Micromechanics of Stress Transfer across the Interface fiber-matrix bonding

Fatiha Teklal, Bachir Kacimi, Arezki Djebbar

Abstract— The study and application of composite materials are a truly interdisciplinary endeavor that has been enriched by contributions from chemistry, physics, materials science, mechanics and manufacturing engineering. The understanding of the interface (or interphase) in composites is the central point of this interdisciplinary effort. From the early development of composite materials of various nature, the optimization of the interface has been of major importance. While there are many reference books available on composite materials, few of them deal specifically with the science and mechanics of the interface of fiber reinforced composites. Even more important, the ideas linking the properties of composites to the interface structure are still emerging. A number of experimental techniques have been devised to measure the mechanical properties of the fiber-matrix and laminar interfaces in composites. A number of analytical solutions have been proposed in order to better understand the stress transfer mechanism across the interfaces between the fiber and the matrix. In our study, we need a direct characterization of the interface; the micromechanical tests we are addressing seem to meet this objective and we chose to use two complementary tests simultaneously. The microindentation test that can be applied to real composites,and the drop test, preferred to the pull-out because of the theoretical possibility of studying systems with high adhesion (which is a priori the case with our systems). These two tests are complementary because of the principle of the model specimen used for both the first "compression indentation" and the second whose fiber is subjected to tensile stress called the drop test. Comparing the results obtained by the two methods can therefore be rewarding.

Keywords— Interface, Micromechanics, pull-out, Composite, Fiber, Matrix.

Fatiha Teklal was with the Mechanics, Structures and Energetics Laboratory (L.M.S.E), University of Mouloud MAMMERI on Tizi Ouzou, BP 17 RP 15000, Tizi Ouzou, Algeria. (Phone: 213 663 255 507; corresponding author: fatihagm07@yahoo.fr).

Bachir Kacimi is with the Mechanics, Structures and Energetics Laboratory (L.M.S.E), University of Mouloud MAMMERI on Tizi Ouzou, BP 17 RP 15000, Tizi Ouzou, Algeria. (Phone: 213 771 771 122; e-mail: kacimiummto@yahoo.fr).

Arezki Djebbar is with the Mechanics, Structures and Energetics Laboratory (L.M.S.E), University of Mouloud MAMMERI on Tizi Ouzou, BP 17 RP 15000, Tizi Ouzou, Algeria. (Phone: 213 555 274 501; e-mail: ar.djebbar@yahoo.fr).

I. Introduction

Fiber-reinforced composites (FRCs) are widely used in advanced engineering applications due to their low specific weight and superior thermo-mechanical stability. Furthermore, the bi-material nature of FRC can be exploited to advantageously tailor the properties according to application requirements. The study of the mechanical behavior of composite materials often boils down to two basic constituents, the matrix and the reinforcement. While there is an abundant literature on composites, studies on the influence of the interface or the interphase are much sparser. The notion of interface or interphase remains relatively vague, as the interfacial zone does not exist in itself but is created during the implementation of the composite. Therefore, it appears very difficult to assign mechanical properties to it. One of the most important phenomena in FRCs is the stress transfer between the fiber and the matrix across the interphase/interface. When composites are subjected to various loading conditions, the efficiency of load transfer across the interface plays an important role in overall performance of the composites [1, 2]. However, these zones (interface / interphase) play a leading role, as shown by Drzal 1986 [3] and Piggott 2004 [4], since the interface and / or interphase ensure the transmission of the forces between the relatively soft matrix and the stiffer reinforcement. Consequently, the contribution of the reinforcement on the mechanical properties of the composite is directly related to the quality of the interfacial zone [5]. Kim et al. [6, 7] showed that a thorough understanding of the interfacial zone is considered as one of the criteria for composite design.

To develop tractable models, many researchers have modeled the interphase region as a homogeneous material [8-12]. However, a few studies considered the inhomogeneous nature of interphase adopting a stair-case variation of material properties across the thickness of the interphase layer [13, 14]. Alternatively, a few investigators proposed an effective interphase model (EIM) and uniform replacement model (URM) to replace the fiber and the surrounding interphase by an effective homogeneous fiber in order to convert a three-phase composite into a two-phase composite [15]. For mathematical convenience and to better describe the variation of properties within the interphase region, several researchers treated the interphase as an inhomogeneous material by smoothly varying the material properties as a function of radius. Usually in such models, the material properties are graded by adopting an empirical law [16-21].

II. PRINCIPLE AND INTEREST OF THE DROP TEST

The drop test is different from that of heaving the particular configuration of the samples: here, the fiber is embedded in a resin micro drop deposited on the monofilament before cooking. During the tensile test, it is maintained by using two plates (Fig.1. a). Can, by this technique, reach lengths of very low entrenchment, up to 30 µm, which is rarely possible pull-out. The only limiting factor is the test, in the case of a thermosetting resin, the initial viscosity of the resin, if it is too high, prevents the deposition of small drops. Finally, the drop test allows, from the pull-out, achieving relatively fast for a large number of samples (the implementation of these do not require specific mounting) [22]. Fig.1.b shows the curves obtained from tensile testing of gout. As in pull-out, these curves to determine the strength of groundwood F_d. The whole problem is then to relate this experimental scale to a specific parameter of the interface [23-25].

A. Geometry of the drop and put into equation

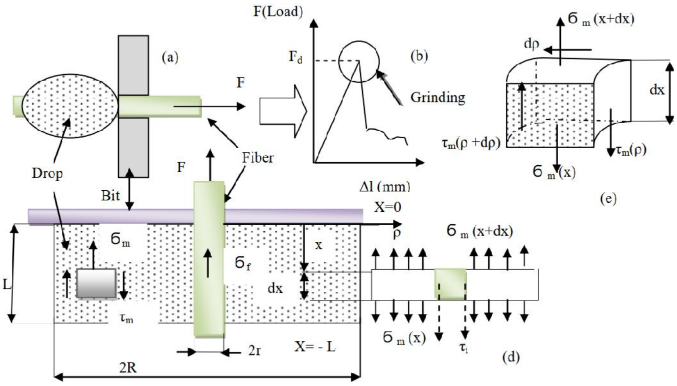

Fig.1 Model of the drop/Fiber (a); load- displacement curve (b); balance of forces on a section (c), (d) and (e).

Thus, it appears that the actual geometry of the test can be modeled as shown in Fig.1.a, a cylinder of length L represents Gout, or L is the length of entrenchment, but the length of the drop. Index, "m", "f", refers to the matrix and the fiber, τ means the shear stress at the interface that is to say $\rho = r$. We put in elastic, linear, with axial symmetry (no twist). We assume that the axial stresses in the matrix σ_m, and in the fiber σ_f, radial effects are negligible. These effects include swelling of the matrix and fiber contraction due to the effects of Poisson's ratio.

B. Setting equation

Let us now apply the balance of forces on various parts of the system, Fiber + drop (Fig.1-a). The balance of forces operating on the fiber we get the whole equation (1).

$$F = \int_{x=-L}^{x=0} (2\pi r)\tau_i(x)dx \quad (1)$$

$$\frac{d\sigma_f}{dx} = -\frac{2\tau_i(x)}{r} \quad (2)$$

$$\tau_i'' - \alpha^2 \tau_i = 0 \quad (3)$$

The resolution of equation (3) gives the evolution of shear stresses at the fiber/matrix interface τ_i (x), the normal stress at the fiber level σ_f and at the matrix level σ_m, are given by the equations (4), (5) and (6) respectively:

$$\tau_i(x) = \frac{-\alpha F}{2\pi r(ch(\alpha L)-1)} sh[\alpha(x+L)] \quad (4)$$

$$\sigma_f = \frac{F[ch(\alpha(x+L))-1]}{\pi r^2[ch(\alpha L)-1]} \quad (5)$$

$$\sigma_m = \frac{-F[ch(\alpha(x+L))-1]}{\pi(R^2-r^2)[ch(\alpha L)-1]} \quad (6)$$

For our simulation we used a calculation software Matlab. The graphical representation of equations (4), (5) and (6) depending on the length embedded in Fig. 2 and 3. We chose for our simulation; thermosetting epoxy matrix (drop) in diameter, 2R = 30µm whose mechanical properties (E_m = 4.5 GPa, G_m = 1.6 GPa) and two types of E-glass filament (r = 4µm, E_f= 73 GPa) and carbon-HT (r = 3.5 µm, E_f= 230 GPa).

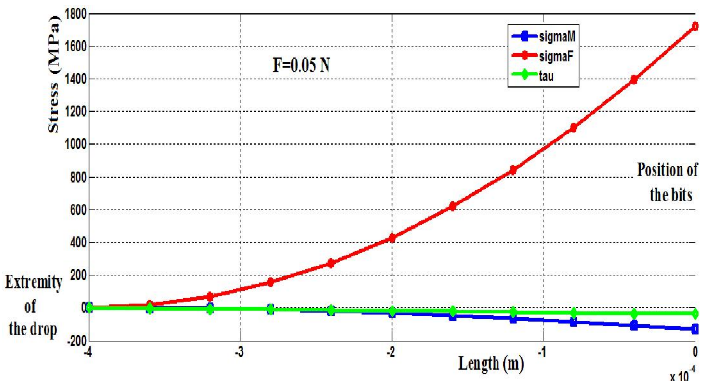

Fig.2 Evolution constraints [$\sigma_f, |\sigma_m|, |\tau_i|$] based on the embedded length for an applied force of 0.05 N (drop test, epoxy / glass).

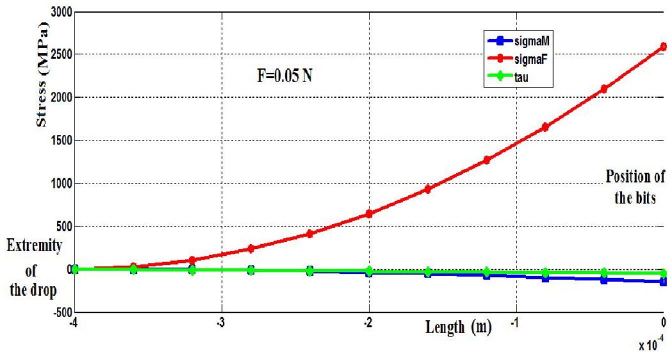

Fig.3 Evolution constraints [$\sigma_f, |\sigma_m|, |\tau_i|$] based on the embedded length for an applied force of 0.05 N (drop test, couples epoxy / carbon).

The evolution of profile of the stresses [$\sigma_f, |\sigma_m|, |\tau_i|$] according to the length enchased for the various values of the load applied are represented by Figs. 2 and 3. From the plotted curves we find that evolutionary constraints [$\sigma_f, |\sigma_m|, |\tau_i|$] is the same for both types of fiber (carbon, glass). The value of the stress σ_m of the matrix and shearing of the interface τ_i varies in a decreasing way with the length of the enshrining until they become null; same for the longitudinal stress fiber σf which decreases with the length embedded. Through the results obtained for the test of drop, we could highlight:

The high values of τ_i obtained for the various couples (carbon/epoxy, glass/epoxy) are not due to a bad evaluation of τ_i nor even to a numerical overvaluation of the force applied.
It is for this reason that the rupture occurs at the interface rather than in the matrix (for lengths of 45µm to 125µm entrenchment). Several explanations can be raised: It appears that the shear stress decreases rapidly away from that of the fiber; this then means that only the interfacial zone is subject to strong constraints, and this could pose a greater resistance than the matrix. According to the results we see three cases that appear:

For the first case when $\tau_i > \sigma_m$ two cases may appear, strong adhesion of the material tested, or due to modeling error. The characteristics of the interface are higher than those of the matrix and what are the properties of the latter that restrict the behavior of the composite. In this case we can not characterize the interface and this case is not real.

For the 2nd case $\tau_i \approx \sigma_m$ values of the embedding length ranges from 135µm to 150µm the two curves show the stress at the interface and the normal stress at the matrix are close to the two lines of evolution and superimposed. In this case the interface behavior follows that of the matrix.

For the 3rd case $\tau_i < \sigma_m$ lengths embedding 400µm to 175µm variants of σm values obtained for the matrix are larger than the interface; the characteristics of the interface are lower than those of the matrix and thus constitute the weak point at the origin of the rupture; this is the case we will consider in the tests because he represents the reality.

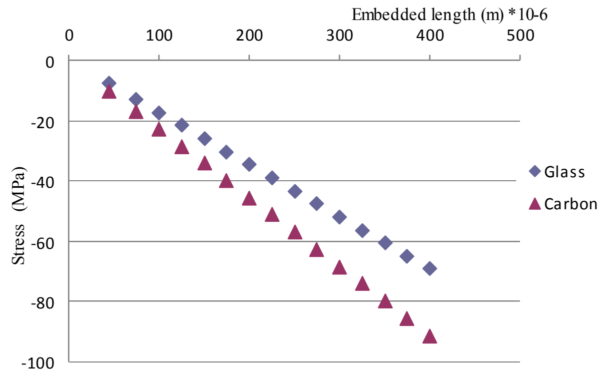

Fig.4 evolution of maximum interfacial stresses as a function of embedded length for a constant force F = 0.09 N for the drop test.

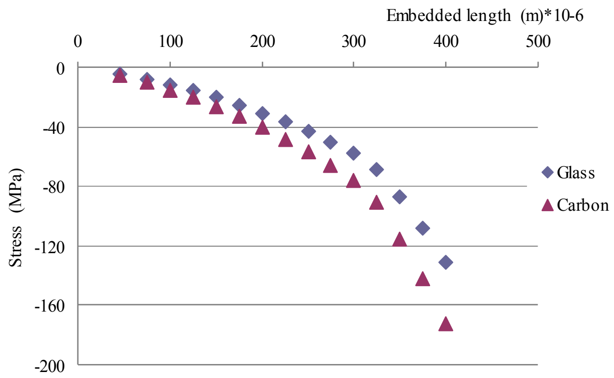

Fig.5 Evolution of maximum interfacial shear stress as a function of embedded length

The maximum stresses at the interface for a constant force of 0.075 N and 0.09 N evolve linearly with the length embedded, such as the shear stress $\tau_{i,max}$ is high for carbon/epoxy (Fig. 4 and 5). The stresses operate in the same way for the two couples, and the maximum stress is more important for carbon/epoxy (Fig.5). These values are indeed greater than the shear strength of epoxy τ = 80MPa; if they represented really interfacial resistance, an interfacial failure could occur, matrix having sold well before. However, we can wonder whether the value of τ, determined by a macroscopic mechanical test on pure resin, really corresponds to resistance to local intrinsic rupture of material. In a massive test-tube, the final rupture generally intervenes by the propagation of a fissure started on a defect, however, in the vicinity of this defect, leading to the rupture of material, is much higher than the measured nominal stress. The apparent discrepancy between our estimate of the interfacial strength and matrix strength is probably less important than it seems.

III. PRINCIPLE AND INTEREST OF INDENTATION TEST

It allows a measure of the force of cohesion in situ real composite (mainly one-way). This method currently has a process of taking action and fully automated data acquisition [26]. It requires the polishing of samples of composites having fibers perpendicular to the surface, and consists of driving, using an indenter on the end of a fiber to fiber debonding of the sheath matrix, to access a feature of the fiber / matrix interface. The final docking of the indenter in the surrounding matrix constitute a criterion for stopping the test [25], [27].

The stress measurement of decohesion σ_d then makes it possible to deduce interfacial resistance to the shearing τ_i which is a rather complex function of σ_d, elastic characteristics G_m of the matrix and E_f of fiber, diameter d of fiber and distance interfibres T_m (Fig. 6 et 7) [23].

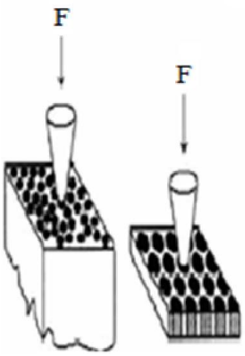

Fig. 6 test of indentation

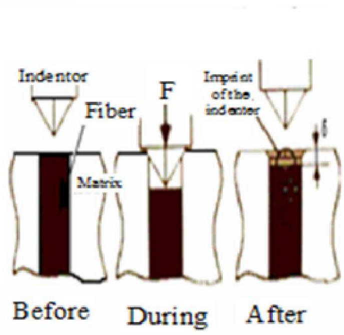

Fig. 7 Schematic of the indentation test according MARSHALL [1984].

A. Analytical modeling

In this model, we have developed an analytical approach that is simpler to implement than in the numerical method and that allows a more direct interpretation of the physical phenomena that are supposed to take place during the tests [28]. The model was based on the work of Piggott, 1980 (themselves influenced by earlier Greszczuk work, 1969), which already took into account the effect of inter-fiber distance but in an overall voltage configuration of the composite specimen. . The Piggott model has been modified by introducing loading conditions and boundary conditions representative of the test conditions. We have considered a perfect hexagonal arrangement of fibers and introduced the notion of equivalent radius, R_{eq}, to reduce the initial geometry to an equivalent value (Fig.8) (the introduced geometry is an axisymmetric model formed of concentric cylinders).

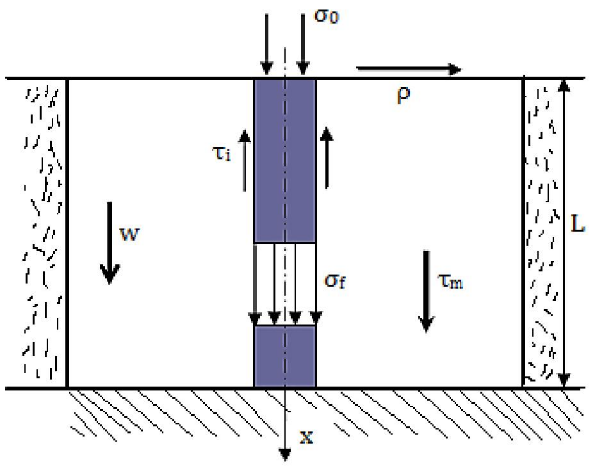

Fig.8 Equivalent geometry (section)

The equivalent radius is defined by: $\pi R_{eq}^{\ 2} - \pi r^2 = A$

Where r is the fiber radius and A is the matrix area contained in the circle of radius R.

It is assumed that the longitudinal displacement is zero at the "equivalent" fiber/matrix interface (at a distance Req) since tests showed that the bordering fibers did not move and that the interfaces were not damaged. Using Piggott's approach, this leads to the differential equation (Eq.7):

$$\frac{d^2\sigma_f}{dx^2} = \frac{n^2}{r^2}\sigma_f \qquad (7)$$

Where $$n^2 = \frac{2G_m}{E_f Ln\left(\frac{R_{eq}}{r}\right)}$$

σ_f is the fiber longitudinal stress, E_f the fiber Young's modulus and G_m the matrix shear modulus. The solution is done from Eq.8.

$$\sigma_f = Bsh(nx/r) + Dch(nx/r) \qquad (8)$$

To write the boundary conditions, it is assumed first that σf is homogeneous on a section of fiber (even on the upper surface) and second that L>>R, where L is the thickness of the sample. We obtain at x=0, $\sigma_f = \sigma_0 = -F/\pi r^2$ and at x=L, $\sigma_f = 0$. Thus (Eq.9):

$$\sigma_f = -\sigma_0\left[ch\left(\frac{nx}{r}\right) - \coth\left(\frac{nL}{r}\right)sh\left(\frac{nx}{r}\right)\right] \qquad (9)$$

If τ_i is the interfacial shear stress, then the equilibrium force on a fiber section leads to Eq.10.

$$\tau_i = -\frac{r}{2}\frac{d\sigma}{dx} \quad (10)$$

This gives (Eq. 11).

$$\tau_i = \frac{n\sigma_0}{2}\left[sh\left(\frac{nx}{r}\right) - \coth\left(\frac{nL}{r}\right) ch\left(\frac{nx}{r}\right)\right] \quad (11)$$

τ_i is maximum at x=0 and $\tau_{i\max} = \frac{n\sigma_0}{2}\coth\left(\frac{nL}{r}\right)$ and as $L/r \to \infty : \tau_{i\max} = \frac{n\sigma_0}{2}$ (indeed, experimentally L=1cm and R=10µm).

Then, for F=F_d, $\tau_{i\max} = \tau_i$, where τ_i is the interfacial shear strength (Eq.12); thus:

$$\tau_i = \frac{F_d}{2\pi r^2}\sqrt{\frac{2G_m}{E_f Ln\left(\frac{R_{eq}}{r}\right)}} \quad (12)$$

B. Determination of R_{eq} :

The real neighborhood of a fiber is different from the idealized case: the nearest fibers are positioned at various distances and generally, they do not have the same diameter. The tests performed induce only a partial debonding of each indented fiber; this means that the model presented above can be applied. The equivalent radius, R_{eq}, is then defined (Eq.13):

$$\frac{\theta R_{eq}^2}{2} = \frac{\theta r^2}{2} + A_m \quad \text{or by} \quad \frac{\theta R_{eq}^2}{2} = \frac{\theta R^2}{2} - A_f \quad (13)$$

Am (resp. A_f) is the area of matrix (resp. of fiber) included in the sector of angle θ and radius R (Fig.9). A_f is approximated by (Eq.14):

$$A_f = \frac{(\pi-\theta)c^2}{2} + cR\left(1-\cos\left(\frac{\theta}{2}\right)\right) \quad (14)$$

So :

$$R_{eq}^2 = R^2 + c^2 - \frac{2cR}{\theta}\left(1-\cos\left(\frac{\theta}{2}\right)\right) \quad \text{where}$$

$$\theta = 2Arc\sin\left(\frac{c}{R}\right) \quad (15)$$

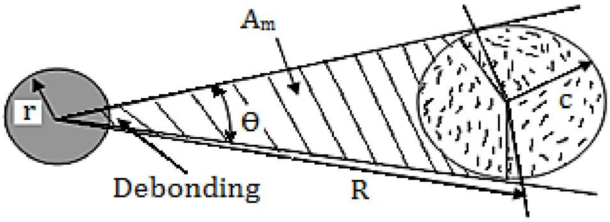

Fig.9 Local determination of the equivalent radius

For our simulation we used a computation software Matlab. The graphical representation of equations (11) and (12) depending on the length embedded Fig. 10 We have chosen for our simulated; an epoxy thermohardening matrix, whose mechanical properties (E_m = 4.5 GPa, G_m = 1.6 GPa) and two types of glass E fiber (r = 4µm, Ef = 73 GPa) and carbon HT (r = 3.5 m, E_f = 230 GPa). We varied the length of embedding tests of 45 µm to 400 µm, the loads applied maximum F_d of 0,05N to 0,17N and the radius equivalent to the radius of the fiber (Req/r) from 2,3 to 6,93.

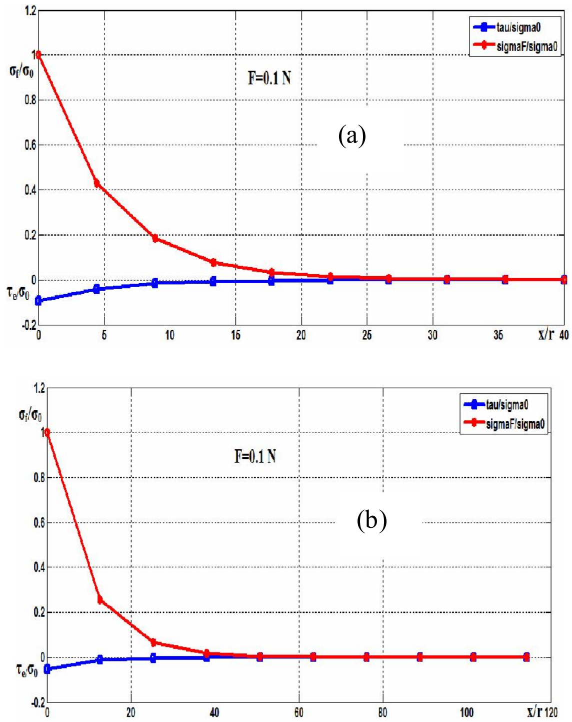

Fig. 10 Profile of the stresses in a test of indentation for fiber glass (a) and carbon (b) of Req/r=2.3 and F=0.1 N, L=400 µm

From the plotted curves we see that the evolution of stress ratio and depending on the ratio x / r is the same for both types of fibers (carbon, glass). In the light of the results obtained for the test of indentation of the two material couples, we have raise the following observations:

The value of the ratio of shear stress at the interface / strain at the top of the fiber (τ_i / σ_0) varies in a manner with decreasing the ratio of embedded length / radius of the fiber (x / r) until they become null; same for the value of the ratio of the longitudinal stress of the fiber / stress at the top of the fiber (σ_f / σ_0) which decreases the ratio of embedded length / radius of the fiber (boundary conditions).

The simulated tests with variable load (F=0,05N to 0,17N) highlighted, the existence of a grinding force (F_d) strength below which no slippage of the fiber is possible. One can see results of test of microindentation simulated for the two couples of samples of composite (glass/epoxy, carbon/epoxy). This force of separation (F_d) is a parameter essential and present the force necessary to break the interfacial connection fiber/matrix; indeed, the couples /matrice fiber which we study present a whole an interface to strong adhesion. It is thus necessary to break this bond before inducing any interfacial slip. during deformation in compression of the fiber will cause, at the interface, same displacement on the level of the matrix (shearing of the matrix) surrounding, will therefore be sheared between the fiber and pressed its nearest neighbors, which, they, only are little affected by this state of stress; the matrix, much less rigid than the fiber, adapt the stress by shearing.

From the plotted curves we note the influence of interfiber distance is clear that, if the aforementioned decreases, the same displacement of fiber generates a rate of higher shearing, thus a more important shear stress interfacial, the grinding will thus be observed more easily than in the case of a strong layer of matrix interfibre especially in the case of materials with glass fiber. We note that the values of the interfacial stresses obtained are higher for the couple glass/epoxy than for carbon/epoxy. The analytical modeling, which we developed thus, is adapted perfectly to the characterization of the composites with carbon and glass fibers for the test of indentation.

IV. Conclusion

Micromechanical tests developed so far, have kept a share of simplicity and specific characteristics; such as type of stress, the dimension and nature of specimens and the boundary conditions. These tests allow a qualitative study of the interface. Based on these tests, interface stand for values below the calculated interfacial resistance but the reality is that the interfaces become damaged at values far below those found numerically for parameters not considered in the assumptions may intervene in the case of finished products. While this study particularly interesting to validate the experimental results of tests. In addition, it will optimize couples reinforcements / matrix and determine the effect of surface treatment of the reinforcement.

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