## MECHANICAL PROPERTIES OF COMPOSITES MATERIALS SIKACARBODURS512, SIKADUR 30 AND SIKADUR 330

D.A.Hernandez<sup>1</sup> , M.O.Orlandi<sup>1</sup> .

Department of Physical Chemistry, Institute of Chemistry Araraquara<sup>1</sup>, São Paulo State university (UNESP).

Prof.Francisco Degni 55 CEP 14800-060 Araraquara-SP. danyhdzb@hotmail.com.

### ABSTRACT.

Tensile tests at standard head displacement rate according to ASTM D3039/D3039M and ASTM D638-14 show that Carbon Fiber Reinforced Polymer (CFRP) plate and adhesive Epoxy have not a ductile behavior and they are not able to sustain several cycles of inelastic deformation since an elastic range governs its energy dissipation. When comparing the strength resistance of CFRP plate and its adhesive epoxy was found that, the adhesive epoxy has a tensile strength resistance of 1% of the CFRP plate tensile strength resistance, a young modulus of 7 % of the CFRP plate young modulus and a smaller strain at break than a CFRP plate. CFRP plate Micrograph analysis shows that cracks propagate through interfacials zones affecting the sharing of force matrix to carbon fibers. Epoxies Micrograph analysis shows fine granular particles bonded among them by covalent alloys, once the strength outstripped the covalent alloys, the material fails in a brittle manner.

Keywords: Composites materials, Mechanical properties, SEM, Sikacarbodur.

### INTRODUCTION.

The interest in FRP composite systems rise from the many drawbacks of poststrengthening structures with steel, the early work completed by the researchers' involved selecting fiber and adhesive types and findings feasible, safe employment of fiber systems. Researcher's initially debated the benefits and drawbacks of various fiber material and mechanical properties, including fiber ratios and pre-stressing, in addition to finding cost/saving ratios for civil engineering projects.

Composites materials have been studied regarding different cycles of loading at which they are subjected. Hassein [1] studied how temperature affects the strength and the fatigue life of steel beams strengthened with externally bonded CFRP plates, using Sikacarbodur S512, sikadudur 30, pre-impregnated laminate MTM 46/sts (24k) and MTM 46 epoxy resin. He determined the material mechanical properties at different temperatures and then the interfacial stresses in epoxy resin between CFRP plate and steel beam. In CFRP tensile test (prepreg laminate), he found that the samples failed suddenly without any sign of plastic deformation. However, the CFRP mechanical properties were assumed temperature independent for values of temperatures lower than matrix glass transition temperature (Tg). Moreover, tensile

tests for sikadur 30 dogbone specimens were carried out at different temperatures to obtain young modulus and he observed that sikadur 30 tensile strength is reduced, and nonlinear stress-strain behavior is obtained when the temperature becomes close and above Tg. For sikadur 30, glass transition temperatures (Tg) depends on the heating and loading rates, meaning that, sikadur 30 glass transition temperature cannot be an exact value. Additionally, two steel elements were bonded together using sikadur 30 and then a tensile load was applied to perform pull-off tests. These tests indicated a dependence in the resin bond strength with temperature.

Most investigations explore the resin bond behavior and its capacity because typically the adhesive of the FRP composite system fails first. Michels et al. [2], studied the influence of the curing condition (room temperature and accelerated curing) in glass transition temperature (Tg) of Sikadur 30 using Dynamic Mechanical Analysis (DMA) in the temperature range from -20 °C to 150 °C, using a constant heating rate of 0.5 °C/min. It was observed that glass transition temperature (Tg) is not a defined material property and it would vary depending on the definition considered for assessing it, and they found values of Tg in the region between 40 °C and 50 °C, as recommended values for civil structures designs. McNutt [3] applied spring theory using Sikacarbodur, sikaWrap Hex 230, sikadur 30, sikadur 330 and LTC 4300 in shear and tensile tests. These tests revel the tensile spring constant, young modulus, shear modulus and tension and shear capacity of each epoxy resin. He concluded that ignoring progressive failure of epoxy resin and assuming the epoxy resin behaves in a linear brittle manner is conservative. He also observed that the weakest component of the composite is the epoxy resin used as adhesive. M.D Gilchrist & N. Svensson [4] used SEM technique to study a fractographic features associated with delamination in multidirectional laminates of T300/914 carbon/epoxy using DCB,ELS,FRMM and MMB fracture mechanics coupons, composite, considering pure mode I, pure mode II and mixed modes I/II of loading under both static and fatigue conditions. From the results of the examination, a very large number of broken fiber seems to be a characteristic of mode I failure, a larger amount of resin debris are present due to fatigue loading than in static failure. Therefore, they characterized the different loadings modes using cusps angles and fiber pull-out on the fractures surfaces. Bradly [7] studied the effect of the moisture absorption on the interfacial strength of polymeric matrix composites using SEM technique, to determine their potential suitability for structural applications for continuous immersion in seawater. He observed that the moisture-induced degradation is associated with a decrease in the interfacial strength rather than the degradation of matrix mechanical properties. Jumahat [8] proposed a possible sequence of failure initiation and propagation of carbon fiber / toughened epoxy composites subjected to compressive loading based on SEM and optical microscopy observation of failed specimens. The micrographs revealed that the misaligned fibers failed in two points upon reaching maximum micro-bending deformation and two planes of fracture were created to form a kink band.

Considering the lack of research about failure surfaces using electron microscopy for civil engineering materials, in this work it was analyzed Carbon fiber reinforcement polymer (CFRP) and epoxy resins, used in retrofitting structures, where retrofitting any structure is required to overcome any external force that attempts to outstrip its capacity. First, standard tensile tests were done for composite material and for epoxy resins. Then to understand much better failure surfaces under tension tests, Scanning Electron Microscopy (SEM) was used to analyze the material before and after tensile tests.

## MATERIALS, EXPERIMENTAL PROCEDURE AND EQUIPMENT.

## Materials and experimental procedure.

In this work, Carbon Fiber Composite material Sikacarbodur S512 and polymer epoxies adhesives Sikadur 30 and Sikadur 330 were used. Composite material tensile tests were performed according to ASTM 3039/D3039M-14 standard and epoxy material tensile tests were performed according to ASTM D638 standard. First, we performed standard tests for the composite material, in this case Sikacarbodur S512, and then standard tests for epoxies adhesives. For standard test of Sikacarbodur S512, it was used a standard head displacement rate of 2 mm/min and the coupon follows the recommended dimensions shown in table 2 of ASTM 3039/D3039M-14 standard. Standard tensile test of epoxy material was performed using a standard head displacement rate of 0.5 mm/min and specimen type IV of ASTM D638.

### Samples Preparation.

SIKA Company manufactures CFRP Sikacarbodur S512, Sikadur 30 and Sikadur 330 epoxies adhesive. CFRP Sikacarbodur S512 plate was cut using a sheet metal cutting equipment (guillotine); the surface of sikacarbodur S512 was cleaned using acetone before sticking the tabs using Sikadur 30 adhesive, later an emery cloth was use to ensure a flat surface, tabs bevel angle of 90° was used. To prepare the epoxy adhesive samples the first step was to prepare the molds, which were made using acrylic sheet and a laser cut equipment. The components were mixed following datasheet procedure of each adhesive and pouring in the molds, they were removed after staying 7 days at 40 ° C. To uniform the thickness of the samples and to remove all the imperfection of the borders as marks of the die or any other imperfection an emery cloth was used. This procedure was followed until the required amount of samples was reached. All the samples were kept into a clean and close container at room temperature for more than one month until the tensile tests were performed.

For the microscopy analysis before and after tests, the samples were cut in small rectangular pieces using an electronic saw, with dimensions of 3mmx3mm for CFRP and 6mmx5mmx1mm for epoxy adhesive. Neither CFRP samples nor the epoxies adhesives samples were coated for the microscopy observation. Special cares were regarded when cutting the samples after test to avoid any modification or contact which could distort the failed side of the samples.

#### Equipment.

The equipment used for mechanical tests was a universal machine INSTRON/EMIC 23-200 from engineering department of UNESP-Bauru. Scanning electron microscopy (SEM) micrograph analysis were obtained using a field emission SEM (JEOL, model 7500F) equipment from Chemistry Institute in Araraquara. For CFRP tensile test it was used a load cell CCE100 kN, for gripping the samples were used jaws GR012 with a maximum capacity of 100 kN. For polymers tensile test it was used a load cell CCE2KN, for gripping the samples were used jaws GR003 with a maximum capacity of 2 kN and an electronic extensometer of 25 mm of gage length was used.

#### **RESULTS AND DISCUSSION**

For CFRP tensile test the specimen was positioned on the testing machine, and the rate of test was settled at 2 mm/min. Some minutes after the test started, the specimen failed. The failure occurred as shown in Figure 1, first with some delamination at zone A, then delamination at zone C, in opposite side of failure of zone A, and delamination process at zones A and C continues until zone B does not resist any load increment and it suddenly bursts. As the applied force is increasing, the specimen is being deformed and the resin, which bonds the carbon fibers, is being released and expelled, until the amount of bonded fibers remaining are not enough to resist any increment of force.

Sikadur 30 tensile test was performed using 4mm samples thickness at 5mm/min, which is the speed of testing specified in ASTM D638 for specimen type IV. However, that speed made the samples failed in less than 0.5 minutes. Therefore, a speed of testing of 1 mm/min and a specimen of 5 mm thick, was used to ensure the samples fail close to 1 minute of testing. The nominal straining rate, regarding a gauge length of 25 mm is 0.04 min-1 (6.67 sec-1). For sikadur 330 it was also used a 5 mm thickness samples.



Figure 1: Cracks propagation in CFRP samples

The stress-strain curves obtained from CFRP tension specimens are shown in Figure 2, presenting a linear relationship until failure. Some samples were discarded because they showed a premature failure due to slippage with grips machine and also due to fiber and matrix imperfections, and those manufacturing imperfections on materials were analyzed by microscopy. All the specimens failed suddenly without sign of plastic deformation as shown in Figure 2a. Therefore, Sikacarbodur S512 does not have plastic range, in terms of energy, the total dissipated energy is governed by the stored energy in elastic range, and Sikacarbodur S512 does not have recovery energy due to the lack of plastic range for ductile materials. However it presents high energy dissipation capacity which is a good indicator of performance. The average yielding stress obtained at room temperature was 3,051 MPa, which is higher than the value of 2,800 MPa shown in datasheet of Sikacarbodur S512 and the average young modulus was 165,235 MPa, which is also close to the value 165,000 MPa shown in datasheet of Sikacarbodur S512.

The Stress-Strain curves of sikadur 30 (Figure 2b) show a similar linear segment among samples at the beginning of the slope. However, ultimate tensile stress and ultimate strain are showing a little difference among them. The average young modulus was 12,049 MPa and the average tensile strength at break was 30 MPa. These values are close to the values reported in the material datasheet (11,200 MPa and 31.0 MPa respectively). Note: 1000 ( $\mu$ e) =0.001 absolute strain.



Figure 2: (a) CFRP stress-strain curves. (b) Sikadur 30 stress-strain curves.

Figure 3 presents the stress-strain curves of sikadur 330 and a linear behavior of the material is observed, some samples failed before reach its ultimate tensile strength due to bubble of air inside the necking region, however the young modulus was not affected as is observed in Figure 3. The average tensile strength obtained was 28 MPa and the average young modulus was 3745 MPa. Both values are lower than the values shown in datasheet, which are 30 MPa and 4500 MPa respectively. The obtained average elongation at break was 0.9%, which is similar than the elongation at break shown in datasheet. Although the obtained values were lower than the values reported in datasheet, they were considered accurate because they were obtained at similar condition that could be found in any other project, i.e., without any special processing.

When comparing stress-strain behavior of sikadur 30 and sikadur 330 it is noticed that, sikadur 330 has better behavior than sikadur 30 since both have almost the same tensile strength at break but sikadur 330 has the biggest elongation at break, more than twice. Moreover, Sikadur 330 has better energy dissipation capacity as can be observed in Figure 3. However, both resins are reported presenting similar values in material datasheets and differences in tensile behavior are not as remarkable as the results found in this study, which shows similar results with those presented by Hassein [1] at 40 °C when elongations at break are considered. Therefore a deeply study is recommended to assure the tensile elongation at break behavior. Both resins present two region, the first one is

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characterized by an elastic behavior and the second one is characterized by a nonlinear viscous behavior. A deeply study of these regions will define the yielding point of both resin and these values are not defined in datasheet values.



Figure 3: (a) Sikadur 330 stress-strain curves (b): Adhesive stress-strain curves relationship

## SCANNING ELECTRON MICROSCOPY (SEM) MICROGRAPH ANALYSIS.

### CFRP Micrograph Analysis.

Figure 4 presents a typical SEM micrograph of the longitudinal distribution of fibers in lamina sikacabodur S512. It is observed that the fiber diameter remains constant all over the length of the plate and that the resin layer changes in thickness because depends on fibers and resin arrangements which is a random arrangement depending of pultruded manufacturing process. Some longitudinal fibers discontinuities and misalignments were found (Figure 4), and this may influence some mechanical properties differences on tensile tests.

In the cross-section image of Figure 4, it was observed that fibers are close packaged. The resin is covering the fibers, although some localized cracks and bubbles can be observed. This effect may be attributed either to the processing of material or the SEM sample preparation. However, regarding Figure 4(a) where fiber discontinuities (shown by arrows) and fiber misalignments (shown by arrows A and B) are observed, these defects could not be provoked by SEM samples preparation. Some fibers discontinuities (but not all of them) might be caused while CFRP plate transportation, considering that an external side of the plate is analyzed. However, fiber misalignments in the same figure (shown by arrow A and arrow B) could not be provoked while CFRP plate transportation. Moreover Figure 4(b) indicates the presence of both, some carbon fiber and interfacials cracks (shown by arrows), whereas Figure 4(c) is showing a cross section without cracks in carbon fibers. Therefore, it is noticed that fiber discontinuities and fiber misalignments are neither provoked while SEM samples preparation nor CFRP plate transportation but while CFRP plate manufacturing.

A volume fraction of carbon fiber of 63% and 37% for resin were calculated using Figure 4(d), and an average carbon fiber diameter of 6.66  $\mu$ m was measured using microscopy tool. This means that Sikacarbodur S512 has enough carbon fiber volume to ensure the maximum mechanical properties at lowest cost.



Figure 4: (a) SEM image of longitudinal direction of Sikacarbodur S512 material. (b) SEM cross-section image of Sikacarbodur S512 material. (c) SEM cross-section image of Sikacarbodur S512 material without defects. (d) SEM image of carbon Fiber volume fraction.

### Carbon Fiber Standard Tensile Test Failures Surfaces.

Figure 5 shows Sikacarbodur S512 after test, and the observed failure is typically of a brittle material where failure occurs in a suddenly way. Figure 5(b) presents SEM micrograph analysis showing that the fiber failure surface is nonplanar and irregular with serrated aspect; no necking shape was observed. It is important to notice that no shrinkage in diameter was observed after tests, assuring a brittle fracture of fibers. Figure 5(c) shows the interfacial crack propagation before matrix debonding. The matrix does not transfer in a uniform way all the forces among fibers and some interfacial cracks are avoiding the force to be transferred in a properly way. Figure 5(d) shows matrix debonding which is a process following the interfacial crack propagation. Figure 5 shows rough surfaces after test. Moreover shows that the failure direction is perpendicular to a fiber and load direction. The surface after test is non-planar and irregular; bsides, some carbon fiber failures are consequence of fiber manufacturing defects as misalignments, discontinuities and cracks in fiber surface. As shown in Figure 5 cracks propagate through interfacial zones (contact zone between fiber and resin), affecting the force transferred to carbon fibers then matrix debonding occurs and finally carbon fibers are pulled out, that is because Sikacarbodur S512 plate is not a ductile material and its matrix cannot transfer properly the force among fibers.



Figure 5: (a) SEM image of carbon fiber failure surfaces.(b) SEM image of carbon fiber failed cross sections.(c) SEM image of interfacial crack propagation.(d) SEM image of Matrix debonding.

#### Sikadur 30 Micrograph Analysis.

Figure 6 shows a cross section backscattered electron image of sikadur 30, where it is possible to identify two different phases. Figure 6(a) shows a predominated granular phase, while Figure 6(b) presents a predominated continuous phase and Figure 6(c) is the interfacial phase (located in the interception zone between granular phase and continuous phase). Some defects are observed in the cross section; those ones are due to trapped air bubble during the samples preparing procedure and could be avoided if a vacuum environment system is settled during the preparing procedure. However, regarding that during the application of the material in any retrofitting project is not common the used of any vacuum system, those defects were considered as a normal consequence of the procedure followed to prepared the samples, and mechanical properties must be determine taking them into account.



Figure 6: SEM image of Sikadur 30 cross section surface before tensile test.(a)SEM image of granular phase (b) SEM image of continuous phase.(c) SEM image of interfacial phase.

Figure 7 shows SEM images of Sikadur 30 after tensile test, presenting rough, irregular and nonplanar surface, with superficial particles bonded by the continuous phase of the resin. Figure 7(a) shows the granular particles are composed by a continuous phase of the resin coated by fine granular particles. Figure 7(b) shows a mixing between fine granular particles and continuous phase of the resin. Figure 7(c) shows that there is a part of the cross section in the cast where is composed just by the continuous phase of the resin.

Before tensile test there was a part in the cast cross section governed by fine granular particles (Figure 6(a)) however once the surface became rough and irregular, that part was mixed with the continuous phase of the resin changing its morphology (Figure 7a and b). The continuous phase remains constant but some cracks are observed as consequence of strength concentration.



*Figure 7: SEM image of Sikadur 30 failure surface. (a) SEM image of granular particles. (b) SEM image of mixing of fine granular particles and continuous phase. (c) SEM image of continuous phase.* 

#### Sikadur 330 micrograph analysis.

Figure 8 shows that sikadur 330 has a homogenous distribution of phases, its granular phase and its continuous phase are scattered in a uniform way as observed in Figure 8(a) which assure that Sikadur 330 has fine granular particles coating the continuous phase of the resin.



Figure 8: SEM image of Sikadur 330 cross section surface before tensile test. (a) Zoom of red rectangle showing granular particles and continuous phase of the resin.

Figure 9 shows some micro cracks propagation in the continuous phase and surrounding the granular phase (shown by arrows). Figure 9(a) shows fine granular particles scattered over the continuous phase of the resin after tensile test, as consequence of the internal movement (heat) of fine particles once they are subjected to a tensile force. Figure 9(b) shows the continuous phase coated by fine granular particles as observed in Figure 8(b) therefore, the sample failed before the tensile force reach this region of the sample.



Figure 9: SEM image of Sikadur 330 cross section surface after tensile test. (a) SEM image of granular phase (b) SEM image of continuous phase.

### CONCLUSIONS.

After analyzing Stress-strain curves of Sikacarbodur S512 obtained, it was found that Sikacarbodur S512 has not a ductile behavior and it is not able to sustain inelastic deformation since an elastic range governs its energy dissipation. On the other hand, structural seismic design theories are based on the assumption that a significant amount of inelastic deformation will take place during a design earthquake. Therefore, special cares must be taking into account when recommending Sikacarbodur S512 as retrofitting or reinforcement material for elements where seismic hazard is high. As expected, Sikadur 30 is a brittle resin and has a tensile strength resistance of 1% of Sikacarbodur S512 tensile strength resistance, a young modulus of 7 % of the Sikacarbodur S512. That means that the weakest material in a strengthened member is sikadur 30.

Sikadur 30 and Sikadur 330 stress-strain curves show that they do not have a significant plastic range and their main behavior is in the elastic range, therefore their elastic range governs the dissipation energy capacity. Sikadur 330 presented a better tensile behavior than Sikadur 30. Considering the low tensile strength resistance and young modulus of each of them (when compared with Sikacarbodur S512), the CFRP retrofitting method selected must increase the maximum nominal load and

ensure all the materials of the adhesive joint will behave in a uniform way (similar strain rate) and will be able to sustain the maximum ultimate loads.

Sikacarbodur S512 micrograph analysis shows that cracks propagate through interfacial zones (contact zone between fiber and resin), affecting the load transferring to carbon fibers then matrix debonding occurs and finally carbon fibers are pulled out, that is because Sikacarbodur S512 plate is not a ductile material and its matrix cannot transfer properly the force among fibers. Micrograph analysis before and after test of Sikadur 30 and Sikadur 330 shows that for both resin there are the continuous phase and the granular one. Once they are subjected to tensile forces the fine particles are scattered throughout the continuous phase as consequence of micro crack propagation, which explain some observed parts presenting a continuous phase and other parts with different granular phase concentration. Micrograph analysis also shows granular particles bonded among them by covalent alloys (continuous phase of the resin), which once the strength outstripped those alloys, the cast suddenly failed.

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