On the mechanical properties and damage mechanisms of short fibers reinforced composite submitted to hydrothermal aging: Application to sheet molding compound composite

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A B S T R A C T

Sheet Molding compound (SMC) composites were subjected to water immersion tests in order to study their durability since such composites are of interest in automotive applications. Water sorption tests were conducted by immersing specimens in distilled water at 25-90°C for different time durations. In order to investigate the combined action of water and temperature over time on composite mechanical behavior, tensile tests and quasi-static loading were conducted. The mechanical properties of water immersed specimens were evaluated and compared alongside to dry composite behaviour. The tensile tests and quasi-static properties of the studied composite were found to decrease with the increase in moisture uptake. This decrease was attributed toinner structure dégradations by means of osmosis phenomenon. It was shown that hydrothermal aging affects mainly the fiber/matrix interfacial zone while a good adhesion between the reinforcement and the matrix was observed for the virgin samples. In order to well understand the damage mechanisms, scanning electron microspy (in-situ three point bending) tests were performed on aged and non aged specimens. Damage mechanisms were identified for different material states. Results display clearly that damage evolution always begins at the interface regions. Furthermore, a quantitave analysis was performed at a local scale in a representative zone of the tensile area.

1. Introduction

In automotive manufacturing industries, design modifications of vehicles have major impact on customer demand. Compared to the preexisting method, this will be more challenging when appropriate materials and methods are used. Vehicle manufacturers introduced various technologies in terms of the structure in order to reduce the weight, directly achieving low CO2 emissions [1]. One of the convincing methods of emission reduction is the application of lightweight structures. Lightweight materials are increasingly used in automobile and airplane industries to realize energy conservation and emission reduction. In particular, ploymer composites as

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the most promising lightweight materials are widely applied, such as Airbus A380, Boeing 787, Peugeot PSA 3008, Mercedes-Benz McLaren SLR, because of the advantages of light weight and high specific strength and impact resistance [2–4]. The glass fiber reinforced composite matrix possess three parts including reinforcing glass fibers (GFs), hosting matrix and interphase between GFs and matrix. This interface between the fiber and the matrix makes a critical contribution to the performance of the composites and a good quality interface can result in good mechanical properties [3,5,6].

However, the current trend in the vehicle design is influenced by awareness of environmental issues. During composite use, especially in automotive applications, different component are subjected to environmental conditions. The study of the exposure of such materials to humidity and temperature over time is of outmost importance, in order to assess the impact of these aging factors on their mechanical behavior.

As far as water is concerned, absorption through polymer composites usually follows Fickian or non Fickian behavior [7–9] and its effect is detrimental for certain material properties. For example, absorbed water has been found to disrupt the interfacial bonds, to occupy the voids of the composite (or polymer) [9,10] and, thus, cause changes in the overall free matrix volume. The latter can lead to accumulation of hydroscopic stresses that can crack and fail the resin. Also water sorption may cause physical ageing : Changes in the resin can be either reversible (plasticization and swelling) or irreversible (dissolution, crosslinking and microcracking). These phenomenons depend on the chemical structure of the resin, the initial adhesion between the componenets, the aging temperature and the total period of exposure to water [11]. Otherwise, The water diffusion into polymer and composite structure may causes leaching of some components [9], which can lead to an irreversible damage of these materials.

Of course these changes affect the mechanical performance; the larger the water content, the greater the osmosis dégradations [10,12,13].

The mechanical behavior of fiber reinforced polymer composites can be strongly influenced by the changes mentionned above. In fact, these variations induces local stressess in the inner structure of the material leading to the formation of micro cracks [14].

Damage and failure mechanisms associated to composite materials are various and complex. Fiber/matrix interface weakening was found to be the most frequent-observed damage mechanism leading to final failure through micro-cracks coalescence [3,15,16]. Moreover, several authors reported that damage starts at the fiber ends and propagates along the interface before coalescence through matrix and failure [5,16].

Other studies dealing with humid aging have shown that the generation of micro-cracks in the matrix creates paths for water molecules to penetrate in the fiber–matrix interface by capillarity. Thus the interface is deteriorated and the material lifetime is consequently reduced [4,17–19].

Many authors have focused their studies on composite damage characterization in order to well understand the damage mechanisms [20–22]. However, few studies were realized to discuss the local damage mechanisms of GFs-reinforced SMC composite under hydrothermal aging.

In this paper, SMC composites were subjected to hydrothermal aging at different temperature. The first intent of this work is to study the SMC behavior under accelerated environmental conditions (immersion in water at various temperature) from a mechanical point of view. The second pupose of this study is obtain an accurate information about crack initiation and propagation in the inner structure of SMC composite by means of microscopic inspections during loading. The used technique to characterize the nucleation and the progress of fracture is the in-situ microscopic observations during testing.

2. Material and methods

The material under study is an E – galss fiber reinforced unsaturated polyester composite provided by Faurecia company. Structurally, this material consists of chopped fibers (28% in mass) embedded in polyester resin filled with $CaCO_3$ particles (37% in mass) . The glass fibers are presented in the form of coated resin bundles with a contant length of 25 mm and a diameter of 15 μm. The SMC plates were manufactered using a thermocompression process under a temperature of approximatevely 165 °C and a pressure of 60 bar. The primary caracteristics have been studied and presented in [23].

In this study, SMC samples were immersed in distilled water at different temperatures 25 °C, 50 °C, 70 °C, and 90 °C. The elevated

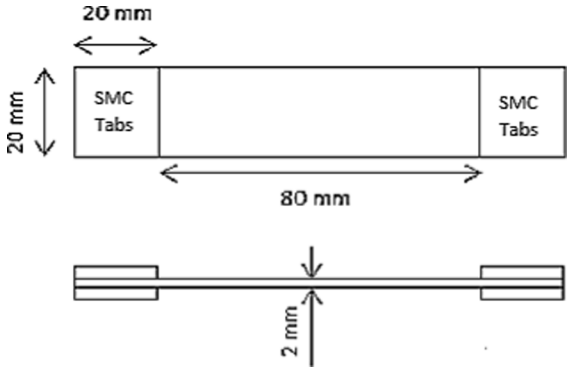

Fig. 1. SMC specimen geometry for mechanical tests.

temperatures are often used as a way to accelerate the testing and so to shorten the conditionning time

To study sorption behavior, specimen mass changes were recorded at regular time intervals using an electronic balance (METTLER AT261 with a precision of $\pm 0.05\text{mg}$). Then, two types of mechanical testing were carried out:

-Tensile tests were performed with 2 mm/min load velocity.

-Quasi-static loading–unloading tensile tests with increasing maximum stress. The analysis of the reloading slope leads to the determination of the corresponding loss of stiffness

These procedures were applied on overall time and temperature (t, T) conditions. At each specific condition (t, T), five specimens were tested.

The overall mechanical tests were conducted on MTS 830 hydraulic fatigue machine using samples as described in Fig. 1. SMC composite end tabs were bonded to all testing specimens in order to homogenize the stress distribution in the working zone.

In order to study the damage mechanisms in SMC composites, in-situ tests were carried out by positionning the specimen in the SEM chamber (HITECHI 4800) and by subjecting them to flexural loading using three-point bending device (DEBEN) with a span length of 27 mm (Fig. 2-(a)). The real time display of force-extension curve is controlled using the DEBBEN acquisition software. The contact center crosshead speed was set up to 0.01 mm/min.

In order to be able to acquire images during the test, the displacement of the crosshead was interrupted in fixed positions while applying a constant load. The observation period was limited to 2–3 min to reduce the relaxation effect of the composite. the SEM observation were carried out in the shell layer subjected to tension (Fig. 2– (b)) of the specimen at a distance of 200–250 μm from the surface.

In order to obtain statistically significant results, an area of 125 × 80 μm was chosen as a representative zone.

3. Results

Water sorption behaviour was closely disscussed and analysed in the previous work [9]. Fig. 3 shows the water sorption behaviour at different aging temperatures over time. Each data point in the figure represents the average value of 3–5 specimens. It is clear that higher temperatures accelerate the moisture uptake behaviour. When the temperature of immersion is increased, the moisture saturation time (MST) is greatly shortened. For samples aged at 90 °C, it takes 6 months ($\approx 2000\text{s}^{0.5}/\text{mm}$) to reach MST whereas for samples aged at room temperature, the MST is not yet achieved. Furthermore, it was found that sorption behaviour follow langmuir diffusion-type based on Carter and kibler's theory [9]. The water sorption process for all aged conditions is linear in the beginning, then slows for a period of aging time. This phenomenon was associated to $CaCO_3$ leaching due to osmosis process. Finally, the diffusion restarts again in the material until the saturation as shown in the result of aged 90 °C specimen.

In the following, the results can be devided into two parts. The first part consider the effect of water sorption on the mechanical properties, and the second evaluates the damage mechanisms through microscopic observation to better undersatnd the causes of mechanical behaviour drop.

3.1. Mechanical behaviour

3.1.1. Tesile tests

Fig. 4 shows the tensile test results of specimens aged at 50 °C at different time. Similar trends were obtained for samples aged at 25 °C, 70 °C and 90 °C although not shown here for brevity.

According to these results, Three different regions can be distinguished:

A first linear and reversible part corresponding to the elastic behavior of the composite material.

The second part is non-linear. It is associated to microscopic damage initiation. During this phase, the initiation of microcracks at the fiber / matrix interface is considered as the predominent damage as discussed in a previous study [25].

Finally, the third part is approximatively linear until breaking completely. Microscopic observations show that this phase is related to the multiplication of micro-fractures. This degradation causes the ruin of the material due to the propagation and the coalescence of macro-cracks.

In this context, and based on the three phases described above, different authors have highlighted these behaviours in their studies [2,15,26]. They showed a chronological order of damage-mechanisms appearance: the rupture of the interface, the matrix cracking and then the rupture of the fibers. These findings highlight the effect of cracking on the degradation of the overall mechanical behavior

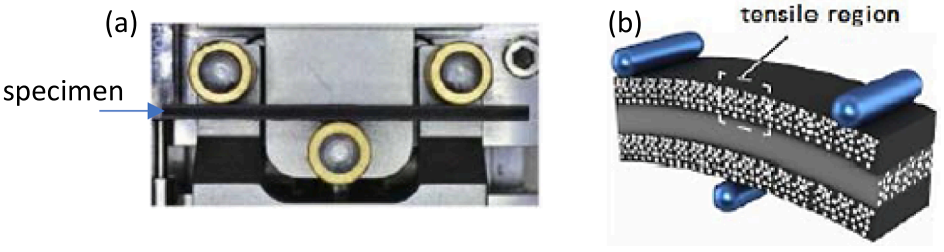

Fig. 2. SEM bending test device: (a) image from experiments (b) illustrated image [24].

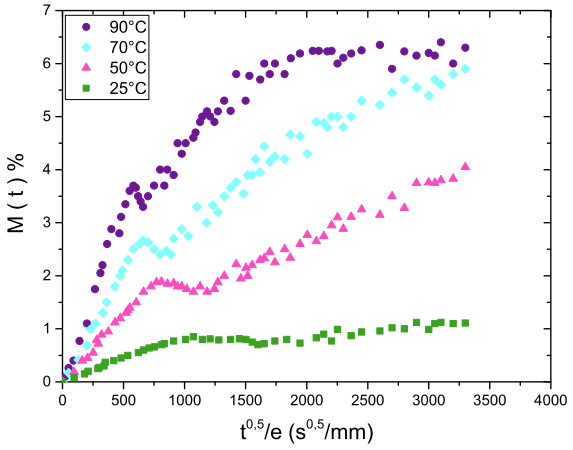

Fig. 3. Gravimetric results for water absorption at 25 °C, 50 °C, 70 °C and 90 °C.

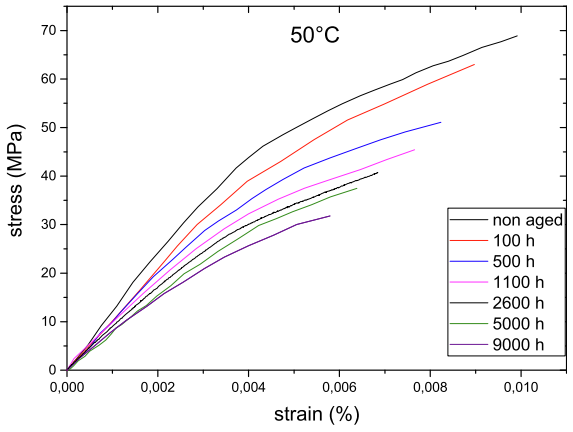

Fig. 4. Stress–strain curve of specimens aged at 50 °C after different aging time.

of the SMC composite.

The results of tensile stress at threshold (σ_s), young modulus (E), and stress and strain at break (σ_r, ε_r respectively) versus time for aged samples are shown in Fig. 5.

Overall the tensile properties have the same trends whatever the aging temperature. Two different behaviors can be clearly distinguished :

• At early periods of aging (t < 2000 h), a visible decrease in properties was observed at the different temperatures. The elastic modulus (E) and the stress at break (σ_r) decrease by approximately 77% ±2%and 60% ±2%for samples aged respectively at 90° C and 70° C, while their variations at 50 °C and 25 °C are respectively 37% ±2%and 15% ±2%. This loss in mechanical properties may be linked to material plasticization caused by water molecules diffusion. Or the plasticization of the polymer network should result in an increase in the strain at break (ε_r). However, here, it records a noticeable decrease on its values. According to some authors [27], this loss could be related to differential swelling due water concentration gradient between the outer surfaces and the inner ones. This phenomenon results in weakining the specimens and decrease their strain at break.

In this study, such behaviour can be related, not only to plasticization effect, but also to high osmotic pressures generated by water diffusion in the heterogenosities (porosities in the matrix, free volume in the polymer network, etc..) leading, thus, to damage evolution at the matix and the interface regions. The analysis of the microstructure shown in Fig. 8 clearly explains these hypothesis. It seems that the degradation by capillary is much greater compared to the changes occuring at molecular scale. Indeed, the cracks generated by osmosis phenomenon have direct effect on mechanical behaviour, and so the strain decreases over time.

• for periods of time t greater than 2000 h, a stabilization of mechanical properties is observed for all the aging temperature. For specimens aged at 70° C and 90° C , the values at stabilization are lower than samples aged at 25° C and 50° C. This can be explained by the fact that the damage is more important at high temperatures.

3.1.2. Monotonic loads

Loading-unloading tensile tests with progressive increase in the maximum load were used to study the loss of stiffness when

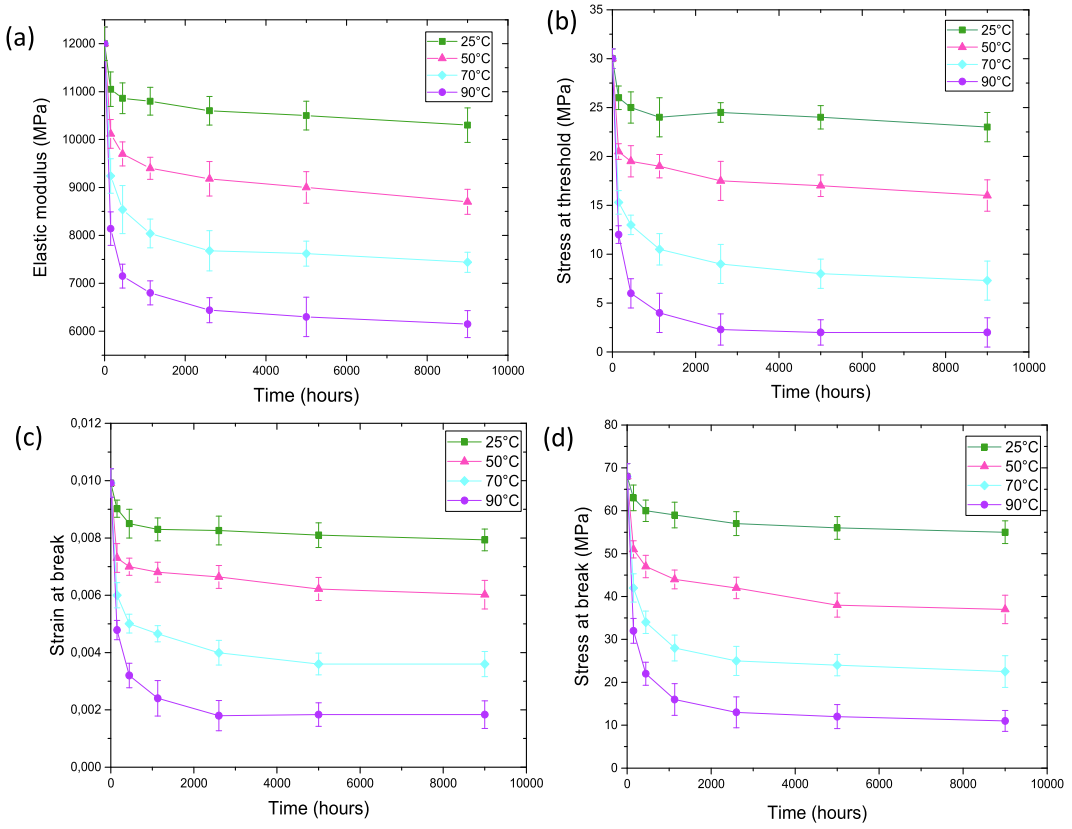

Fig. 5. Evolution of (a) elastic modulus, (b) ultimate stress, (c) stress at break, and (d) strain at break at different aging time and temperature.

applying a progressive stress in order to discuss, macroscopically, the changes in damage kinetics for aged samples. Here we only show the average results of each aging time and temperature (Fig. 6).

In Fig. 6, it is clear that all the specimens have the same trend : a stabilisation in the begining (generally explaied by the fact that the applied load is lower than the stress at threshold), then a decrease in stifness, in a regular way, has been recorded when increasing the loading (as the applied load is higher than stress at threshold).

Here we propose to describe the evolution of the loss of stiffness during monotonic loading by a linear function based on the stress at threshold, σ_s. It is expressed by:

$$E/E_0 = 1 + a\left(\sigma^{imp} - \sigma_s\right)$$

where E_0 and E are the Young modulus of the non-loaded sample and the damaged one, σ^{imp} is the loaded stress, and a is the kinetics of damage.

Based on the results shown in Fig. 6, it is clear that the stress at threshold is sensitive to temperature and time. It decreases by 73%, 56% 33% and 16% respectively at 90 °C, 70 °C, 50 °C and 25 °C after only 500 h in water compared to the unaged state. Similarly, for a given temperature, the stress at threshold decreases over time and, for example, for samples aged at 50 °C a decrease of 20%, 33%, 46% and 55% were recorded after respectively 150, 500, 1000 and 2500 h in water. This is ultimately logic. Indeed, the hydrothermal damage is more and more significant with the increase in temperature as the diffusion kinetic is more and more important.

Otherwise, the loss of stiffness kinetics, denoted “a”, seems to be relatively constant with an average value of 0.00748 whatever the aging conditions (T, t) as shown in Fig. 7.

These behaviours will be discussed in Section 4.

3.2. Damage mechanisms

As mentioned earlier, the embrittlement in the composite occurs at the beginning of exposure to humidity. In order to identify the mechanisms involved in this embrittlement, SEM analysis of the microstructure were carried out on virgin-non loaded sample and aged-non loaded samples taken at different times. Fig. 8 shows the effect of hydrothermal aging at 50 °C on the fiber/matrix interface of SMC composite at different times of exposure. One can easily notice that with the increase of exposure time, small interstices start to appear around the fibers while the virgin sample shows a good adhesion between the reinforcement and the matrix. In fact, after 15%

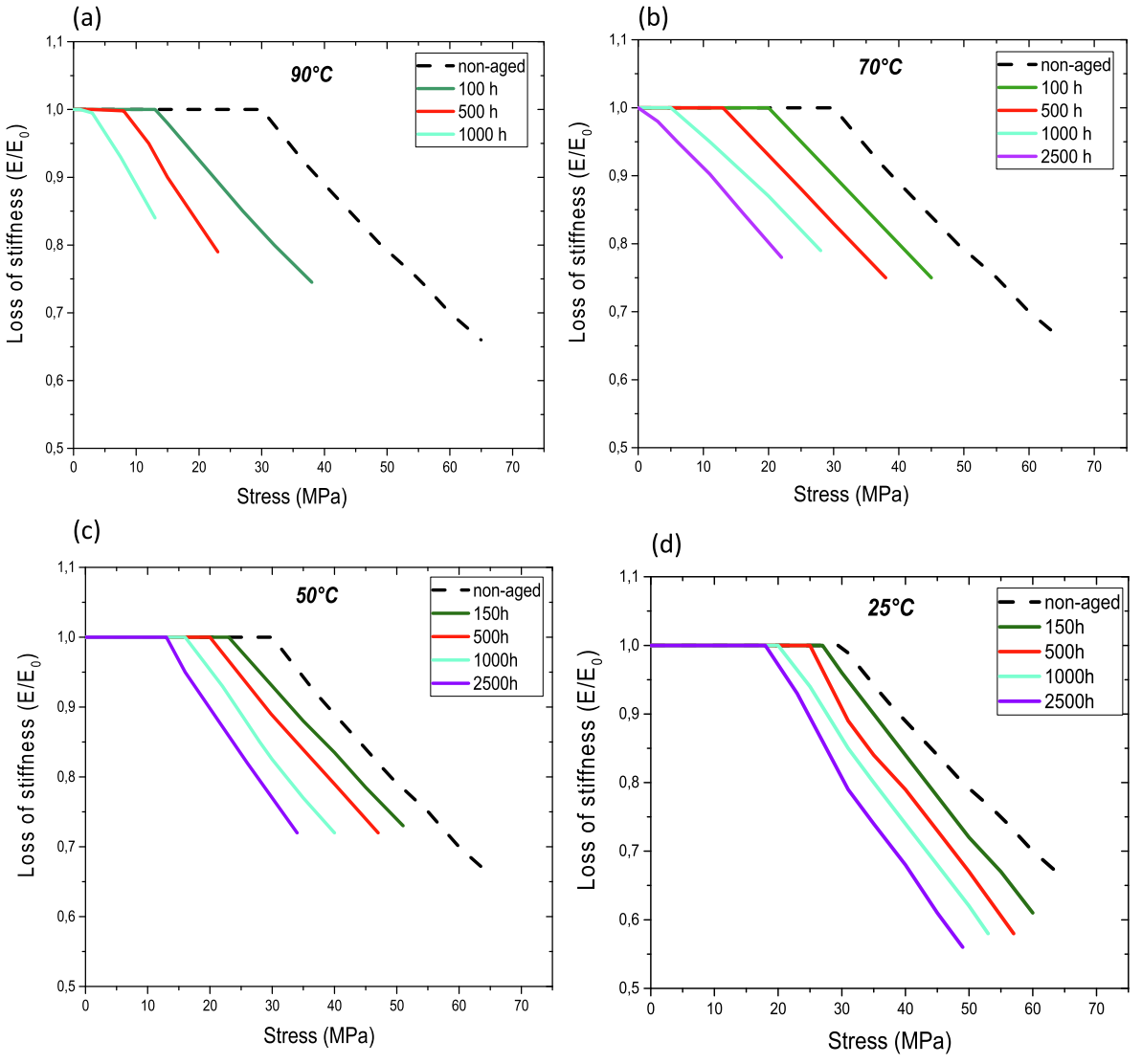

Fig. 6. Loss of stiffness under monotonic loads at different aging time and temperature: a comparaision between time-aged specimens and non-aged ones.

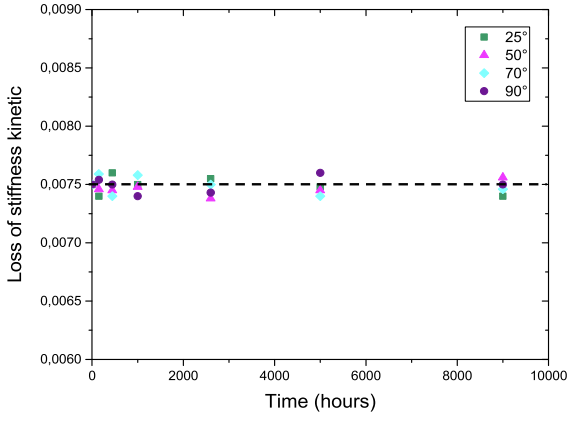

Fig. 7. Loss in stiffness kinetic during the aging time at different temperatures.

of water uptake, interstices (recalling micro-cracks) start to appear covering, thus, 20–30% of the interface perimeter. The micro-cracks spread around the fiber to cover more than 40% of the fiber perimeter after 30% of water uptake and almost the whole fiber after 60% of water uptake.

The appearance of these micro-cracks allows consequently the penetration of the water molecules during thermal water aging and so extends the damaged zones. Therefore, in situ bending tests were performed on hydrothermal-damaged specimens in order to explain the trends observed mechanical behaviour.

Micro-damage evolution

In situ three points bending tests were performed while maintaining the same procedure for all the studied specimens. Here, we only discuss the results obtained on unaged samples and aged at 50 °C after 15% and 30% of water uptake. Fig. 9 represents the force/displacement curve of an aged specimen taken after 30% of water uptake at 50 °C. The same trends were obtained on all the studied samples. The curve shows clearly the interruption of the crosshead displacement needed to take photos of damage initiation and propagation. One can observe that the interruption stages are not enough long to produce significant relaxation of the matrix.

Fig. 10 describes the micro-cracks propagation and evolution in the tensile zone of the tested sample. It shows that the initial form of failure is fiber/matrix debonding which is a very common mechanism for short fiber-reinforced composites due to local stress concentration at the interfaces [10]. Besides, with the increase of water uptake ratio, the interfacial damage increases and, thus, favors the early initiation of cracks which generates an expansion of the damage.

Therefore, final failure occurs faster with increasing water diffusion as shown in Fig. 8. Qualitatively, one can conclude from in situ bending tests that the same phenomenon occurs for all temperatures of aging. Fig. 10- (c, g, k) reveals that once fiber/ matrix debonding appears, failure spread in the form of micro-crack growth linking the closest debonded fibers through matrix cracking. The marked zones in Fig. 10- (c, g, k) indicates that matrix cracking grows beyond the interfaces bridging the gaps between the fiber–matrix interfaces causing a total propagation of the cracks meanwhile other new micro-cracks start to appear developing a new damaged area. Thus, one can conclude that two mechanisms occur simultaneously: fiber–matrix interface degradation and micro-cracks coalescence and opening.

Thereby, one can define two local damage indicators: the first one is related to the evolution of fibers density submitted to interfacial damage (df). It is given by:

$$\mathrm{df} = \frac{\mathrm{damagedfibers}}{\mathrm{totalfibers}} \times 100$$

The second indicator (dS) is related to the opened overall surface of the micro-cracks. The evolutions of damaged fibers and the total damaged surface in the representative tensile zone of the specimens were obtained by image analysis using ImageJ software. The

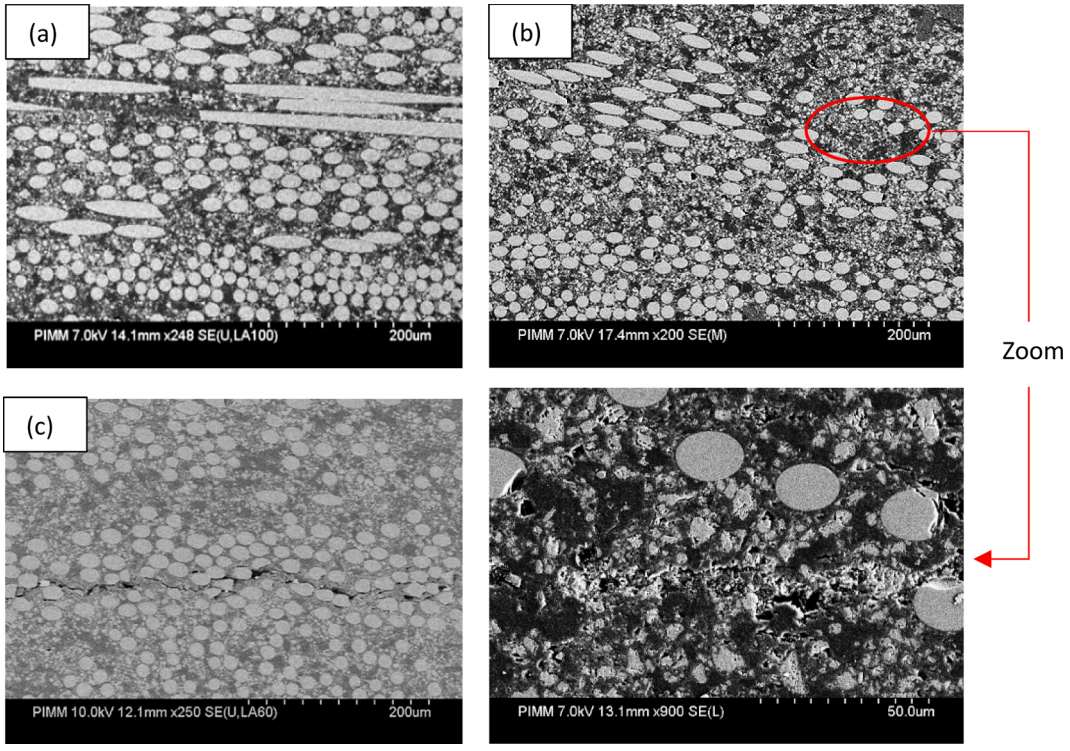

Fig. 8. SEM micrographs of the inner microstructure evolution of the (a) non aged sample, (b), (c) 50 °C aged samples after respectively 15%, 30% of water uptake.

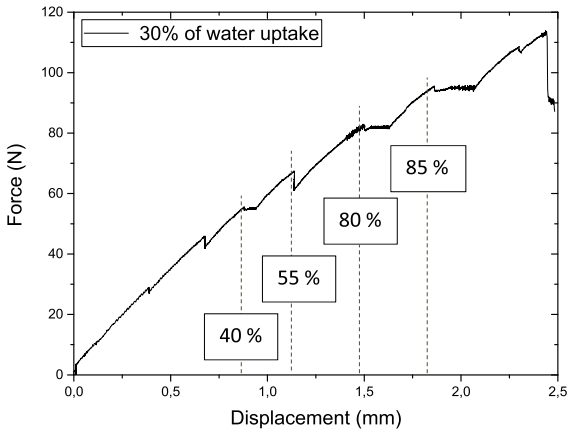

Fig. 9. In-situ three bending curve of SMC composite aged at 50 °C after 30% of water uptake.

results are shown in Fig. 11. The represented values of df and dS are average values for the tested samples.

The microscopic analysis described in Fig. 11 clearly shows:

- A steady progression of interfacial damage under mechanical loading.
- An increase in the embrittlement of interfaces with aging time and mechanical loading, leading to a larger damaged surface.

It can be noted that the number of damaged fibers increases continuously and rises rapidly to reach a damage rate of 70% for only 1.7 mm of displacement in the case of 15% of water uptake. Nevertheless, the virgin sample only reached them towards the end of the test at 3 mm of displacement. It can therefore be concluded that the increase in the exposure time in humid environment leads to a decrease in the damage threshold while following the same kinetics.

4. Discussions

When the composite is exposed to moisture, water molecules diffuses in the material, precisely in the heterogeneities pre-existing in the matrix mainly microporosities and free volumes between molecules which are induced by manufacturing process.

With the increase in the moisture sorption over time, the pressure inside the heterogeneities increases leading, thus, to osmosis phenomenon. Consequently, when the osmotic pressure reachs its critical value micro-cracks appears. As the composite cracks and get damaged, capillarity and transport via micro-cracks become active. The capillarity mechanism involves the flow of water molecules along fiber/matrix interfaces. Therefore, the water molecules attack the interface, resulting in debonding of the fiber and matrix [4,20]. These cracks further contribute to more water penetration into the interface through the induced microcracks. Consequently, this attack leads to fracture size growth which favors their propagation and their coalescence.

If the composite material contains minerals as reinforcing particles phase such as calcium carbonate, the diffusion of moisture in such a material is, then, accelerated due to the hydrophilic property of these particles. In this case, water can also act on the polymer / chalk particles interface, promoting the detachment of chalks from the matrix and subsequently their migrations towards the external surface of the sample as described in our previous study [9].

Matrix cracking, debonding of the fiber/matrix interphase region, and delamination are irreversible processes resulting in a permanent decrease the mechanical properties. At eachvaging temperature, It is interesting to observe that the remarkably decrease in mechanical behaviour is corresponded to the evident increase in moisture uptake indicating a close dependency of mechanical properties on humidity. This could be associated with a premature failure caused by the degradation of the interface and interphase due to hydrothermal ageing and mechanical. In the case of tensile testing, shear stresses can cause debonding of fiber–matrix interfaces of the composite. This is strongly approved under 3-point bending tests

On the tensile area of the cross section of the composite under 3-point bending, stresses generated by mechanical loads affect mainly the interfacial regions, reducing then the effective loaded area, thus, increasing the stresses on these regions. The severe loss in mechanical propertiesdecreases significantly its resistance to mechanical loads, magnifying this detrimental phenomenon. This can be supported by the microscopic observations shown on the fracture surfaces of the aged and non aged samples. Indeed, the micrographs of the observed fracture surfaces show that there is good adhesion between the matrix and the glass fibers for non aged samples (Fig. 12 – (a)). However, the micrographs observed fo 50 °C aged samples after 30% of water uptake show that there is no sticking of the matrix on the fibers (Fig. 12 –(b)) indication, thus, a weak interfacial adhesion due to water attack.

5. Conclusions

Based on obove analysis, The following conclusions may be drawn:

- The effect of water absorption on the mechanical properties of short fiber reinforced SMC composite has been studied following immersion at different temperatures. Mechanical properties decrease at elevated temperatures due to significant moisture induced

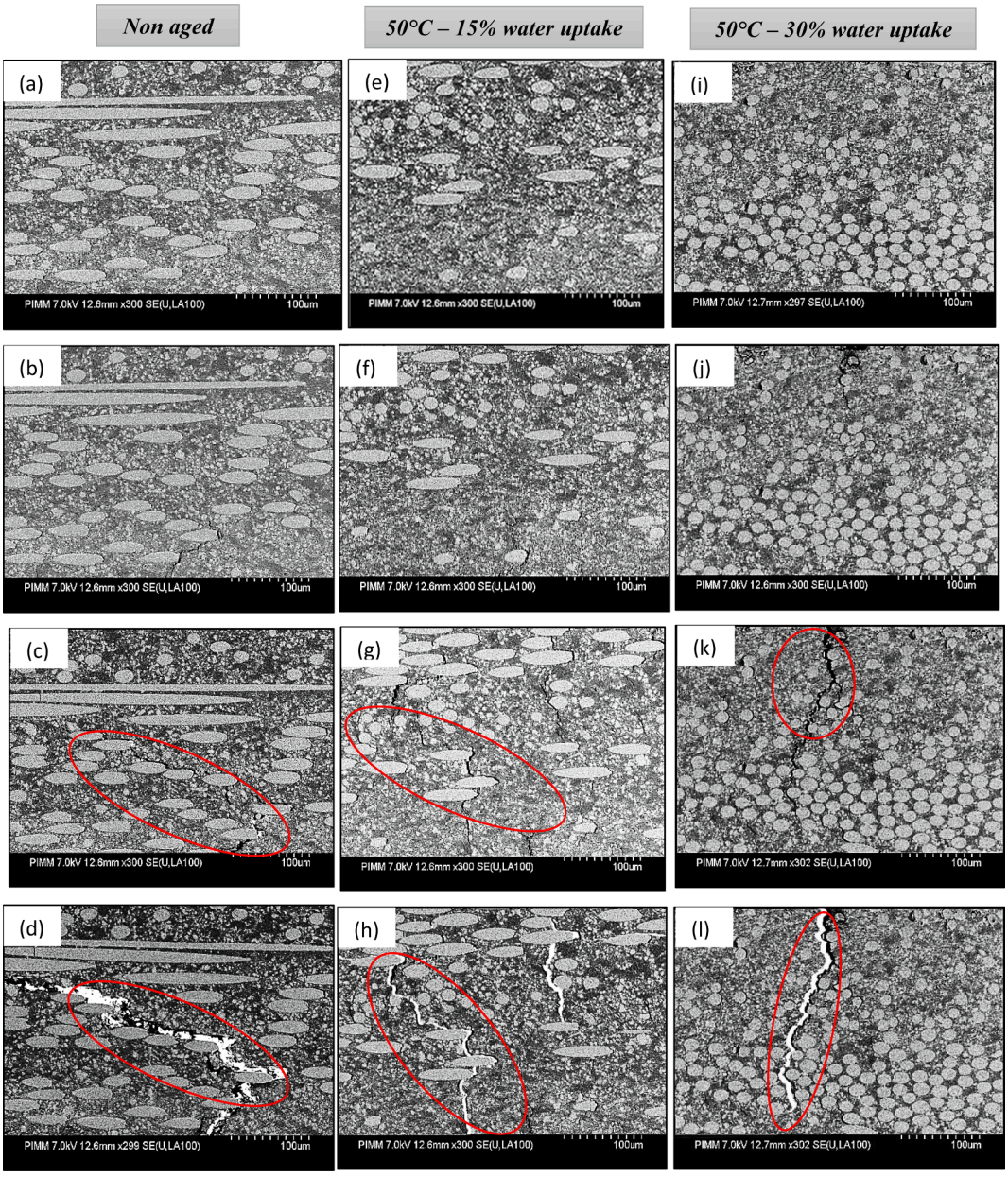

Fig. 10. Cracks evolution of non-aged and an aged specimen after 15% and 30% of water uptake at 50 °C after 40%, 55%, 80% and 85% of the force at break.

degradation. It was shown that moisture uptake results in important drops in tensile properties due to matrix cracking, fiber/matrix debondings and $CaCO_3$/matrix interfacial degradation.

- The predominant damage mechanisms in aged SMC composites are matrix cracking and fiber/ matrix debonding with the increase of exposure time. Furthermore, the fiber/matrix interface weakens during hydrothermal aging and small micro-cracks around the fibers appear promoting the early damage initiation.

- Experimental results on using in situ three bending tests showed that the thermal aging does not affect the damage mechanisms compared to non aged tests. However a decrease in the critical values was recorded with the increase in hydrothermal conditions. Based on the tests observations, the damage chronology lies to fiber matrix debondings at stress yield followed by micro-crack growth linking the closest debonds from other fibers through matrix cracking.

The effect of moisture on SMC composite properties is an important issue and that is why further study is necessary. In this context, a future study is recommended to study and modelize the coupled effects of moisture and temperature on long terme mechanical behaviour under repetetive loads (fatigue tests).

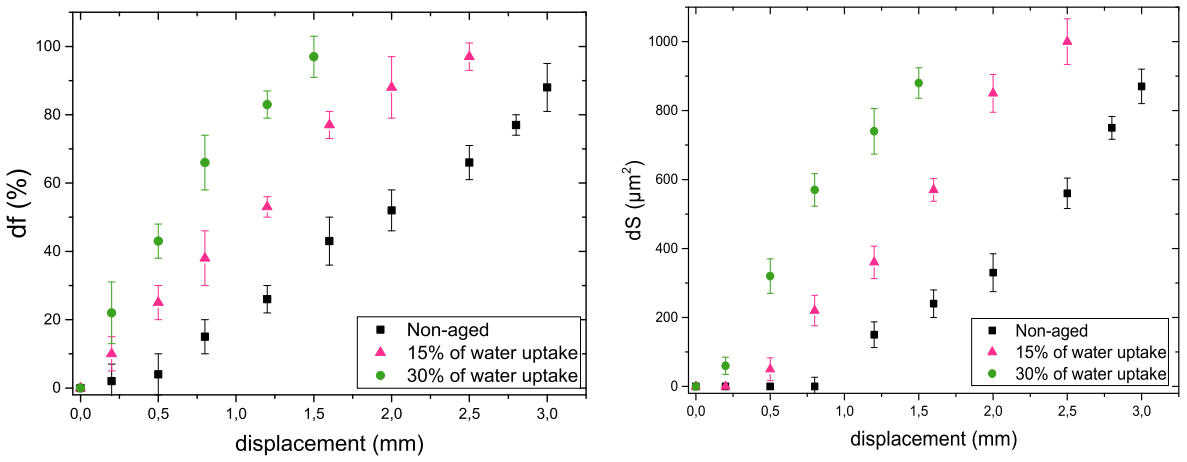

Fig. 11. Damaged fibers and damage surface évolutions of non aged and 50 °C aged samples after 15% and 30% of water uptake.

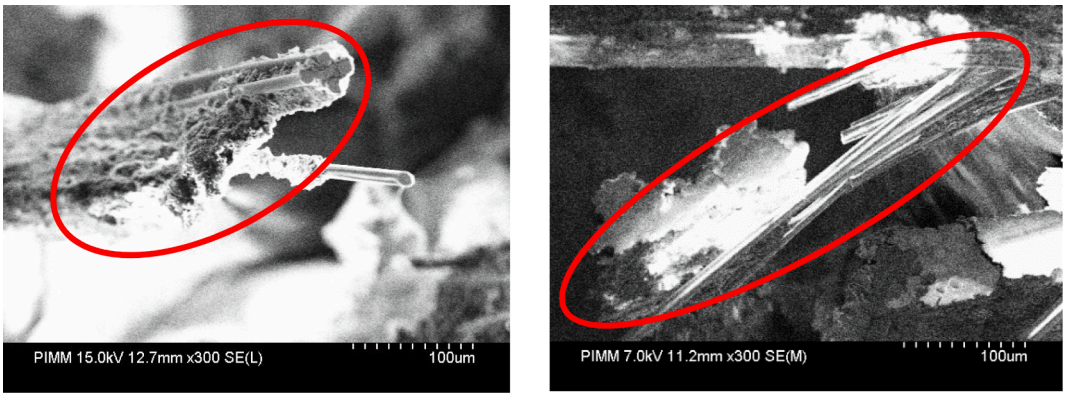

Fig. 12. Micrographs of the fracture surfaces due to mechanical loads (a) before aging, (b) after 30% of water uptake at 50 °C.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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