IDENTIFICATION OF GLASS FIBER DEFECT DENSITY USING MULTIFRAGMENTATION TEST

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SUMMARY: This article presents a method to interpret the data issued from a multifragmentation test. The goal of this experiment is to evaluate the defect concentration along the fibre. The coupon is constituted by a fibre embedded with matrix which transfers the load along the fibre so that many fibre breaks appear during the test. Because of the length of the load transfer, the gauge length varies during the test. Moreover the most critical defects hide other less critical defects. Then the estimation of the concentration of fibre defects is not direct. The paper presents a methodology and a transformation to interpret the test results. Based on a numerical simulation, the transformation takes into account both process of hidden defects and variation of gauge length. Numerical tests are presented to prove the efficiency of the method and finally an experimental test is interpreted.

KEYWORDS: defect density, multifragmentation test, results interpretation, test simulation, hidden defects, variant gauge length.

INTRODUCTION

Probabilistic models [2, 8, 6] to estimate the composite materials strength, need input data coming from experimental tests. Among them, the defects distribution along the fiber is certainly the most influent property in these modelisations. This property due to the manufacturing, the stock, and handling of the raw material is a physical characteristic of the fibres. There are three methods used to determine this distribution: Test on one fiber, on fiber tuft [14] and multifragmentation test on monofiber coupons [4]. In the two formers, the accessibility to the various populations of defects depends on the coupon length. According to the frequency of the most critical defects, the population of mean defects that may influence also the crack properties of the composite is or is not accessible. Moreover, extrapolation of a distribution using the scale effect to estimate the distribution of a smaller fiber length, gives about 50% of error [9]. If the length of the coupon is so that a low strength defect is surely present in the coupon, then the mean properties are out of the possible scope of the test. Moreover, the influence of the various defect changes also in function of the capability of the matrix to transfer load around fibres breaks. Then it is really difficult to know the part of the defect population which really influences the crack before knowing the model results. In this way, probabilistic model applied to a glass E / epoxy composite [6] has demonstrated the influence of the mean population of defects. Then it is inaccurate to limit the study to the most critical defects by choosing unsuitable tests.

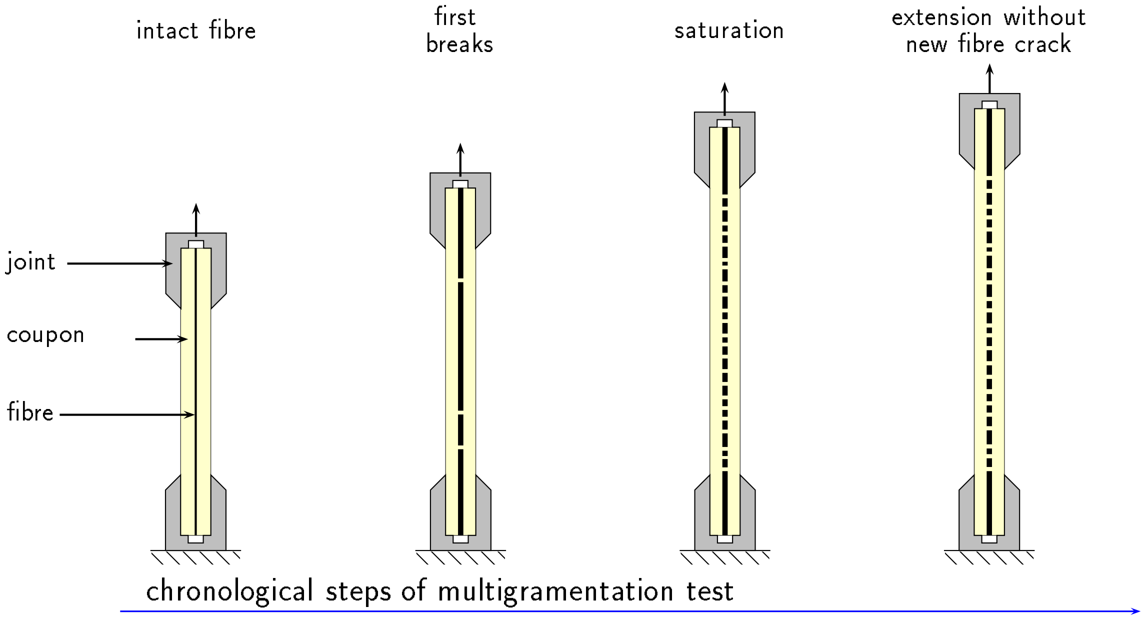

Figure 1: *Various steps of the multifragmentation test*

The multifragmentation test is able to give results over a larger population. Because of the composite nature of the test (a fiber embedded in matrix), experimental output are physically and theoreticaly linked with input of the probabilistic model. Moreover the scale of transfer length is the same in multifragmentation test and probabilistic models. On the other hand, besides its execution, one of the test difficulties is the statistical interpretation of results. Results are not a variable depending only on a coupon but they are echos associated with fiber-strain with an inconstant jauge length. Then a coupon provides many results and the last results are influenced by the previous ones. So, we need to discover through a phenomenum hiding some data, the law of strength along the fiber. The various methods actually used [1, 7, 9, 11, 13], are difficult to apply or are unable to interpretate the all results.
So we have tried to improve the interpretation of multifragmentation test results. After a short presentation of the test, we presents a new method and its use on glass fiber in the laboratory.

EXPERIMENTAL RESULTS

The multifragmentation test coupon is composed by a fiber embedded by resin. This coupon is charged in the fiber axe. The resin limit strain must be higher than fiber one; authors [3, 15] recommand a factor equal to 3, but it is impossible for some particular fibers. So the most deformable resin must be choosen. During the test, matrix transmits strain to the fiber by the shear stress until the apparition of the first break. At the break, the stress is null in the fibre, but because of the transfer of strength due to the matrix [5], the fibre is reloaded after a transfer length $\delta/2$. Then during the test, several breaks occur in the fibre on the defects location by decreasing order of criticism magnitude. But, because of transfer length around fracture, some fibre length are never stressed and never broken. Finally, the break number reaches a maximum n_t when all fiber bits are smaller than the transfer length. Strain increases, but the strain transfer is no more strong enough to cause another break: it is the phase of saturation. These steps are shown in figure 1.

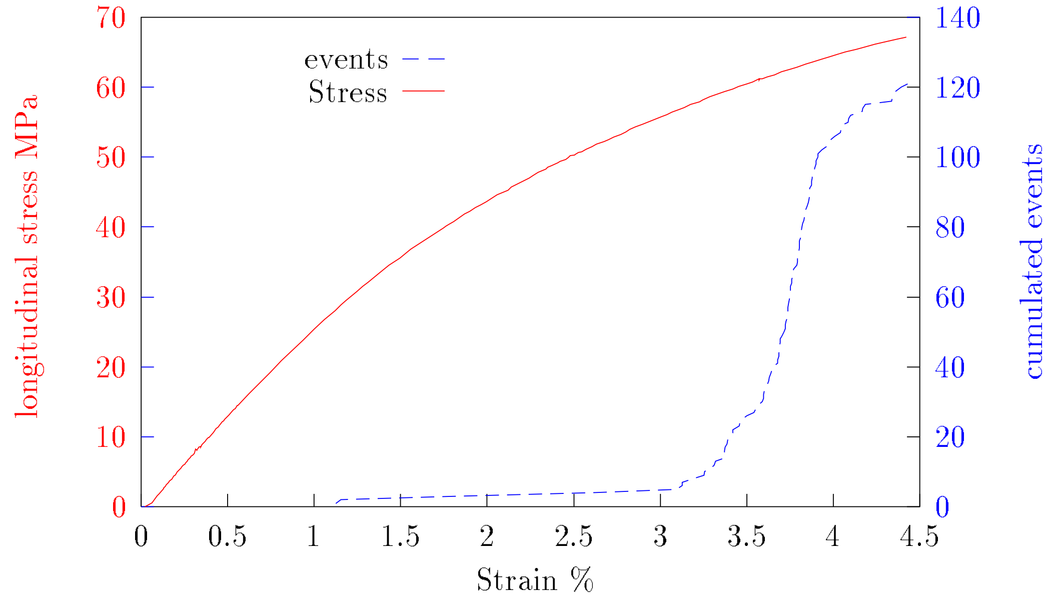

Figure 2: *Experimental results: strain-stress in the coupon and recording of the breaks by acoustic emission*

The breaks are recorded by using acoustic emission with two captors fixed at the ends of the gauge length. Several authors have shown [1, 3] a good correlation between acoustic emission and the number of fiber breaks for multifragmentation tests. This technic allows to locate the break in the fiber to verify that the event has occured in the gauge length. The coupons have a bone shape, the fiber material is an E-glass, and the resin is composed with a 72% of LY 5052 araldite and 28% (mass percent) of HY 5052 catalyst. This resin has been choosen for its good break strain, between 4 and 8%. Ten tests have been realised and 1499 breaks recorded. Finally the test provides the number of fibre breaks n_r in function of the stress σ.

INTERPRETATION OF THE TEST

The test results are interprated to give the law of probability of the strength of the fibre. Related to the theory of the weakest link and fragile break, the probability law is searched has a function $P(\sigma)$ which can be writen:

$$P(\sigma) = 1 - exp(-L\theta(\sigma)) \tag{1}$$

where L is the gauge length and $\theta(\sigma)$ the density of the cumulated defects which the strength are lower than σ. According to the expression of θ, P may be a Weibull, Duxbury or sigmoid law. But it is impossible to interprate the results of the multifragmentation tests as a classical traction test with a constant gauge-length. Due to stress transfer around the fiber breaks, some parts are not loaded, then L varies and $\theta(\sigma)$ can't be evaluated as $n_r(\sigma)/L$. This leads to evaluate the real gauge length L_e and then the length substracted to L after each break.

This length is interprated as a "transfer length". At the break, the fibre is not loaded, but due to shear stress between fibre and matrix, the fibre is stressed far from the break. The transfer length is defined as the minimal length which can be charged at the maximal stress (This definition is similar to the ineffective length in the probabilistic model of composite break). The phenomenum of charge transfer takes place on bit length equal to $\delta/2$. Every bit longer than δ is led to break. Then $\delta/2$ corresponds to the minimal distance between 2 breaks.

To modelise the multifragmentation test, an estimation of δ is necessary. The experimental measurement of the transfer length is difficult but there are micromechanical models of charge transfer for a fibre embedded in a matrix. They are all based on the shear-lag hypothesis. A stress shape corresponds to all hypothesis of interfacial behaviour. The interaction between 2 bits is not taken into account. In literature, there are 3 majors modelisations which are associated with different matrix behaviour:

- perfect adhesion with elastic behaviour
- debounding associated with perfect elasto-plastic model
- total debounding with a perfect plastic behaviour

These modelisations and behaviours lead to micro-mechanical models. Many of them are described in [12]. The main difficulty to apply these models, is the identication of physical parameters. So, until creation of an experimental process, the choice of the best model is based on qualitative hypothesis. Each model gives its own estimation of transfer length which can't be considered as a material constant.

Supposing a perfect elastic behaviour and adhesion at the interface, Rosen [16] finds:

$$\frac{\delta}{2r_f} = \frac{1}{2}\left[2\ln\left(\frac{r_l}{r_f}\right)\frac{E_f}{G_m}\right]^{1/2}\ln\left(\frac{1}{1-\alpha}\right) \tag{2}$$

where r_f (11 μm) is the fibre radius, E_f (69000 MPa) the fibre Young's Modulus, G_m (1054 Mpa) the matrix shear modulus, r_l (10 r_f) the limit radius beyond which the matrix is considered to be out of influence of the break. Because the reload shape after break has an exponential shape, the fibre is mathematically never relaoded, then it is necessary to use a ratio α (0.9) of stress for which the fibre is considered to be reloaded.

In this model, δ is independant of applied stress. In the other hand, stress increases in the transfer zone. So in contradiction with its definition, a fibre break may occur in the transfer length. Moreover, experimentaly debounding often appears in many multifragmentation tests. The model is unrealistic in many systems. Nevertheless, spectroscopic studies have shown a good fitness with stress shape.

There is also, a statistical evaluation of δ [7, 13, 10]. Making hypothesis the bit length l_b follows a symetric law of distribution included in $]\delta/2, \delta[$, then δ is calculated simply by considering the mean $E[l_b]$ of bit length:

$$\delta = \frac{1}{2}E[l_b]_{\text{at saturation}}(1+\frac{1}{2}) = \frac{3}{4}E[l_b]_{\text{at saturation}} \tag{3}$$

It is still now the most usefull method. This problem of behaviour of matrix and stress shape along the fibre is an interesting and important problem to treat test results. The mean length of bit is 333 μm leading to δ equal to 437 μm. The value found with the elastic model (Equation 2) for the tested coupon is 440 μm. Then the hypothesis are considered to be validated.

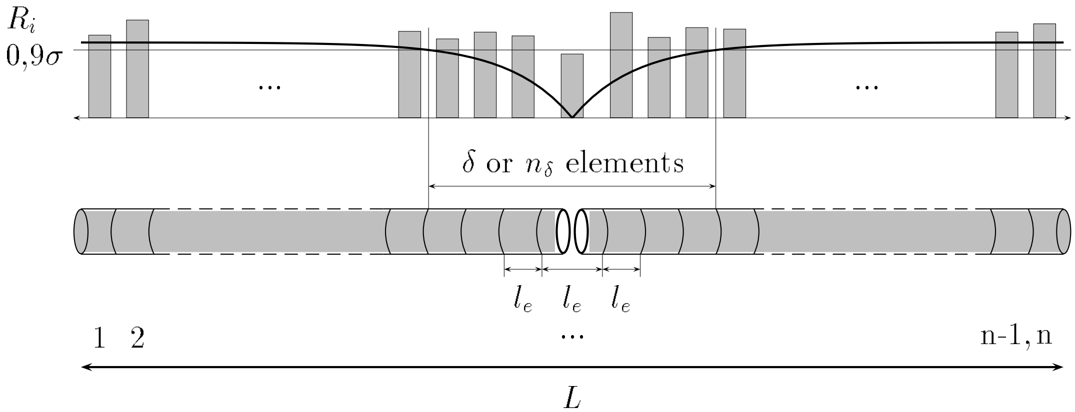

Figure 3: *Modelisation of the multifragmentation test*

Then a first evaluation of θ can be made by a linear approximation [3] where n_t is the total number of recorded breaks:

$$\theta(\sigma) = \frac{n_r(\sigma)}{L - n_r(\sigma)\delta} = \frac{n_r}{L - \frac{4}{3}E[l_b]} = \frac{n_r}{\left(1 - \frac{4n_r}{3(n_t+1)}\right) L} \tag{4}$$

MODELISATION OF THE TEST

To verify the preciseness of this evaluation, a modelisation of the test has been made. The fibre is modelised as a serie of n elements of length l_e. According to equation 1, the probability of strength of these elements is equal to:

$$P_e(\sigma) = 1 - \exp(-l_e f(\sigma)) \tag{5}$$

where $L = nl_e$. The strength R_i of each element is randomly generated. The weakest element is considered to be the first broken. The n_δ elements around the broken one are considered to be unloaded ($n_{delta} l_e = \delta$). They represent the transfer length. Then the second break occurs on the weakest element on the remaining elements. The n_δ elements around it are also considered unloaded, and so on until all elements are considered unloaded or broken. Thereby, an evaluation of n_t and $n_r(\sigma)$ is obtained and then θ may be estimated. The figure 4 shows the approximation of the input data $f(\sigma)$ with the linear evaluation (indexed by ^{l}) of L_e given by equation 4 for unimodal and bimodal law. The linear approximation is not precise enough to interprate the test result. Moreover, it is impossible to know with this approximation if the original function f is an unimodal or bimodal one because the approximated function θ_u^l is clearly bimodal.
The modelisation of the test allows also to evaluate the real value of L_e which is clearly non-linear. Instead of substracting $n_\delta l_e$ after each break, it is possible to test if some of the n_δ neighbouring elements are yet broken and unlaoded and to substract only the real unlaoded length. But even with this modelisation, the curves θ_u and θ_b are unable to provide the input curve f.

Study of the data during the test

To solve the problem of the interpretation of the test data, the test simulation is analysed in term of probability in a Weibull space. The figure 5 shows the various transformation from the input fonction $f(\sigma)$ to its approximation ($-ln(1 - P_{n_r})/r_2$ in figure 5). The

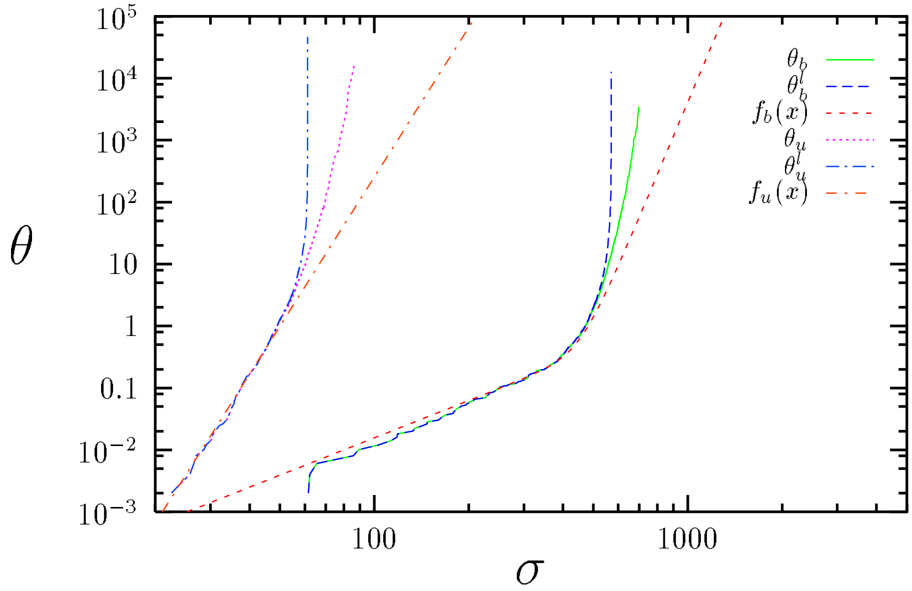

Figure 4: *Simulation of defect densities: an unimodal Weibull law f_u and a bimodal one f_b and their approximations: calculated with a linear approximation of L_e (θ^l) and a non-linear one (θ)*

first step is to generate the n strength of the elements from the input data $f(\sigma)$, defect density of a fiber with a length equal to unity. Because they have a length l_e, their strength distribution is given by $P_e(\sigma)$ (equation 5); that gives the transformation T_1 which is a translation of $\log(l_e)$.
The next step is the selection of the critical defects. This selection gives practically the distribution law of the defects which is given by the simple satistical evaluation:

$$P_{n_r}(\sigma) = \frac{n_r(\sigma)}{n_t + 1} \tag{6}$$

This function can be extracted from experimental result. $l_e.f(\sigma)$ may be also expressed as the result of a test where n_δ is equal to 0. Then it can be considered as the curve in the Weibull's space of $P_R(\sigma) = \frac{R(\sigma)}{n+1}$; by developing the expression of P_{n_r} and P_R around 0, it leads that the transformation T_2 is $\log(n) - \log(n_t) \approx \log(n_\delta - 1)$. This is really the numerical translation of the sorting of defects which is applied during the test. Because l_e and n_δ are completly determined by the physical value δ, the transformations are independant from the modelisation and overall from $f(\sigma)$, then this transformation may be applied whatever the expression of $f(\sigma)$ is. On the other hand, at the end of the transformation $-ln(1 - P_{n_r})$ is no more linear and the transformation T_2 is no more available. Note that the same phenomenum appears on the experimental evaluation of $-ln(1 - P_{n_r})$ (fig. 7); The function is obviously bimodal until a high stress value where the curve rate decreases. So the numerical simulation represents well the experimental problem. So at the end of the test the function P_{n_r} is underestimated. n_r is represents no more the number of defects breaking under the stress σ in the length L_e. The less critical defects are hidden by the breaks of the most criticical.
The problem is to find a transformation T_3 independent from the input value and the modelisation value allowing to fit precisely the law $f(\sigma)$. The figure 5 proves that this transformation exists. It is simply given by:

$$T_3 = \frac{-\ln(1 - P_{n_r}(\sigma))}{f(\sigma)} \tag{7}$$

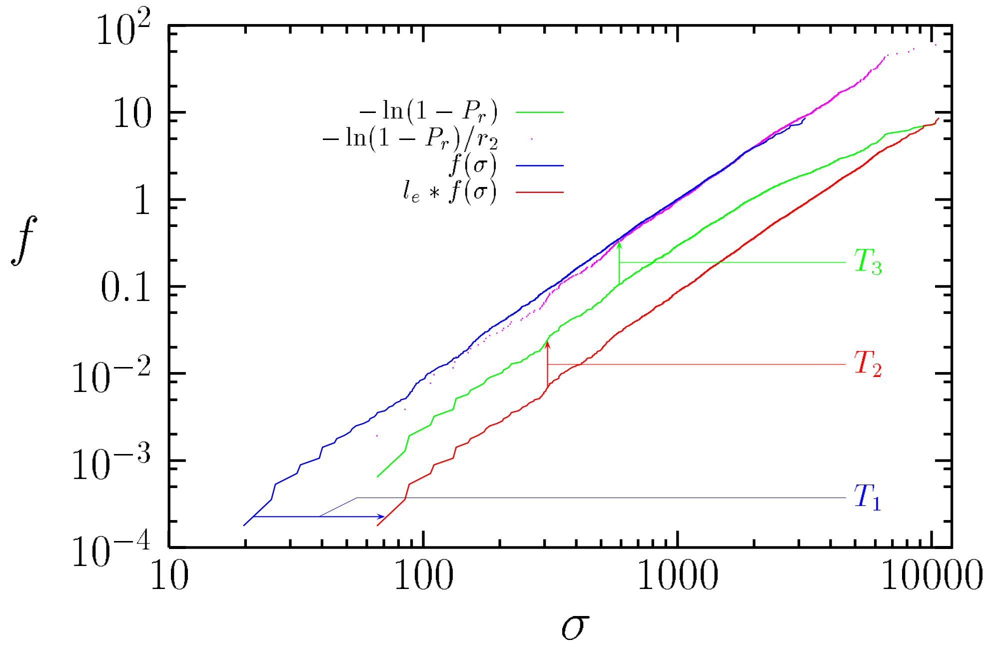

Figure 5: *Various transformations of the data during the test simulation*

If T_3 calculated by simulation is expressed in function of $n_{\%} = n_r/n_t$, T_3 is independant from the type and the parameters of laws. Three laws types have been tested (Duxbury, sigmoid unimodal and bimodal, Weibull unimodal and bimodal) and for various parameter. Finally 12 sets (type-parameter) have been tested as $f(\sigma)$. For each example T_3 has been evaluated. Because it is a random process, T_3 was calculated as the mean of 100 simulations. For each example, the determination coefficient between 2 values of T_3 is greater than 0.999. Then T_3 is considered to be independant from the type and the parameters of law as the previous transformations.

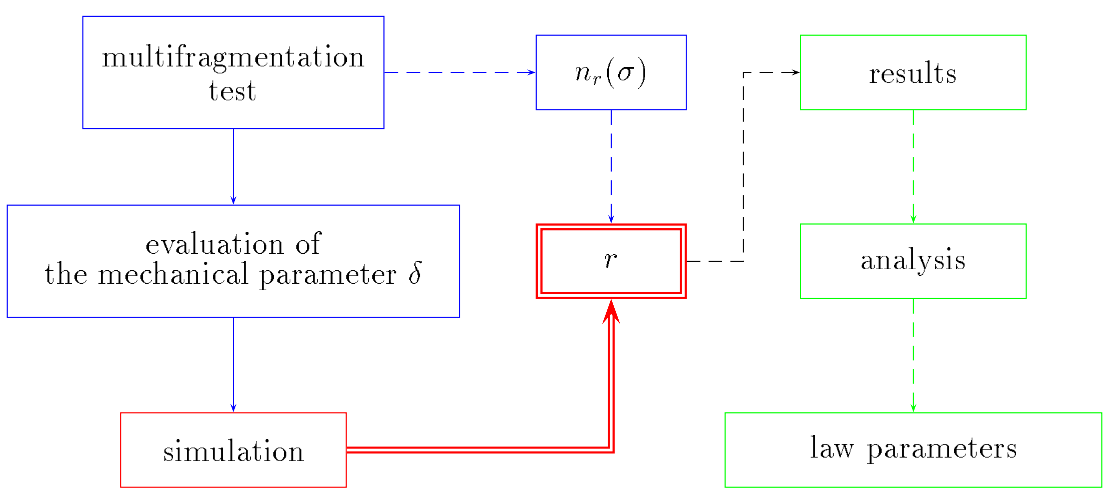

Figure 6: *Process of interpretation of results*

Test of the transformation T_3

After having evaluated T_3, its usability has been tested. From simulated results, the input $f(\sigma)$ has been approximated by $T_3 * P_{n_r}$. For unimodal f, the errors on paramaters are

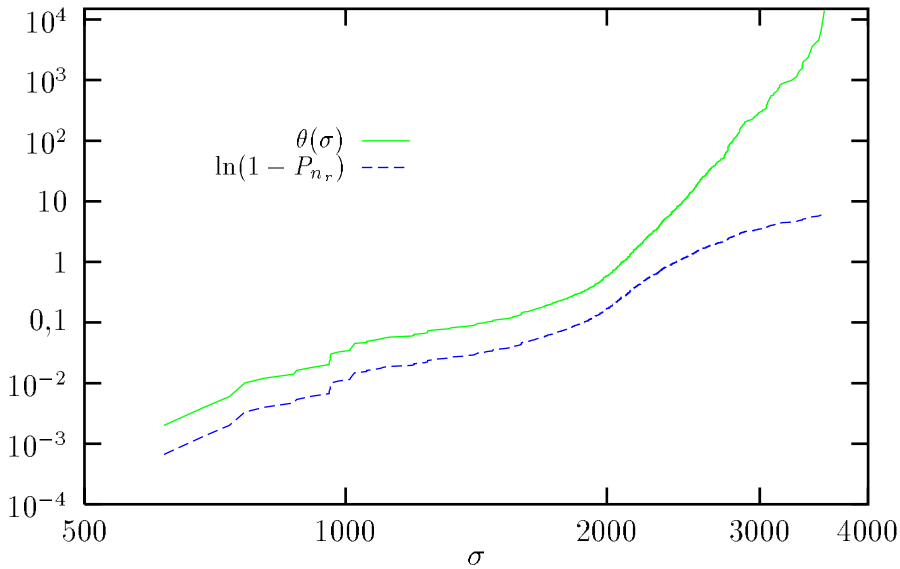

Figure 7: *Experimental data $-ln(1-P_{n_r})$ and its transformation θ*

less than 1.5% instead of 25% with the use of the linear part of θ^l_u. For bimodal f, the interpretation is more difficult (there is less points to find 2 others parameters) but the maximal error is near 10% to compare with 200% with the old method.
Then to interpret multifragmentation result, the method is to evaluate δ, thereby to generate T_3 by simulation, thereafter to apply T_3 to the experimental value of P_{n_r} and to interpret this curve as any statistical law (cf. fig. 6).

type of law	σ_1 (MPa)	ρ_1	σ_2 (MPa)	ρ_2	σ_3 (MPa)	ρ_3	R^2
Weib.bi.[1]	2487	3,49	2014	7,66	-	-	0,979
Weib.bi.[2]	3675	2,48	2102	9,21	-	-	0,989
Weib.tri.	3600	2,48	2050	9.21	2225	7	0.9995

Table 1: *Parameters of the law fitting the experimental result (cf. fig. 8)*

RESULT OF AN INTERPRETATION OF AN EXPERIMENTAL TEST

The figure 8 shows the experimental density of defect interpolated by various Weibull laws. Because of the shape of the experimental f, a law at least bimodal must obviously be used to fit the curve. Then there is two populations of defects in the fibre: unusual and scattred critical defects (low second parameter of the Weibull law Tab. 1) and common and less scattered defects. Due to the experimental dispersion of result, it is possible to evaluate two bimodal weibull laws from the test results. The first one "Weib. bi.[1]" fits the extreme parts of the curve to interpolate the most linear part of the curve. The second omitting the most scattered defects, provides a less scattered law. Nevertheless both approximations are unable to fit precisely the experimental results. The real correlation between the two populations is less than the one evaluated by the bimodal law. The experimental results are more curved. Then we introduce a mathematical mode in the bimodal Weibull law to improve the accuracy of the approximation. The function $f(\sigma)$ is searched as a trimodal function:

$$f(\sigma) = \left(\frac{\sigma}{\sigma_1}\right)^{\rho_1} + \left(\frac{\sigma}{\sigma_2}\right)^{\rho_2} - \left(\frac{\sigma}{\sigma_3}\right)^{\rho_3}$$

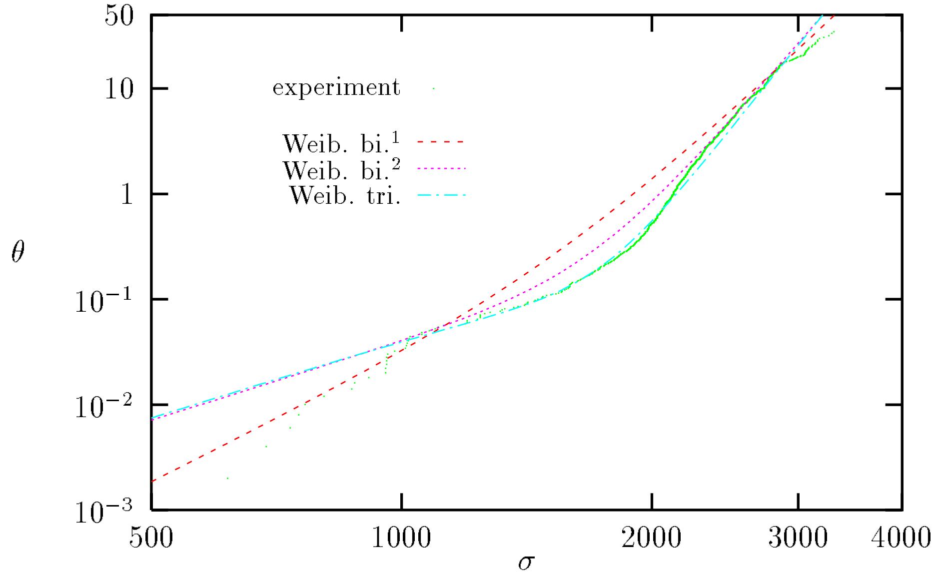

Figure 8: *Experimental density of defect interpolated by various Weibull laws*

where the parameters σ_i and ρ_i must verify $\sigma_1 > \sigma_3 > \sigma_2$ and $\rho_1 < \rho_3 < \rho_2$. The new mode is only a mathematic trick. The difficulty is the determination of the parameters but the figure 8 shows the interest of its use. Moreover the use of a bi- or trimodal law infuences highly the results of the probabilistic model of composite strength [6].

CONCLUSION

This article has presented a method to interpret the data issued from a multifragmentation test. Defects in function of the applied stress are recorded by acoustic emission. Thereby a approximation of the density of defects is searched, leading to the need of the evalution of the gauge length. Because the gauge length is not constant during the test, the estimation of the density is not obvious. A numerical simulation of the test has demonstrated that a linear approximation of the real gauge length in function of the breaks number is unable to provide interpretable results. Moreover it is impossible to know the real shape of the probabilistic law. A non-linear approximation of the gauge length has been made with a numerical simulation but even if the approximation of defect density is better, the error remains too important to estimate the real parameters of the probabilistic law.
An analysis of the various transformations applied to the defect density has shown that not only the gauge length is not constant during the test but the defect population under the scope of the test is not constant. The broken defects hide a part of the population of the mean defects. The experimental result shows also this tendancy. By the simulation of the test, a transformation is calculated to correct the evalution of the population. Many tests where the law type and its parameter vary, have proven that the transformation is a function of the ratio of broken defects. Then because the transformation is independant from the input parameters of the test, it is applied to the experimental result. This methodology may be applied to all the multifragmentation tests. It is only necessary to generate the transformation by using the numerical model of test. Finally an experimental result is interpreted and the parameters of a trimodal Weibull law are given.

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