoxy pre-polymer with aluminum particles and curing agent was prepared applying mechanical stirring (85 rpm) for 2 min at 20 mmHg. The mixture was poured into silicone rubber models to fabricate the test specimens.

### 2.3 Cure and Post-cure Procedure

Experiments were carried out to evaluate the influence of the post-cure on the tensile strength and the dynamic mechanical properties of the material. Specimens were cured at room temperature for 24 h, according to the procedure suggested by the supplier, and post-cured in a conventional thermal oven using the post-cure routines shown in Table 1.

**TABLE 1**
**POST-CURE ROUTINES APPLIED TO THE CASTING EPOXY SPECIMENS**

| Specimens | Post-cure routine |
|---|---|
| A | 2 h at 70ºC ; 2 h at 90ºC; 2 h at 120ºC; 2h at 150ºC |
| B | 4 h at 90ºC ; 4 h at 150ºC |
| C | 4 h at 70ºC ; 4 h at 150ºC |

### 2.4 Tensile Tests

Tensile test specimens were obtained, according to the standard ASTM D638 type I, using the epoxy-aluminum composite. Five specimens were tested for each post-cure cycle, at uniaxial tension and at room temperature, using an EMIC universal testing machine with a displacement rate of 5mm/min. The Young's modulus was determined through the stress–strain correlation considering the linear region (Table 2). The strain was measured using an incremental extensometer.

### 2.5 Fractography

Fracture surfaces of the tensile specimens were observed with a scanning electron microscope (Philips XL30) in order to investigate the fracture topography. Each specimen was coated with gold in a Bal-Tec Sputter Coater SCD005. Fracture image analysis was carried out using Analysis Pro 2.11.002©Soft-imaging Software GmbH in order to investigate the failure.

### 2.6 Dynamic Mechanical Analysis (DMA)

Dynamic mechanical analysis was carried out on specimens with dimensions of 10 mm x 40 mm x 3.5 mm over a temperature range of -70ºC to 200 °C using a DMTA analyzer (Elkin-Palmer Instruments) in the three bending point mode geometry, according to the standard ASTM D5023. The specimens were scanned isochronally at 3 °C/min. The damping factor (Tan □) and storage modulus (Log E') were recorded at 1 Hz.

## III. RESULTS AND DISCUSSION

Figure 1 presents the stress-strain curves for the post-cured specimens. Table 2 shows the main values for the elastic modulus and the tensile strength for the post-cured specimens and it can be observed that specimens post–cured using routine A presented higher tensile strength values.

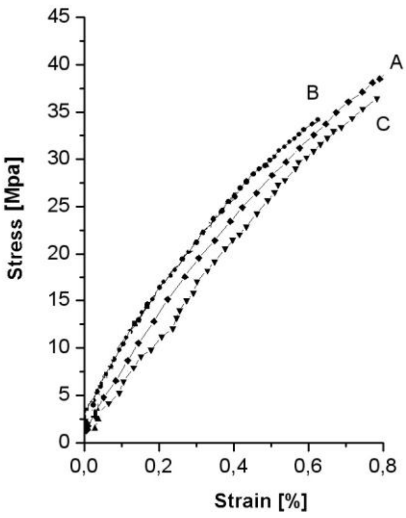


**FIGURE 1. STRESS VERSUS STRAIN CURVES FOR THE EPOXY SPECIMENS POST-CURED USING DIFFERENT ROUTINES.**

**TABLE 2**
**ELASTIC MODULUS AND TENSILE STRENGTH VALUES FOR SPECIMENS POST-CURED USING DIFFERENT ROUTINES.**

| Post-cure routine | Elastic Modulus [MPa] | Tensile strength [MPa] |
|---|---|---|
| A | 6270 (±200) | 37 (±3) |
| B | 6350 (±300) | 33 (±3) |
| C | 6220 (±300) | 35 (±3) |

These results are related to the self-controlled diffusion characteristic of the thermoset polymeric systems, leading to a greater improvement in the crosslink density when gradual thermo-curing ramp routines are applied, i.e. in A routine.

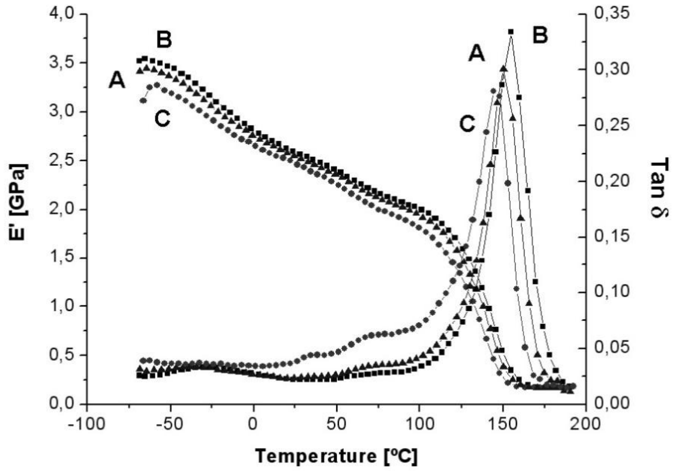


**FIGURE 2. STORAGE MODULUS E´ AND TAN δ CURVES FOR THE EPOXY SPECIMENS POST-CURED USING DIFFERENT ROUTINES.**

The storage modulus E´ and Tan δ are properties related to the stiffness and toughness of epoxy systems. Post-cure routine B resulted in higher modulus and stiffness values for the specimens. The glass transition temperature, Tg, is obtained when Tan δ is maximum and higher Tg values were obtained for the B specimens confirming a higher cure degree.

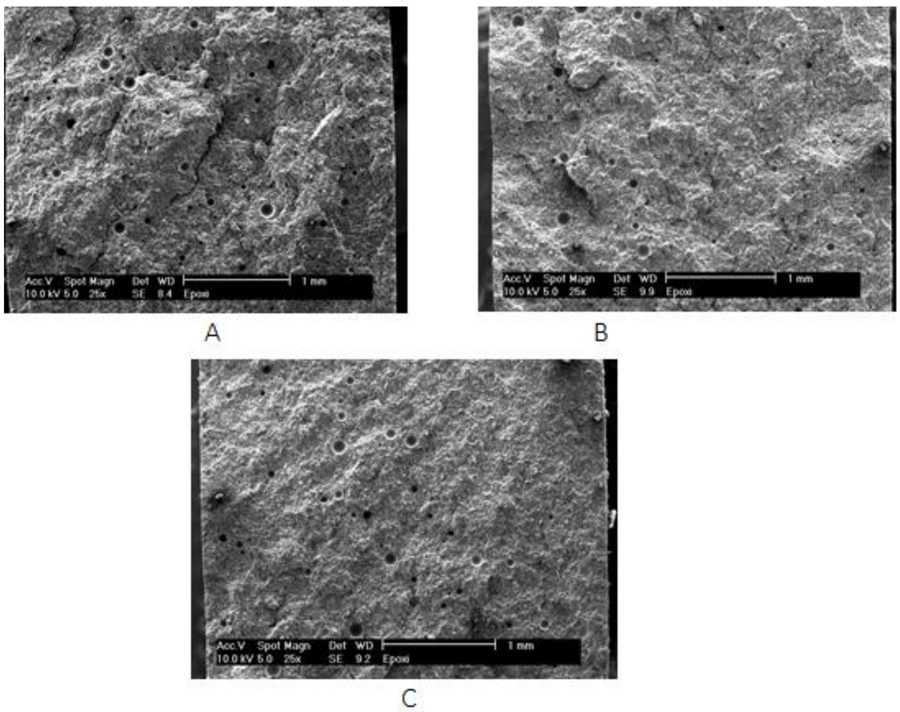


**FIGURE 3. FRACTURE SURFACES OF SPECIMENS POST-CURED USING DIFFERENT ROUTINES.**

The fracture surface analysis showed the presence of small defects which appeared as empty spaces (bubbles) in the epoxy matrix, probably due to the mixing and casting process. The investigation of the relationship between the defect size and the specimen strength showed a direct correlation, as observed in Figure 4.

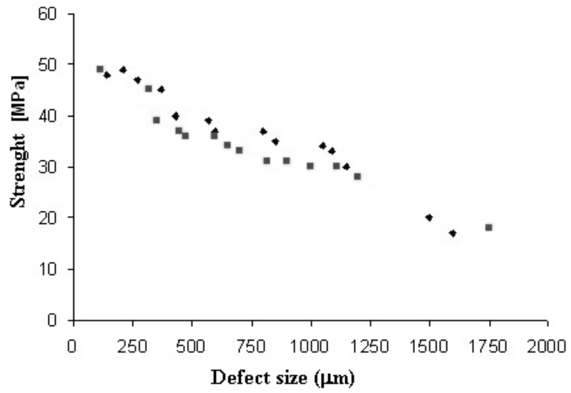


**FIGURE 4. RELATIONSHIP BETWEEN DEFECT SIZE AND STRENGTH FOR EPOXY SPECIMENS.**

The Weibull modulus characterizes the material friability and the influence of defects. Figure 5 shows the slope of the curve for the Weibull modulus, with a value of 7.95 for the epoxy specimens, characterizing low toughness and defects in the material, as shown in the fractography analysis, independent of the post-cure process applied.

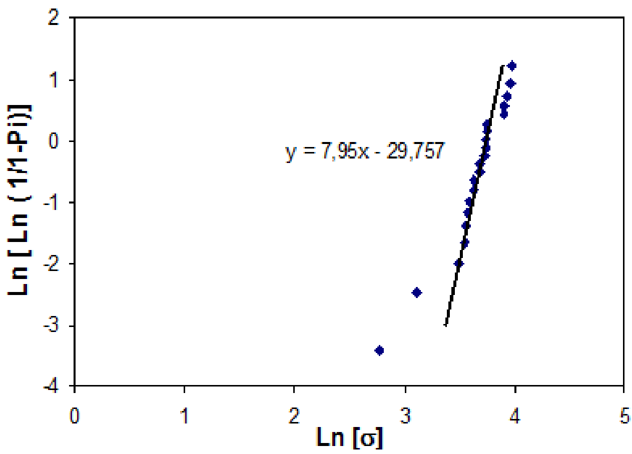


**FIGURE 5. THE WEIBULL MODULUS CHARACTERIZING THE MATERIAL FRIABILITY**

Figure 6 shows the probability of failure as a function of the applied stress. For an applied stress higher than 30 MPa the failure probability increases rapidly reaching 50% and 80% when the stress is close to 38 and 42 MPa, respectively.

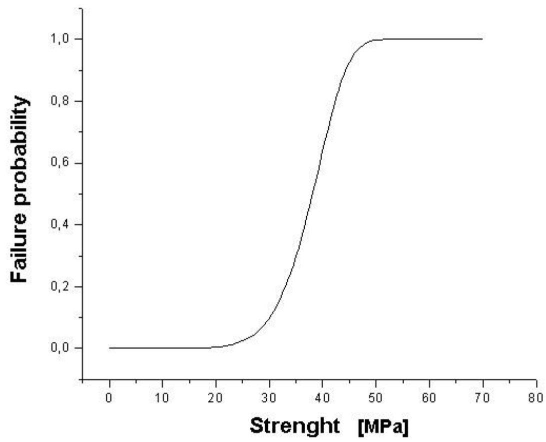


**FIGURE 6. THE FAILURE PROBABILITY FOR EPOXY SPECIMENS.**

## IV. CONCLUSIONS

The relationship between the resin structure and properties provides useful information for the casting manufacturing, application and quality control of tools. The mechanical properties of epoxy-aluminum specimens post-cured by different routines were found to be related to self-controlled diffusion, which is characteristic of thermoset polymeric systems. A greater improvement in the crosslink density occurred when gradual thermo-curing ramp routines were applied, reaching higher tensile strength values and a lower number of defects due to a more homogenous cure. A high-temperature post-cure routine resulted in higher values for the modulus, stiffness and glass transition temperature, Tg, for the specimens. The fracture surface analysis showed the presence of defects due to the mixing and casting process, which appeared as empty spaces in the epoxy matrix. The defect size and the specimen strength showed a direct correlation. The Weibull modulus was 7.95 for the epoxy specimens, characterizing low toughness and defects in the material, as observed by fractography. For applied stress higher than 38 MPa the failure probability increased rapidly to 50%. The results demonstrate that epoxy-aluminum composites show interesting thermal and mechanical properties for tools manufacturing. However, the aluminum content and manufacturing defects can limit their use under critical conditions such as specific geometries and in high stress molding.

## V. ACKNOWLEDGMENTS

The authors would like to thank FAPESC-PRONEX, CNPq and AEB (Brazilian Aerospace Agency) for financial support.

## REFERENCES

[1] Jacob P.F. (1999) From Rapid Prototyping to Rapid Tooling, ASME, New York.
[2] Salmoria G.V., Gonzalez V.J, Ahrens C.H, Soldi V, Pires A.T.N. (2005). Stereolithography Somos 7110 photosensitive resin: study of curing kinetic and thermal degradation. Journal of Material Processing Technology. 168 (11), 164–71. doi:10.1016/j.jmatprotec.2004.11.012
[3] Salmoria G.V., Ahrens C.H., Fredel M., Soldi V, Pires A.T.N. (2005). Stereolithography Somos 7110 resin: mechanical behaviour and fractography of parts post-cured by different methods. Polymer Testing 24, 157–62. doi:10.1016/j.polymertesting.2004.09.008
[4] Westphal M.G., Pouzada A.S., Salmoria G.V., Ahrens C.H. (2006). Performance and Friction Properties of Injection Hybrid Molds with Stereolitography Molding Zones. Materials Science Forum. 514, 1673-1677. doi:10.4028/www.scientific.net/MSF.514-516.1673
[5] Gonçalves M.W., Salmoria G.V., Pouzada A.S. (2007). Study of tribological properties of molds obtained by stereolithography. Virtual and Physical Prototyping. 2, 29-36. doi:10.1080/17452750701295765
[6] Salmoria G.V., Lafratta F.H., Biava M.M., Ahrens C.H. (2008) Rapid Manufacturing and Rapid Tooling of Polymer Miniaturized Parts Using Stereolithography. Journal of the Brazilian Society of Mechanical Sciences and Engineering. 30 (1), 7-10. http://dx.doi.org/10.1590/S1678-58782008000100002
[7] Salmoria, G.V., Ahrens .C.H., Villamizar F.A.Y., Sabino-Netto A.C. (2008). Influência do Desempenho Térmico de Moldes Fabricados com Compósito Epóxi/Alumínio nas Propriedades de PP Moldado por Injeção. Polímeros. 18 (3), 262-269. http://dx.doi.org/10.1590/S0104-14282008000300013
[8] Sabino Netto, A.C., Salmoria, G.V., Ahrens, C.H., Pouzada, A.S.(2008). Fritction Properties of Steel Fibre Reinforced Epoxy Composites used in Molding Blocks of Hybrid Molds, Materials Science Forum, 587, 217-221. doi:10.4028/www.scientific.net/MSF.587-588.217
[9] Salmoria G.V., Klauss P., Pires A.T.N. (2008). Investigations on cure kinetics and thermal degradation of stereolithography Renshape 5260 photosensitive resin. Polymer Testing. 27, 698–704. doi:10.1016/j.polymertesting.2008.05.008
[10] Sabino Netto, A.C., Salmoria, G.V., Ahrens, C.H., Pouzada, A.S. (2008). Mechanical properties of epoxy composites filled with short steel fibres for hybrid injection molds, Advanced Materials Forum, 587, 217-221. doi: 10.4028/www.scientific.net/MSF.587-588.222
[11] Salmoria, G.V., Ahrens .C.H., Beal V.E., Soldi V., Pires A.T.N. (2009). Evaluation of post-curing and laser manufacturing parameters on the properties of SOMOS 7110 photosensitive resin used in stereolithography. Materials and Design. 30, 758-763. doi:10.1016/j.matdes.2008.05.016
[12] Leite J.L., Rasteiro M.G., Salmoria G.V., Ahrens C.H., Pouzada, A.S. (2010). Epoxy/steel fiber composites-A simple model to predict the fiber sedimentation. Polymer Composites. 31, 1378-1386. doi:10.1002/pc.20923
[13] Martinho P.G., Sabino-Netto A.C., Salmoria G.V., Pouzada A.S, (2011). Hybrid Molds with Epoxy-Based Composites – Effects of Materials and Processing on Molding Shrinkage and Warpage. Internatinal Polymer Processing., 26 (3) 256-264. doi: 10.3139/217.2435
[14] Souza C.F.M., Leite J.L., Salmoria G.V., Pouzada A.S. (2013). Influence of graphite and carbon nanotubes on the mechanical and electrical properties of cast epoxy composites. Materials Science Forum. 730, 909-914.

doi:10.4028/www.scientific.net/MSF.730-732.909

[15] Neves A.F., Salmoria G.V., Ahrens C.H., Pouzada A.S., Silva M.A. (2013). Assessment of Injection Molded Parts of PP/Nanoclay Produced with Hybrid Molds. Materials Science Forum. 730, 963-968. doi:10.4028/www.scientific.net/MSF.730-732.963

[16] Leite J.L., Salmoria G.V., Ahrens C.H., Pouzada A.S. (2013). Characterization of epoxy/steel fibres composites for hybrid injection molds. Materials Science Forum. 730, 277-282. doi:10.4028/www.scientific.net/MSF.730-732.277

[17] Leite Howarth J.L., Pouzada A.S., Maia J.M., Salmoria G.V., Ahrens C.H. (2013). A study on fiber sedimentation velocity in epoxy/steel fiber composites used for hybrid injection molds. Journal of Composite Materials 47, 2436-2447. doi: 10.1177/0021998313509861