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Fracture toughness of hydrothermally aged epoxy systems with different crosslink density

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Abstract

The present work investigates the fracture toughness behaviour of Single Edge Notched Bending (SENB) samples of epoxy systems subject to water uptake aging. Two epoxy systems with a significantly different Glass Transition Temperature, T_g, are in particular considered: a typical commercial non-aeronautical grade resin matrix for composite applications, reaching a T_g of 90 °C, and a DGEBA+DDS epoxy system achieving a T_g of 230 °C. The materials have been conditioned by hydrothermal aging in a thermal bath at the temperature of 50 °C. Transmission Photoelastic Stress Analysis is carried out on SENB samples during water aging, monitoring the presence and evolution of swelling stresses. The K_{IC} Fracture toughness is measured and correlated with the internal stress field, with the water diffusion kinetics evaluated by gravimetric tests, and with the data from DMTA analyses. Results have highlighted significant differences in the fracture behaviour of the two epoxy systems.

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Keywords: Fracture Toughness; Hydrothermal Aging; Thermosetting Resin; Swelling Stresses; Photoelastic Stress Analysis, Image Analysis.

1. Introduction

Thermoset polymers used for structural applications such as composite matrices or adhesives are generally subject to water absorption. Exposure to hygroscopic environments is hence a cause for activating aging transformations induced by the water ingress into the material. Many works are now available which have investigated the kinetics of water diffusion, and the concurrent physical and chemical transformations in the material [1-5]. From a structural

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point of view two major aspects of the mechanical behavior are mainly involved in water aging: the modification of fracture toughness due to plasticization and degrading effects in the polymer network structure, and the swelling deformation, which can determine internal swelling stresses when the water distribution is not uniform, or when interfaces are involved between materials with different hygroscopic swelling coefficients [6].

Several works have in particular reported a reduction of the glass transition temperature, T_g, in high density cross linked epoxy systems, as a consequence of water absorption [1,2,6]. Such reduction is in general associated to an increased mobility of the polymer network structure, and hence it is referred as a plasticization effect. Very few works, though, have investigated water aging plasticization effects by directly measuring the material fracture toughness, which should be a mechanical parameter directly affected by such modifications. Linear Elastic Fracture Mechanics toughness parameters such as the critical Stress Intensity Factor (*cSIF* or K_{IC}) provide a consolidated and robust approach for characterization of toughness in brittle thermoset polymers [7]. In fact two main standards are available for this characterization, i.e. ASTM D 5045 and ISO 13586, specific for plain strain crack tip conditions.

One side aspect of measuring the fracture toughness in samples that have been conditioned by water absorption, is the likely presence of internal stresses arising due to non-uniform swelling deformation. Such internal stresses may also be significantly enhanced by the presence of the notch and sharp crack [8].

The authors have recently proposed a new approach to the study of swelling stresses in transparent glassy thermoset polymers, which uses the full-field non-contact technique of Photoelastic Stress Analysis (PSA), demonstrating the potentialities of this technique to monitor the transient internal stress state during water absorption [6,9]. In [8] the technique was also extended to monitor Single Edge Notched Bending samples prepared according to ASTM D 5045 in order to measure the material fracture toughness. Some first results have regarded a DGEBA epoxy sample hydrothermally aged at 80 °C, and have highlighted a strong influence of the swelling stresses in the measure of fracture toughness when the water absorbed has not reached the saturation stage. The PSA technique has also allowed to verify that samples in the saturated stages regain a stress free state and the fracture toughness in this final condition is a reliable indication of the intrinsic fracture toughness of the aged material.

In this work, the standard characterization of fracture toughness assisted by PSA is proposed to compare the behavior of two different epoxy materials: a highly cross-linked DGEBA epoxy, and a commercial low-density cross-linked epoxy system able to cure in room temperature conditions. The PSA and Fracture analyses have been carried out together with gravimetric tests, assessing the absorption kinetics, and Dynamic Mechanical Thermal Analysis (DMTA), highlighting changes in the Glass Transition Temperature, T_g. Aging was performed in a water bath at 50 °C to accelerate time to saturation and maximize the soaked water. Results have evidenced significant differences in the mechanisms of influence of swelling stresses, and the level of water absorbed and material plasticization.

2. Experimental procedure

2.1. Materials and samples preparation

The materials analysed in this work are 2,2-bis[4-(glycidyloxy)phenyl]propane (DGEBA), epoxide equivalent weight 172-176, cured by 4,4' diamino-diphenyl sulfone (DDS), both supplied by Sigma Aldrich and an epoxy resin supplied by Mates Italiana srl, with trade name of SX10Evo and hardener type M (medium). The first system was prepared by mixing the monomer with a stoichiometric amount of DDS amine, fully dissolved by mechanical stirring at 130 °C for 30 minutes [6]. The two blended systems were casted into a steel plate with a glossy surface finish, able to provide easy release and optimal transparency of the cured panels. The curing process for DGEBA consisted of a permanence at 180 °C for 2 hours, while the Mates SX10 was cured at room temperature. Before the post-curing cycles, the samples were prepared cutting rectangular beams with 36x8x3 mm^3 nominal dimension. These dimensions were also suitable for the DMTA test requirements. The post-curing cycles consisted in the permanence of DGEBA-DDS and SX10 at respectively 200 °C and 65 °C for two hours, followed by a slow cooling to room temperature in 24 hours. Such slow cooling was also able to determine a fully stress free condition in all samples, which were then placed in the thermal bath to start their aging conditioning.

2.2. Gravimetric and DMTA analysis

The samples were aged in a thermal bath at 50 °C in order to accelerate water diffusion with respect to hygro-

thermal conditioning. This relatively low temperature was chosen to keep the SX10 safely away from its T_g, which was measured to be about 90 °C. The choice of the bath temperature was in particular supported by a preliminary check on the SX10 system. This consisted in keeping a sample of SX10 in the oven at 50 °C for one week and compare the DMTA results before and after this purely thermal conditioning. This allowed to verify that SX10 did not degrade and its T_g was little affected by the permanence at 50 °C.

Gravimetric analysis was carried out by weighing a number of samples in an electronic balance with 0.01 mg resolution. Weights were taken at regular intervals after wiping off any residual surface water from the samples. The mass uptake in terms of relative mass change, M_r was evaluated by the following equations:

$$M_r = \frac{M_t - M_i}{M_i} \times 100 \tag{1}$$

where M_t is the mass at the actual conditioning, M_i is the mass at the beginning of absorption [1].

Dynamin Mechanical Thermal Analysis (DMTA) was performed on a Rheometrics DMTA V in a single cantilever beam arrangement at a heating rate of di 5 °C/min, frequency of 1.8 Hz and elongation of 0.02%. In this work, the temperature at the peak of the $Tan\delta$ curve is considered as indicative of the Glass Transition Temperature T_g.

The DMTA and Fracture Toughness characterisations were carried out at three specific times:
1) start of the aging conditioning (material in the stress free post-cured condition);
2) at the reach of a maximum internal stress state according to the photoelastic analysis;
3) at the reach of complete stress relaxation of the DGEBA system (i.e. after about 1300 hours).

2.3. Photoelastic stress analysis

The stress field measured by the PSA arises from the initial non uniform swelling induced by the slow water ingress and diffusion within the sample. In fact, the initial stages of water absorption determine a swelling of the sample outer zones, reached by the water. This initial swelling is though restrained by the internal kernel of material, not yet reached by water. This determines a state of self-equilibrated stresses generating compression near the surfaces and traction in the kernel [6]. The nature and photoelastic behavior of such swelling stresses in polymers has some strong similarities with the case of residual stresses in tempered glasses, where similar photoelastic methods have been applied [9,12-15].

It is well known that PSA allows the quantitative evaluation of the principal stresses difference (σ_1-σ_2). This quantity is directly related to the relative retardation δ that can be obtained by combining digitally acquired images with different angular positions of the optical elements (phase shifting methods [16]). If the analysis is carried out in zones where the isoclinic parameter θ=0 (where θ is the angle between the x axis and the closer principal stress direction, see Fig. 1), one particularly convenient phase shifting scheme is the Tardy Phase Shifting [9]. This is able to determine the retardation by combining only three images acquired in monochromatic light with three angular positions of the analyzer, β_A. In this case the three emerging intensities $I$and the retardation are given by:

$$\beta_A = 0^\circ \rightarrow I_1 = I_f + \frac{I_0}{2}\left(1 - \cos 2\pi\delta\right) \qquad \text{(dark field circular polariscope)} \tag{2a}$$

$$\beta_A = 45^\circ \rightarrow I_2 = I_f + \frac{I_0}{2}\left(1 \pm \sin 2\pi\delta\right) \tag{2b}$$

$$\beta_A = 90^\circ \rightarrow I_3 = I_f + \frac{I_0}{2}\left(1 + \cos 2\pi\delta\right) \qquad \text{(light field circular polariscope)} \tag{2c}$$

$$\delta = \frac{1}{2\pi} arctan \frac{I_1 + I_3 \pm 2I_2}{I_3 - I_1} \tag{3}$$

where the upper and lower sign in (2b) and (3) are respectively for the angle $\alpha = 0°$ and $\alpha = 90°$ between the principal stress σ_1 and the horizontal reference axis x (see also Fig. 1 for a schematic identification of directions and angles).

The Tardy Phase Shifting was successfully applied on un-cracked samples, where the vertical symmetry axis can be easily verified to be also a zero order isoclinic [6]. In the case of a SENB cracked sample the presence of the singularity is expected to have a significant influence on the stress field distribution at the crack tip. The isoclinic parameter along the vertical symmetry axis of the SENB sample needs to be determined in order to conclude that the Tardy Phase Shifting can still be applicable. This was done in this work by implementing a second phase shifting procedure in white light and with a plane polariscope [17]Introduction. The isoclinic is in particular obtained by combining four images corresponding to the angles $\omega = 0°$, $\omega_2 = 22.5°$, $\omega_3 = 45°$, $\omega_4 = 67.5°$ of the analyzer and polarizer couple with the horizontal reference x axis. The four emerging intensities are given by:

$$I_{i,j} = I_{f,j} + I_{w,j}\sin^2 2\left(\theta - \omega_i\right) \text{ with } i=1,2,3,4 \text{ corresponding at the four angles } \omega \text{ and } j = r, g, b \tag{4}$$

By considering the average of the three red, green and blue images for each angle ω:

$$I_i = I_r + I_g + I_b \,. \text{ with } i=1,2,3,4 \tag{5}$$

the isoclinic parameter can be determined by:

$$\theta = \frac{1}{4} arctan \frac{I_4 - I_2}{I_3 - I_1} \tag{6}$$

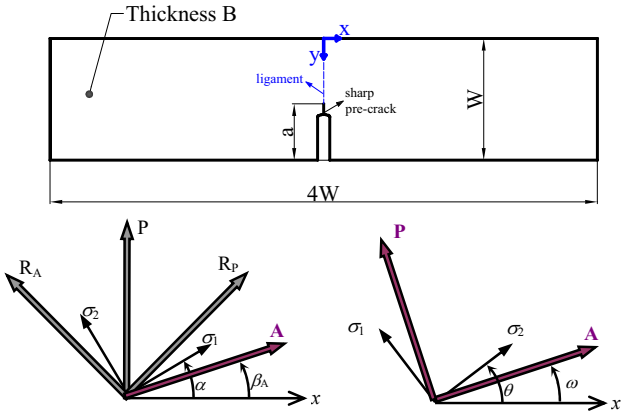

Fig. 1. scheme of SENB sample (top), and definition of axes for the circular and plane polariscope in the Tardy Phase Shifting (bottom left) and isoclinic determination (bottom right).

2.4. Fracture Toughness

The value of the critical stress intensity factor K_{IC} is determined from SENB specimens prepared according to ASTM D5045-96. In particular a span of 32 mm was set, for a sample height of about W=8 mm and a value of B compatible with the required range of 2B<W<4B (see also Fig. 1 and ASTM D5045) [7]. Samples we prepared by first machining a notch with a diamond band saw of thickness 0.4 mm. Then a crack tip from the notch root was obtained by razor tapping. The transparency of the sample allowed to control the straightness of the crack front and the length of the razor tapping grown crack, which were compliant with [11]. Furthermore, the size criterion for the existence of Plain Strain conditions was largely satisfied for both materials.

3. Results and discussion

3.1. Gravimetric data and DMTA

Figure 2 shows the curve of mass uptake for the two epoxy systems. Both materials exhibit an absorption behaviour fairly close to a one dimensional Fickian diffusion model [1,6]. The samples monitored had nearly identical dimensions, hence SX10 has absorbed a significantly less amount of water than DGEBA up to 1300 hours. It is here observed that both systems have not yet reached a fully saturated condition, although, as will be shown in the next sections, the DGEBA system reaches a complete stress free condition at 1300 h.

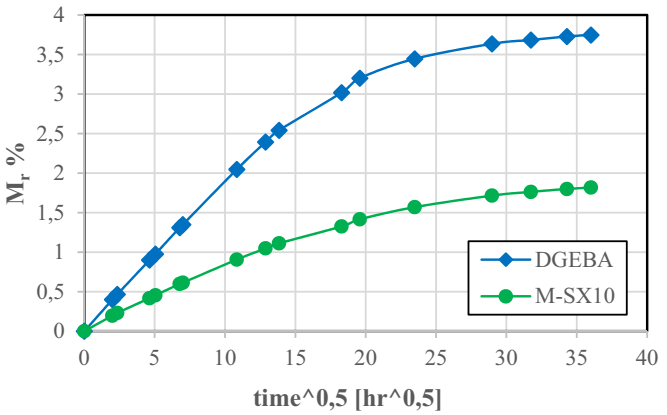

Fig. 2. Mass uptake curve of the analysed epoxy systems.

Figure 3 shows the $tan\delta$ curves from DMTA at the three monitored stages. It is in general confirmed that DGEBA is a highly cross-linked system, with a T_g in not aged conditions of about 230 °C, versus 90 °C of the SX10. It is suggested that the lower crosslink density in the SX10 determines a more flexible network structure, which is able to compact more, so reducing the amount of free volume [10]. This would explain the lower amount of absorbed water.

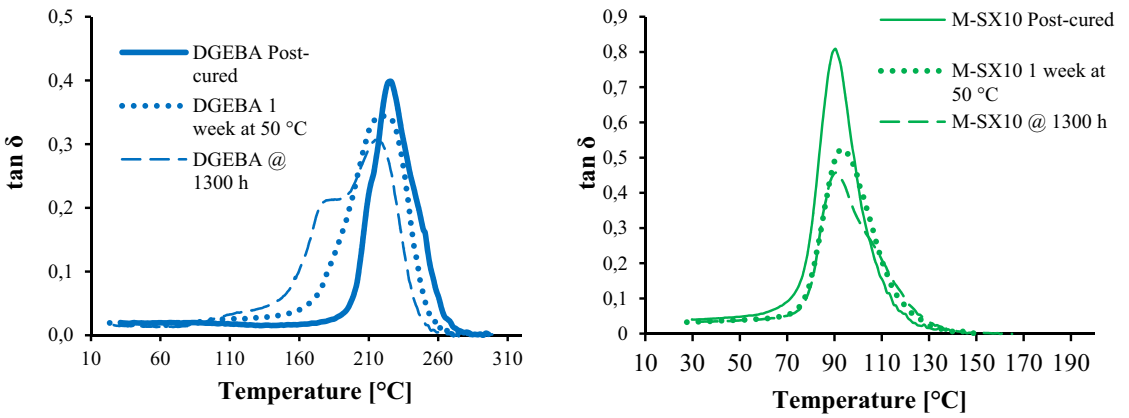

Fig. 3. $tan\delta$ vs temperature curves from DMTA.

Regarding the transformations induced by aging, DGEBA confirms the behaviour already observed at higher aging temperatures [6,8], i.e. there is a reduction of T_g and a widening of the $tan\delta$ curve. At 1300 hours in particular there is

a marked shoulder towards lower temperatures, with the presence of a double peak [1]. The behaviour for SX10 is quite different. In fact, the main peak does not move significantly, indicating a stable T_g. It is instead noticed the formation of a small shoulder towards higher temperatures. This effect might indicate an attempt of the materials to post-cure, probably induced by the long permanence at 50 °C. This should determine an embrittling effect more than a plasticisation, since a higher crosslink density reduces the network mobility.

3.2. Swelling stresses analysis: un-notched samples

In order to perform a quantitative PSA, the photoelastic constant of the material is needed. This was measured on beam samples of dimensions 90×15×3 mm^3, tested in four point bending [6]. The photoelastic constant was in particular measured on the not-aged condition and at 1300 h of aging conditioning. The not-aged material gave the following values: C_A=6.53x10^{-5} mm^2/N and C_{SX}=2.77x10^{-5} mm^2/N, respectively for DGEBA and SX10. The aged material gave the following values: C_A=5.2x10^{-5} mm^2/N and C_{SX}=3.4x10^{-5} mm^2/N, respectively for DGEBA and SX10. It is then observed a reduction for the plasticised material, i.e. DGEBA, and an increment for the embrittled material SX10. These results are in line with other experiences made by the authors on other epoxy systems, all indicating that a decreasing T_g usually correlates with a reduction of the photoelastic constant C and vice versa.

PSA of un-cracked rectangular beam samples allowed monitoring the swelling stresses that arise during the absorption process. Figure 4 shows images of the isochromatics from a circular polariscope in white light at the time when the maximum σ_x-σ_y value is reached on the border along the longer side of the beam sample.

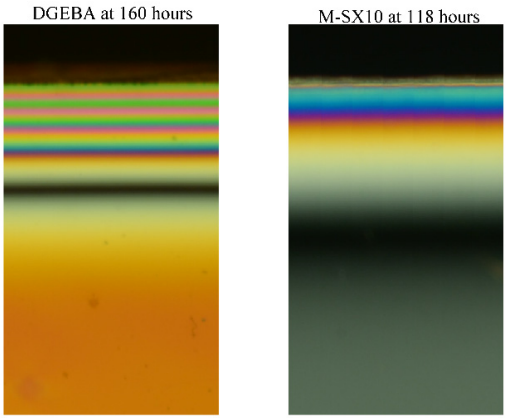

Fig. 4. maps of isochromatic on un-notched samples from a circular polariscope in white light.

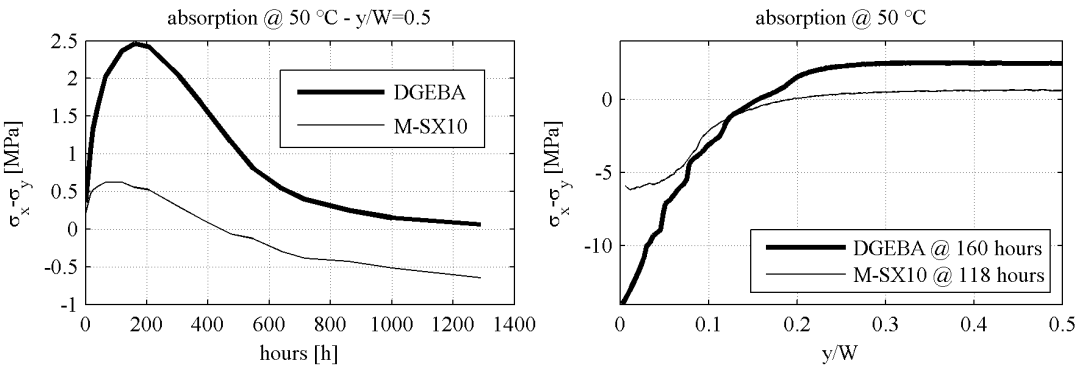

Fig. 5. plots of σ_x-σ_y along the vertical symmetry axis versus time (left) and position (right).

Figure 5 reports results of the isochromatic retardation along the vertical symmetry axis of the sample, from the edge, y/W=0, to the centre of the sample, y/W=0.5. It has been observed by the authors that the results of the Tardy Phase Shifting analysis provide in particular the difference of the normal stress components σ_x-σ_y along the y axis [6,9]. It is in particular observed that the DGEBA system reaches significantly higher stresses (more than three times) then the SX10, in both compression (near the edge) and tension (in the inner central area). It is also observed that when the DGEBA system regain a fully stress-free condition, i.e. complete relaxation due to the swelling of the sample kernel, the SX10 has instead a negative peak of σ_x-σ_y, indicating that the swelling is still non uniform.

3.3. Swelling stresses analysis: cracked SENB samples

The evolution of isochromatics in the SENB samples is shown in Fig. 6, where the central crack zone is shown at different times from the aging start: 0, 40, 375, 1300 hours for DGEBA (figure 6, top row), and 0, 25, 118, 1300 hours for SX10 (Fig. 6, bottom row). Some very peculiar differences can be observed in the behaviours of the two epoxy systems. In fact, the DGEBA system is developing a stress concentration at the crack tip since the very early stages of water absorption. High stresses set along the straight border opposite to the notch, around the notch and along the flanks of the crack. Such stresses reached a maximum after about 40 h. At 1300 h the DGEBA system is almost fully relaxed in the ligament section, i.e. from the crack tip to the straight edge.

In the SX10 system there seems to be no sign of stress concentration at the crack tip in the early stages of absorption (e.g. see 25 h and 118 h). The isochromatic fringes at the notch root at 25 h are parallel to the round profile of the notch, while the flanks of the crack seem to not influence the isochromatics. It seems that there is a stress concentration only due to the notch singularity and not to the sharp crack singularity (as observed for DGEBA). This behaviour is well explained by the lack of water penetration within the crack. The crack is perfectly closed by the compression stresses developed at the notch root, and the material is not absorbing water through the crack flanks, as it seems to happen for DGEBA. Moreover, with the progression of the aging time the crack tip of the SX10 starts to develop a stress concentration well visible at 1300 hours, i.e. when the DGEBA is fully relaxed. This seems to suggest that the crack flanks of the SX10 begin to locally absorb water at a later time, when the sample edges (notch and straight borders) have almost fully relaxed their stresses.

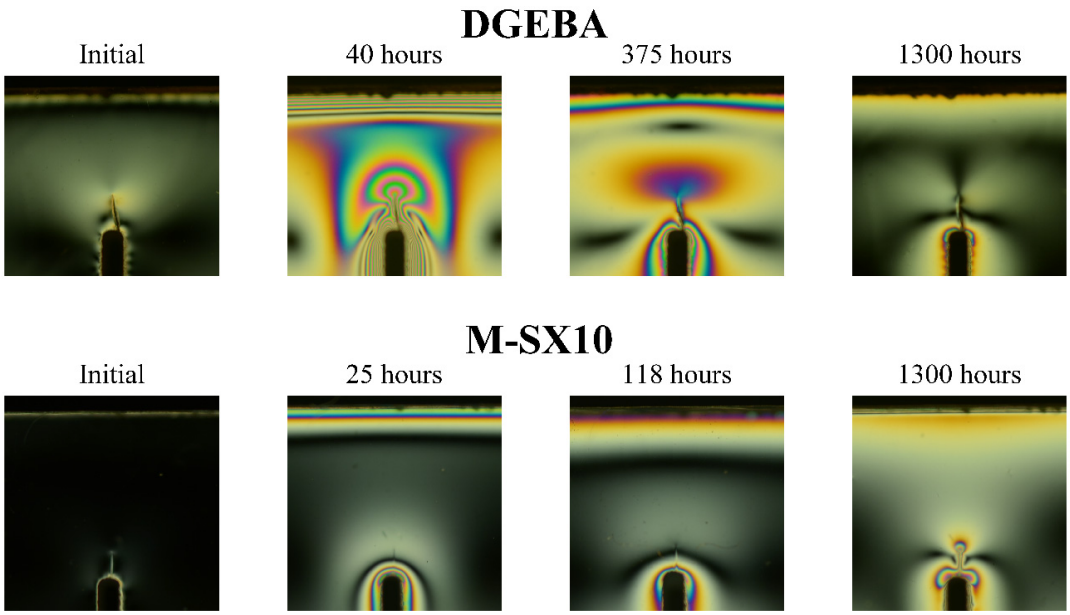

Fig. 6. Evolution with aging time of isochromatic maps from SENB samples for DGEBA (top row) and SX10 (bottom row).

Figure 7a shows the plots of σ_x-σ_y versus the aging time at a point of the ligament y/W=0.9 (i.e. very near the crack

tip), while Fig. 7b shows σ_x-σ_y in all points of the ligament at four times. It is again observed that the DGEBA system reaches much higher stress values. The zone ahead of the crack has an increment of σ_x-σ_y that is believed to be balanced by a compression between the flanks of the crack, i.e. in the wake of the crack (see also [8] for a more extended discussion of this effect). From Fig. 7b it is observed that σ_x-σ_y at the crack tip of SX10 is decreasing from 25 hours to 300 hours. This is in agreement with what observed in Fig. 6 at the crack tip of SX10 at 1300 hours, indicating that a negative stress concentration is arising due to local swelling.

Figure 8 shows the maps of the isoclinic parameter θ from a DGEBA and a SX10 SENB samples. These maps have been obtained by the phase shifting procedure outlined in section 2.3. In this work it is only observed that the value of θ along the vertical symmetry axis, i.e. in the ligament going from the crack tip to the opposite sample edge, has a value of θ=0°. This means that the σ_x and σ_y stress components in the ligament are also principal stresses, and it is possible to apply the three images Tardy Phase Shifting simplified method.

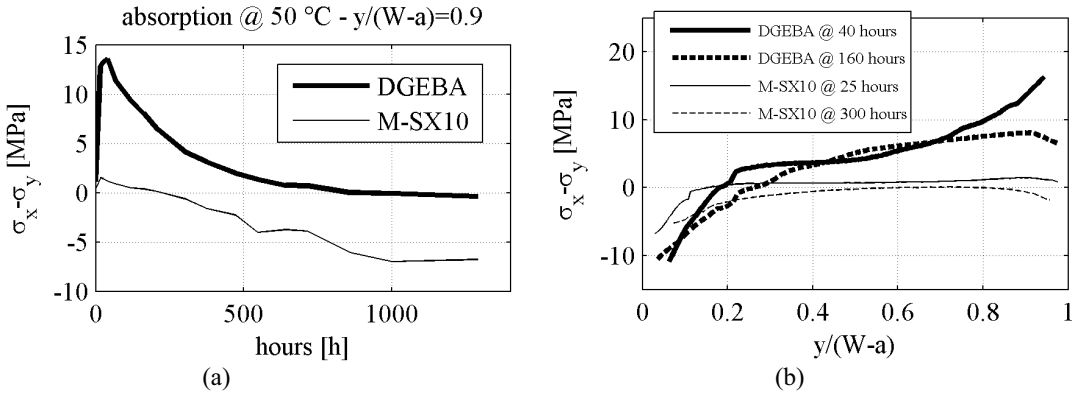

Fig. 7. plots of σ_x-σ_y along the vertical symmetry axis (ligament) versus time (left) and position (right).

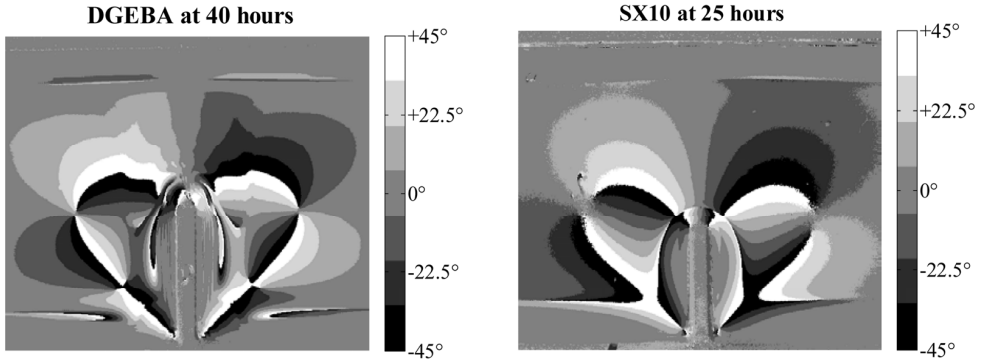

Fig. 8. Maps of isoclinic parameter over the centre cracked zone of a SENB sample.

3.3. Fracture toughness evaluation

Figure 9 summarises the measured values of fracture toughness K_{IC} at the three stages of observation. Results for DGEBA confirm the behaviour already observed with hydrothermal aging at 80 °C [8]. It is in particular confirmed the increment of fracture toughness due to initial high swelling stresses (one week aging), which is due to the compressive stresses arising in the wake of the crack. Since the material is almost stress free at 1300 h (which is a near saturation condition for DGEBA), the increment of K_{IC} here is explained by the plasticisation induced by water in the material, also confirmed by the DMTA.

Regarding the SX10 it is seen that its initial fracture toughness is higher than the DGEBA. This was expected since

SX10 has a much lower T_g and hence it is a more ductile epoxy. It is also found that K_{IC} is not changing significantly after one week aging, and this result can be correlated with the DMTA, where it was also observed that the T_g of the material is substantially unchanged. It is finally observed that K_{IC} at 1300 hours is slightly higher. This increment is probably correlated to the local compression stresses arising around the crack tip in SX10, and hence is not correlated to any aging induced changes in the material network structure.

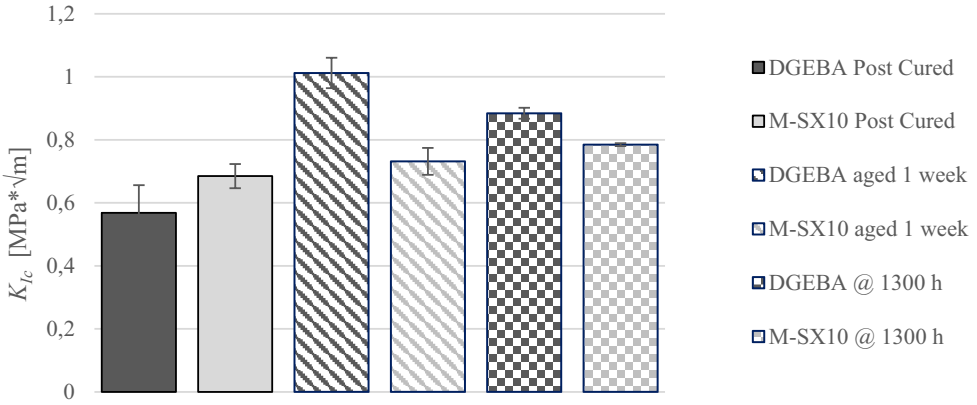

Fig. 9. Values of Fracture Toughness K_{IC}.

4. Conclusion

In this work Photoelastic Stress Analysis has allowed to monitor the development and evolution of swelling induced stresses in rectangular beam and cracked Single Edge Notched Bending epoxy samples. The qualitative and quantitative interpretation of the isoclinic and isochromatic fringes has in particular allowed to determine the nature of the stress field arising near the crack tip during aging.

The proposed analysis has been applied to the study of two epoxy systems differing for the crosslink density: a high temperature thermally cured DGEBA+DDS, achieving a T_g of 230 °C, and a commercial epoxy resin, SX10, cured at room temperature, and achieving a T_g of 90 °C. The different crosslink densities also determine a lower fracture toughness, measured by K_{IC}, for DGEBA, due to its more rigid structural network. Both systems have been aged in a thermal bath of water at 50 °C.

In general it has been observed that DGEBA absorbs a significantly higher amount of water than SX10. Furthermore the water is able to penetrate the sharp crack introduced in the SENB sample by razor tapping. This generates a stress concentration at the crack tip and in the wake of the crack, which enhances the measured value of K_{IC}. As water absorption approaches saturation, DGEBA regains a fully stress-free condition, meanwhile the DMTA reveals a progressive reduction of T_g and a widening of the $tan\delta$ curve, which should indicate a plasticisation of the polymer network. The value of K_{IC} in this near saturation condition has indeed confirmed this plasticisation, indicating an increase with respect to the un-aged condition.

The SX10, on the contrary, evidences a stable fracture toughness throughout the initial stages of absorption, during which the DMTA also evidences a stable T_g. It was in particular observed also that water was initially not able to penetrate the flanks of the sharp crack, and the stress field at the crack tip remains unaffected. With the progression of aging, water finally penetrates into the crack, but only when the straight edges of the sample are already stress relieved. This creates a local stress concentration state of compression along the flanks of the crack and at the crack tip, determining an increase of K_{IC} which is not related to material transformations.

In general this work has confirmed the precious contribution of Photoelastic Stress Analysis in investigating the reciprocal influences of swelling stresses and material network changes in the water aging of glassy thermoset polymers. In particular Photoelasticity is able to provide also a quantitative analysis by the implementation of optimised Phase Stepping techniques. In general this methodology requires low cost equipment, simple laboratory set-ups, and is able to reveal swelling stresses with high sensitivity. It though requires a specific know how of the

optical technique, which is probably not common among polymer scientists.

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