

# Temperature as an Accelerating Factor for Long-Term Durability Testing of FRPs: Should There Be Any Limitations?

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**Abstract:** Fiber-reinforced polymer (FRP) composites are increasingly being used in civil engineering applications due to their numerous advantages. Moreover, some environmental conditions can potentially enhance their long-term durability. Therefore, the study of their long-term behavior is crucial to ensure their durability. To perform durability study in a reasonable time limit, accelerating factor, such as high temperature, is generally used. However, the use of very high temperature of conditioning could amplify the reduction of the properties leading to conservative prediction of long-term properties. The present paper attempts to clarify the effects of high temperatures on the mechanical and barrier properties (moisture absorption) of GFRP's internal reinforcement, by presenting some experimental results and conclusions of laboratory accelerated studies.

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## Introduction

The civil engineering industry is known for a long time as conservative regarding the use of new products. However, the success of recently developed materials has forced the construction industry to modify its conventional approach. New technologies involving, for example, the use of fiber-reinforced polymer (FRP) composites has drawn a particular attention from design engineers and administrators of public works and infrastructures. There is a large-scale resistance to the use of FRP composite materials. It is partially based on the lack of information regarding the long-term durability behavior of these materials in critical conditions of service, to maintain their structural performance in severe and variable environmental conditions under sustained loads. The loss of mechanical and barrier properties, leading to an increase of moisture absorption, of FRP composites during their service life is a crucial issue for the development of their applications in civil engineering structures.

The use of accelerated aging tests to simulate the field conditions, to evaluate their residual mechanical strength, and to predict their long-term property loss is very common to the researchers working in this field. In accelerated aging tests, the material is aged at a high rate using an accelerating factor, generally temperature, according to the Arrhenius law. This law assumes that the degradation of a compound/material is caused by the same process in a certain temperature range. The effect of time and temperature will become equivalent in this temperature range. Thus, the effect of time may be simulated by increasing the temperature.

Many research works, based on the Arrhenius law, are carried out to investigate the durability of GFRP materials under different environmental conditions representing the actual service conditions. Porter et al. (1997) in their work, exposed three different types of E-glass FRP bars (manufactured using an isophthalic polyester resin) to high alkaline solution and at maximum temperature of 60°C for a period of 2 to 3 months. Their test results indicate that the accelerated aging severely reduces the ultimate tensile strength and maximum strain capacity of GFRP bars. DeJke (1999) reports that glass fibers are known to degrade in the presence of water, and moisture can decrease the glass transition temperature ( $T_g$ ) of the resin. It acts as a plasticizer, potentially having significant effect on the flexural strength. The reaction of GFRP composite with alkali of concrete is one of the major durability concerns for design engineers. Typically, concrete environment has high alkalinity, which depends on the design mixture of the concrete, and the type of cement used (Diamond 1981; Taylor 1987). This alkaline environment damages glass fibers by reducing the toughness, strength, and embrittlement. Recently, Chen et al. (2007) studied the durability of bare FRP bars and bars embedded in concrete immersed in different solutions at different temperatures and time intervals of exposure. The writers report that GFRP bare bars and those bars embedded in concrete show significant strength loss when exposed to simulated envi-

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**Table 1.** Mechanical Properties of the Used 9.5-mm Diameter GFRP Bar

Property	Unit	Value	COV (%)
Average ultimate tensile strength	MPa	653	6
Tensile modulus of elasticity	GPa	38.5	5
Guaranteed tensile strength <sup>a</sup>	MPa	608	7
Design tensile strength <sup>a</sup>	MPa	426	4
Flexural strength	MPa	888	4

<sup>a</sup>According to ACI 440.1R.06 [American Concrete Institute (ACI) 2006].

ronments, especially in solutions at high temperatures of 60°C.

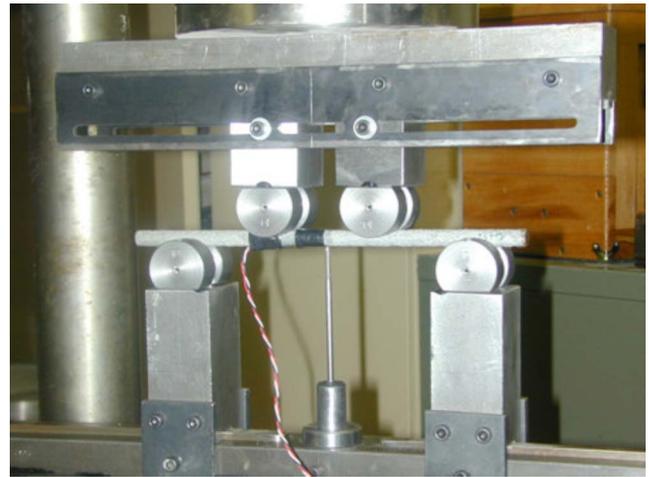
Glass fibers are damaged due to the combination of two processes: (1) chemical attack on the glass fibers by an alkaline cement environment and (2) concentration and growth of hydration products between individual filaments (Murphy et al. 1999). The embrittlement of fibers is due to the nucleation of calcium hydroxide on the fiber surface. The hydroxylation can cause fiber surface pitting and roughening. These act as flaws severely reducing fiber properties in the presence of moisture. In addition, calcium, sodium, and potassium hydroxides found in the concrete pore solutions aggressively affect glass fibers. Therefore, the degradation of glass fibers is not only due to high pH level, but also due to the combination of alkalis, high pH, and moisture. The reaction rates of all these degradation phenomena increase with temperature. The degradation phenomenon of FRP composite material subjected to accelerated aging in aqueous solutions is being assumed as a set of simple reactions related to the same mechanisms of degradation during the whole range of conditioning temperatures. Many researchers are selecting very high temperatures (up to 80°C) to get the maximum rate of aging (Bank et al. 1998; Bakis et al. 1998; Dejke 2001) to evaluate the durability of GFRP reinforcing bars. But the use of very high temperature of aging could lead to overdegradation of the polymer matrix and underestimation of the durability of GFRP materials. It is for these reasons that standardized test methods for the accelerated test for alkali resistance of FRP reinforcing bars were established [American Concrete Institute (ACI) 2004; CAN/CSA-S806 2002]. According to ACI 440.3R and CSA S806, the temperature of aging shall be equal to 60°C.

The present paper aims to explain the effects of the use of high temperature of aging on the reaction rate of degradation of FRP composite, submerged in an aqueous solution. Experimental results are presented regarding the loss of flexural properties and the increase in moisture diffusion at saturation.

## Experimental Program

### Material

The GFRP bars used in this study have a diameter of 9.5 mm. The bars were made of continuous E-glass fibers impregnated with a vinylester resin using a pultrusion process. Table 1 presents the tensile and flexural properties in terms of average, ultimate, guaranteed, and design tensile strengths (according to ACI 440.1R.01) and average flexural strength. The mass fraction of glass determined by thermogravimetric analysis according to ASTM E1131 standard was 74.5%. The relative density according to ASTM D792 standard was equal to 1.95.



**Fig. 1.** Experimental setup for four-point flexural tests on FRP bars

### Conditioning of GFRP Bars

The specimens were completely immersed in distilled water at four different temperatures (23, 40, 60, and 80°C), and were removed from the water after 40, 100, and 120 days. After each period, usually six GFRP bar samples were removed from the water and tested under flexure to compare their flexural strength retention values to those of the reference specimens.

### Flexural Tests

Flexural strength tests were conducted according ASTM D4476 (ASTM 2003) standard. This method was used with some modifications such as using full bars instead of half bar (cut longitudinally) (Zhang et al. 2007). An overhang of 10% of the supported span was allowed at each support. Fig. 1 shows the experimental setup specially designed to evaluate the flexural strength of FRP bars. The tests were carried out using a 270 kN capacity manually operated universal testing machine. The specimens were loaded under load control at a rate corresponding to an approximate stress of 5 MPa/s in the bar, approximately.

### Absorption Tests

Moisture uptake measurements were periodically recorded by removing some specimens from distilled water, wiping them dry, and measuring their weight immediately, and this is continued until their saturation, according to ASTM D570 standard. The percentage moisture uptake was calculated with Eq. (1), where  $%M$  = mass gain due to moisture uptake;  $M_{\text{cond}}$  = mass of the specimen after its conditioning; and  $M_{\text{dry}}$  = mass of the specimen before its conditioning. The gain in mass was corrected to take into account possible mass loss of the specimens due to various dissolution phenomena, such as hydrolysis, by drying later completely the specimens by placing them in an oven at 100°C for 24 h, and comparing their masses to their initial masses. The recorded mass losses were around 0.1%. Eqs. (2) and (3) were used to determine the mass loss after immersion and corrected mass gain of specimens, respectively. In those equations,  $%M_{\text{loss}}$  = mass loss of the specimen after conditioning due to various dissolution phenomena;  $M_{\text{redry}}$  = mass of the specimen completely dried after conditioning; and  $%M_{\text{corr}}$  = corrected mass gain due to moisture uptake.

$$\%M = \frac{100(M_{\text{cond}} - M_{\text{dry}})}{M_{\text{dry}}} \quad (1)$$

$$\%M_{\text{loss}} = \frac{100(M_{\text{redry}} - M_{\text{dry}})}{M_{\text{dry}}} \quad (2)$$

$$\%M_{\text{corr}} = \%M - \%M_{\text{loss}} \quad (3)$$

### SEM

Scanning electron microscopy (SEM) observations and image analysis were performed to evaluate the microstructure of specimens and the integrity of the GFRP material after exposure to elevated temperatures. The samples observed in SEM were (1) reference specimens and (2) specimens aged in distilled water at 80°C for 120 days. All specimens were first cut, polished, and coated with a thin layer of gold-palladium by a vapor-deposit process for observing in the SEM. Microstructural observations were thereafter performed on a JEOL JSM-840A SEM. These observations were conducted to determine the potential degradation of the polymer matrix, possible glass fibers, and interface.

### TMA

Thermomechanical analysis (TMA) experiment was carried out in a TA Instrument Q-400 thermomechanical analyzer. The test was performed on the transverse section of the bar, perpendicularly to the fiber orientation, to measure the change of the coefficient of thermal expansion (CTE) related to the resin. Approximately 50 mg of sample was placed on the sample holder and heated to 150°C at a rate of 5°C/min. Output from the TMA consists of the dimension change in micrometers of the GFRP bar specimen as a function of temperature. The slope of the curve gives the CTE of the specimen.

## Experimental Results

### Flexural Strength Loss

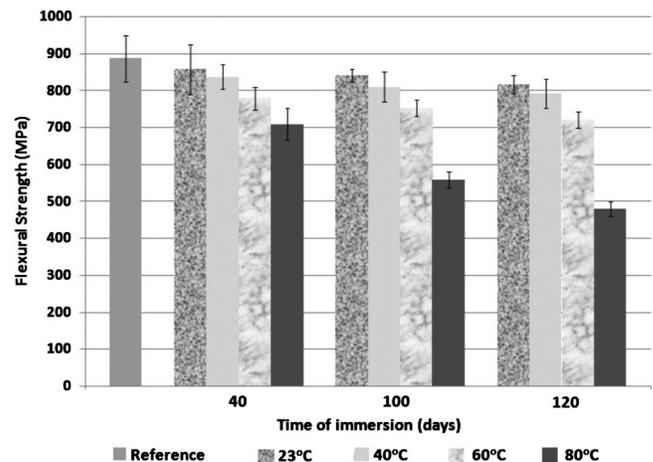
Table 2 and Fig. 2 show the experimental results obtained during the flexural tests of aged bars tested after immersion, respectively. Fig. 3 shows flexural strength loss observed with GFRP reinforcing bars conditioned in distilled water during 120 days. Experimental data of Table 2 and Fig. 2 show that the effect of temperature on the mechanical properties, such as tensile or flexural strength, of FRP composites submitted to an accelerated aging, using temperature as accelerating factor, is not constant between room temperature and 80°C. Fig. 3 shows that the material degradation is larger between 60 and 80°C than between 40 and 60°C, which is itself larger than between room temperature and 40°C. Since the magnitude of the mechanical strength loss is larger at higher temperatures, the predictions based on elevated temperatures (>60°C) may underestimate the long-term durability of the material and overestimate the degradation (i.e., hydrolysis) or modification (i.e., plasticization) of the polymer matrix. After immersion, optical microscope observations were conducted and no discoloration or debonding of the sand-coating of the GFRP bar was observed.

**Table 2.** Experimental Flexural Strength of Reference and Conditioned Specimens

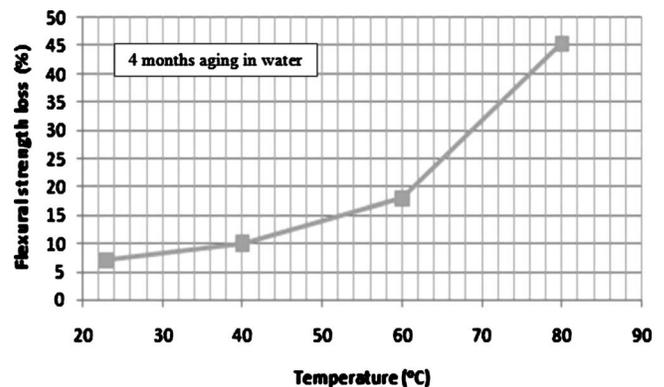
Time of immersion (days)	Temperature (°C)	Average flexural strength (MPa)	COV (%)	Flexural strength retention (%)
0	23	888	7	100
40	23	858	8	97.5
	40	838	4	95.2
	60	780	4	88.6
	80	710	6	80.7
100	23	842	2	94.6
	40	811	5	92.2
	60	753	3	85.6
	80	560	4	73.6
120	23	818	3	93.0
	40	792	5	90.1
	60	722	3	82.0
	80	481	4	54.7

### Mode of Failure

Fig. 4 shows the mode of failure during flexural tests for reference and specimen aged at 80°C during 120 days. It can be seen in Fig. 4 that the tested specimen was loaded until rupture occurs in the extreme tensile fibers and that aged and reference GFRP



**Fig. 2.** Flexural strength after different agings in water



**Fig. 3.** Flexural strength loss as a function of temperature

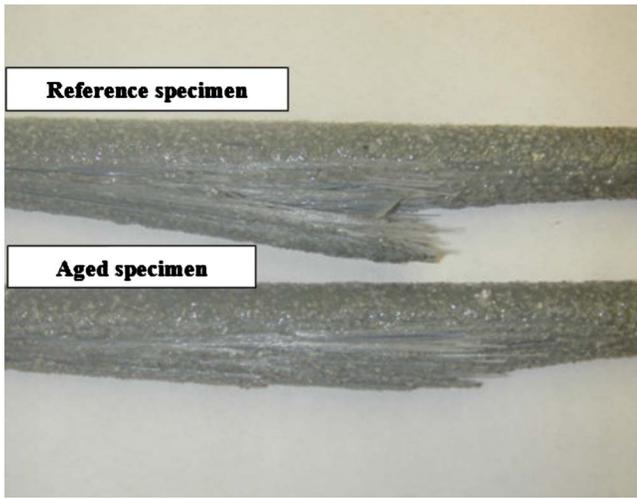


Fig. 4. Typical mode of failure during flexural tests

bars have similar mode of failure. Note that the extreme compressive fibers did not show any premature compression failure. Progressive damage propagation occurred through matrix cracking, fiber breaking, and interfacial debonding of the extreme tensile fibers side of specimens.

### Moisture Absorption

Fig. 5 presents the mass gain of GFRP bars specimens immersed in distilled water as a function of time (Wang 2005). The results presented are the average values obtained from five test specimens. As expected, higher temperature and longer time of immersion resulted in greater mass increase. The measured saturation levels are approximately equal to 0.8% for specimens immersed at 23, 40, 60°C and to 1.8% for specimens immersed at 80°C. This observation indicates a major difference between diffusion of moisture at low (23, 40, and 60°C) and high temperatures (80°C). This can be explained by a thermomechanical phenomenon as described in the discussion section below.

### Microstructural Observation

The micrographs presented in Fig. 6 show the surface of the reference GFRP bar specimen and those conditioned in distilled

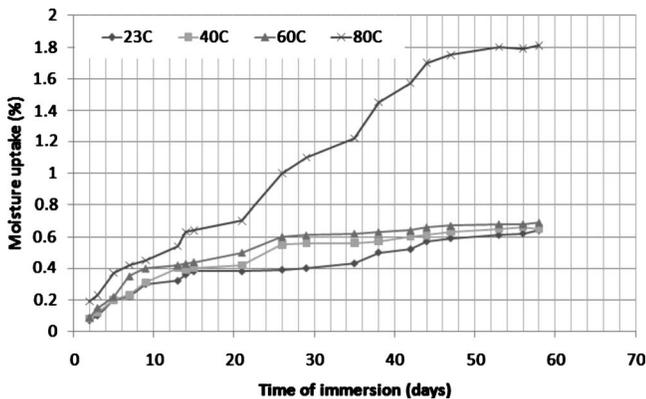


Fig. 5. Average mass gain of GFRP bars immersed in distilled water as a function of time

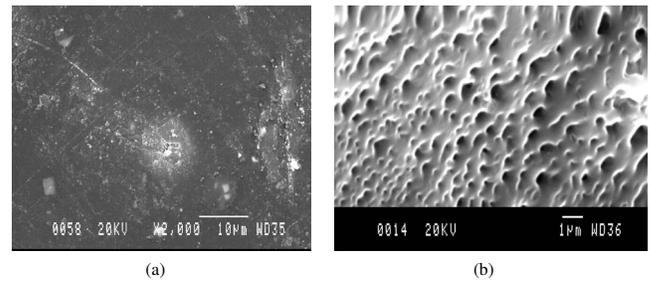


Fig. 6. Micrographs of the external surface of GFRP bar

water at 80°C for 120 days. The comparison of these micrographs shows that significant damage occurs to the polymer resin for GFRP bar conditioned in distilled water at 80°C for 60 days (b) compared to that of the reference sample (a). The presence of voids and microvoids on aged specimen surface can explain the increase of the rate of absorption of solution and of the mass increase at saturation. These observations confirm the degradation of the polymeric matrix of GFRP bar immersed in distilled water at high temperature and explains the loss of mechanical properties. The solution potentially reacts with the vinylester via hydrolysis process which increases at high temperature.

### Thermomechanical Measurements

Fig. 7 presents the dimension change of the GFRP bar specimens as a function of temperature. When the temperature of the material (around 80–85°C) becomes close to the  $T_g$  of the resin (between 95 and 130°C), the transverse CTE rapidly increases since it is highly dependent on the resin. The measured transverse CTEs are approximately equal to 29  $\mu\text{m}/(\text{m}^\circ\text{C})$  below 80°C and to 108  $\mu\text{m}/(\text{m}^\circ\text{C})$  above 85°C. This observation indicates also a major difference between dimension change at low (23, 40, and 60°C) and high temperatures (80°C). This can be in similar way explained by a thermomechanical phenomenon as described in the section below.

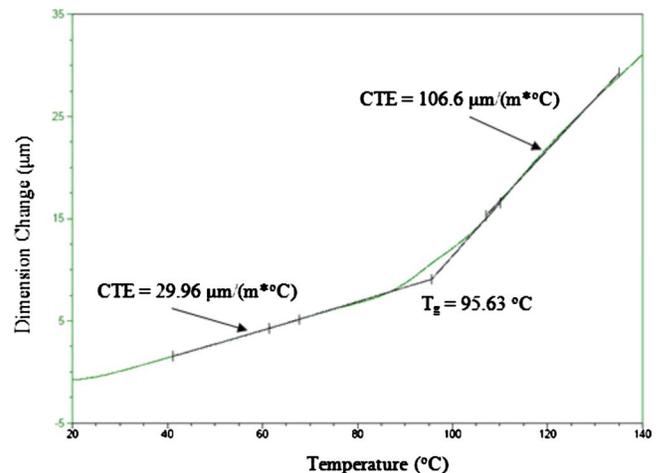


Fig. 7. Dimension change of GFRP bar as a function of temperature

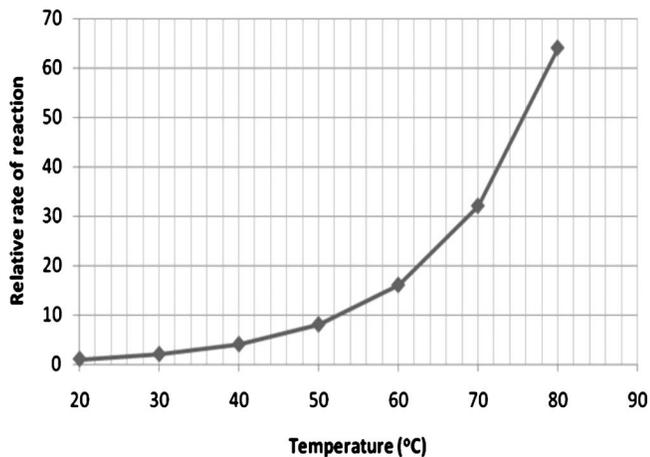


Fig. 8. Relative rate of reaction as a function of temperature

## Discussion

The exponential increase of the loss of flexural strength and the increase of the moisture absorption at saturation with conditioning temperature can be related to two mechanisms. The first one is kinetic, which could be the most important, and the second one is thermomechanical.

### Kinetic Phenomenon

It is assumed that the mechanical strength loss of FRP materials occurring during their conditioning under different situations (type of environment, temperature, loading, etc.) is related to simple chemical reactions. Furthermore, it is assumed that the level of retained mechanical properties is inversely proportional to the level of degradation, i.e., the rate of chemical degradation of the material. In the case of glass fibers/vinylester composites subjected to a conditioning in an alkaline solution, it reacts with the glass fibers through mechanisms of dissolution/leaching/etching and also with the vinylester through a hydrolysis process. The Arrhenius equation relates the temperature to the rate of reaction as per the following relation:

$$k = A e^{-E_a/RT} \quad (4)$$

where  $k$  is the rate constant;  $E_a$  = energy of activation;  $R$  = gas constant; and  $T$  = absolute temperature.

In most of the reactions, this equation shows that for each 10°C increase, the rate increases by a factor of 2 to 3 (generally close to 2). Fig. 8 shows the effect of the temperature increase from 20 to 80°C on the rate of reaction, which doubles for every 10°C increase. It is observed that the increase in reaction rate is almost linear between room temperature (23°C) and 50°C, whereas at higher temperatures (more than 60°C) the rate is characterized by a rapid (exponential) increase of the reaction/degradation. Consequently, it is assumed that a small increase of temperature in the highest temperature range would lead to a maximum mechanical loss. Comparing Figs. 3 and 8, the effect of temperature on the behavior of the flexural strength of a GFRP composite conditioned in water confirms that it is similar to the rate of a simple chemical reaction.

Fig. 9 shows the flexural strength loss of GFRP bars after 120 conditioning days as a function of a reaction rate increasing by a factor of 2 for each 10°C increase. By plotting these data, we obtain a straight line which shows that the mechanical properties

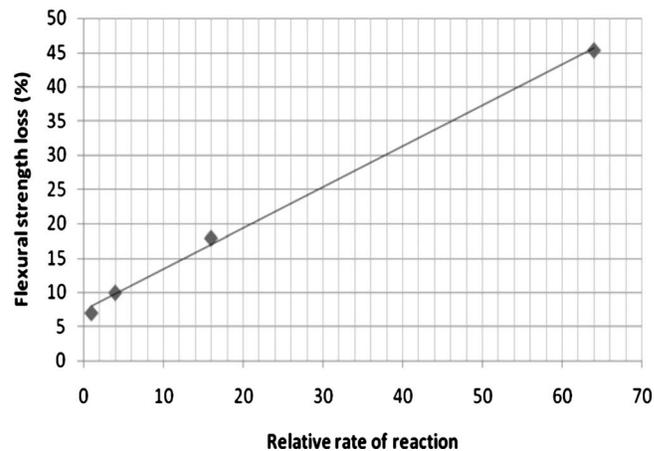


Fig. 9. Relationship between flexural strength loss and relative rate of reaction

may be treated similar to simple chemical reactions. Therefore, it becomes possible to calculate the accelerating factor as a function of temperature from a reference temperature corresponding to a mean annual temperature in the field (e.g., 6°C in Montréal according to Environment Canada). It has to be noted that in regions of large temperature variations such as Canada, 1-year aging at an average temperature is not equivalent to 1-year aging at temperatures varying around this median temperature. In fact, for two areas having the same average temperature, the degradation will be lower in the region having the lowest temperature variation because of Arrhenius equation, which shows that the degradation is not linearly proportional to the temperature but increases exponentially. Fig. 10 shows the accelerating factor values as a function of conditioning temperatures for a reference temperature equal to 6°C. From this curve, the accelerating factor corresponding to 60°C is equal to 40, which means that the degradation level of a material conditioned during 1 year at 60°C should be the same to that level after 40 years at 6°C, for the same material.

### Thermomechanical Phenomenon

As required by Arrhenius theory, the degradation mechanisms affecting a material have to be, quantitatively and qualitatively, the same on an entire range of temperature used to perform acceler-

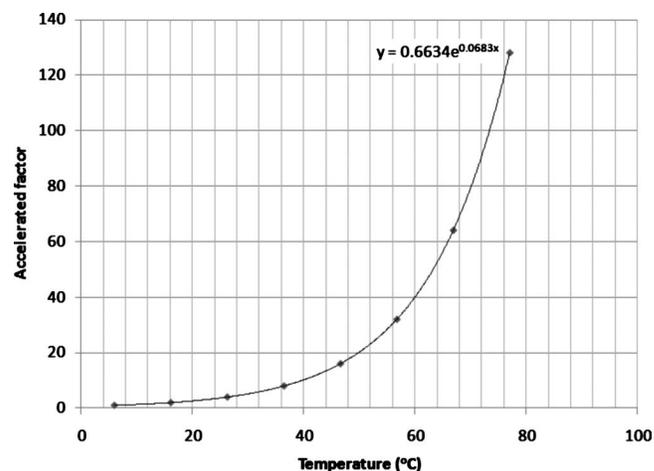
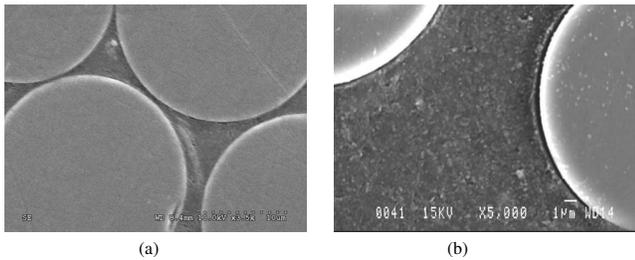


Fig. 10. Accelerated factor as a function of temperature



**Fig. 11.** Micrographs of the fiber/matrix interface of GFRP bar

ated aging tests where temperature is an accelerating factor (Bank et al. 2003). GFRP materials are composed of glass fibers, which are very stable for a very large temperature range. However, the polymeric phase cannot sustain such a large range of temperature. When the glass transition temperature  $T_g$  is reached (between 95 and 130°C for vinylester resins), the structure of the resin changes from a glassy state to a viscoelastic state. Moreover, some changes can be observed before  $T_g$ , and it is recorded that certain properties begin to be affected at temperatures as low as  $T_g=50^\circ\text{C}$  (Turi 1997). One of the main characteristics of the resins which is affected around  $T_g$  is the thermal expansion. In its vitreous state and far below  $T_g$ , the thermal expansion coefficient of the composite resin is constant. When the temperature of the material becomes close to  $T_g$ , this coefficient, and consequently the free volume, rapidly increases (Fig. 7), leading to a less dense material. Furthermore, the stiffness and modulus of the material start to decrease. These two phenomena create a more porous material, which can absorb a greater amount of solution (Fig. 5), enhancing the degradation mechanisms. In the case of glass fibers/vinylester composites, it is likely that the solution diffusing through the material will fill the interfacial free space and start reacting with the surrounding molecules, i.e., the vinylester resin and/or the silica bonded together. Since the amount of water and eventually dissolved ions will be more concentrated at the interface than in the bulk resin, the reactions of degradation will be harsher in this region. For example, the hydrolysis reaction leading to the rupture of vinylester chains could be highly localized at the interface and almost nonexistent in the rest of the material. Consequently, the amount of hydrolyzed chains could be very low (and maybe undetectable) as compared to the total amount of resin present in the material and concentrated (in the interface region) at the same time. Since the interface region is already the weak region of composites, even small changes occurring at this level may dramatically affect the behavior of the whole material by reducing the stress transfer between the fibers.

In addition, it is to be noted that the thermal expansion coefficients of the resin and glass fibers are approximately equal to  $33 \times 10^{-6}$  and  $9 \times 10^{-6}$  mm/mm/ $^\circ\text{C}$ , respectively. When the material is heated, this relatively large difference between the two components of the bars generates shear-type stresses mainly located in the region where the two components are in contact. This may induce a peeling/debonding phenomenon and the occurring of an empty “gap” at the interface. Fig. 11 shows the fiber/matrix interface for reference GFRP bar and bar aged in air at 80°C for 2 h. It can be seen that there was significant damage (delamination) to the interface between the polymer matrix and the glass fiber after conditioning at high temperature. The use of higher temperature or thermal cycling can exacerbate the degradation phenomenon. Moreover, it has been observed that the thermal expansion coefficient of the bars in the transverse direction, which is controlled by the resin expansion (whereas the longitu-

dinal one is controlled by the fiber expansion) is not constant in the entire range of temperature as it increases when the material is above 40 or 50°C (Gentry and Hudak 1996; Gentry and Husain 1999; Masmoudi et al. 2005). Therefore, the thermomechanical-induced degradation mechanisms would be amplified as this temperature range is reached. In fact, the thermal expansion coefficient of the resin would rapidly increase leading to a more porous material increasing the moisture diffusion in the composite material. These mechanisms would lead to degradation phenomenon, such as hydrolysis and delamination at the interface between fiber and resin, which does not correspond to those occurring under the service conditions of GFRP bars.

## Summary and Conclusions

The degradation of GFRP materials conditioned in aqueous solutions being related to chemical reactions of degradation (which may involve several different chemical processes), the effect of temperature is the most affecting factor as compared to other factors, such as time or sustained loading. For example, if the duration is taken as a parameter of degradation, it can be easily understood that the level of degradation should be linearly proportional to the duration as long as the composite material is fully and equally saturated. In this case, doubling the time will double the degradation level. Contrarily, the effect of temperature is more important since the chemical degradation rate is not proportional but increases exponentially with the temperature.

From these observations and discussions, it may be concluded that the models used to extrapolate an accelerated aging to the real service life have to take into account the phenomena presented. The use of too elevated temperatures, higher than 60°C, which represents the superior limit for accelerating aging of vinylester based GFRP bars according to this study, would not only accelerate the kinetic of degradation and the reactions involved at lower temperatures. It would also modify the thermomechanical-induced degradation mechanisms themselves. The resin matrix structure is not only affected as  $T_g$  is reached. It may also occur at lower temperatures. Dynamic thermomechanical data have shown that the flexural modulus, i.e., the stiffness of the material, begins to decrease several tenth degrees below  $T_g$  (Turi 1997). Such a softening of the resin “ignites” potential degradation reactions, through changes brought to properties such as porosity, polymer chain mobility, moisture diffusion, or hydrolysis. In this case, the degree of degradation would be not only higher than those occurring under real service conditions of the GFRP bars (this is the objective of accelerated aging testing using Arrhenius theory), but it would induce uncontrolled degradation mechanisms, inhibited at lower temperatures, leading to an overestimation of the loss of properties, and consequently to an underestimation of the long lasting of GFRP in service. The maximum temperature of aging in alkaline solution recommended by the ACI 440.3R and CSA S806 standard test methods is 60°C.

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