

# The Combined Effect of Temperature and Humidity on the Fatigue Parameters and Reliability of Optical Fiber

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## ABSTRACT

The lifetime of an optical fiber depends on its environment. Previous work extensively measured and characterized the separate effects of humidity and temperature on the fatigue parameters using three different kinetics models, but the combined effect has not been determined in detail. In this work, the details of how the fatigue parameters vary with temperature in a humid environment were investigated. It was found that the kinetics model parameters were different from values obtained elsewhere in liquid water. This may be the result of differences in the apparent activation energy for fatigue in liquid and vapor environments.

Keywords: optical fiber reliability, lifetime models, humidity, temperature

## 1. INTRODUCTION

Most models currently used for making lifetime predictions of optical fiber do not explicitly incorporate the details of the service environment.<sup>1</sup> Therefore there is an implicit assumption in such predictions that the environments relevant to the lifetime prediction (*i.e.* service and proof test environments as well as the environment used to measure the fatigue parameters) are all the same. In general these environments are not the same but some compensation for this can be made by using “worst case” fatigue parameters (*e.g.*, as in Ref. 2). However, clearly, if there are significant deviations between the test and service environments reliability predictions will be erroneous. Specifically, environments more aggressive (hotter and more humid) than typical laboratory test environments of  $\sim 25^{\circ}\text{C}$  and 50% humidity will lead to earlier failure while benign environments (*e.g.* space applications with extremely low water content) will result in much longer lifetimes than the models predict. It is therefore clearly desirable to have lifetime models that explicitly incorporate the character of the service environment. For this we need detailed information on how the fatigue parameters vary with environmental factors such as temperature, humidity and pH; preferably over a broad range of parameter values.

In earlier work<sup>3</sup> the effect of humidity on the fatigue parameters of pristine fiber at  $25^{\circ}\text{C}$  was extensively studied and the results were interpreted in terms of three kinetics models (the power law and two exponential forms). It was found that the chemical kinetics model proposed by Wiederhorn and Bolz,<sup>4</sup> which assumes that the stress at the crack tip modifies the activation energy of the chemical reaction via an activation volume, provides the most consistent description of the effect of humidity. Similar work that examined the effect of temperature in liquid aqueous environments<sup>5</sup> did not favor any one of the three kinetics models but did show that the interpretation of the results in terms of an activation barrier for fatigue does depend on which form is used. However, a general result was found; the stress dependence of the activation entropy is the dominant factor and that the activation enthalpy does not alone explain the observed temperature dependence. The activation entropy increases with applied stress. A generalized chemical kinetics model for fatigue suggested that the apparent activation parameters are not unique but will depend on the environment to some extent.<sup>6</sup> This was later validated by comparing fatigue in pure water and in pH 7 buffered water.<sup>7</sup> This therefore means that the activation parameters will not be the same in liquid water and water vapor.

Up until this point, the effects of humidity and temperature have been studied independently of each other or have only been studied in terms of their effect on strength rather than on the fatigue parameters. Duncan, France and Craig<sup>8</sup> presented strength data which imply that the strength degradation can be solely described by the dew-point temperature,

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thus combining the effects of temperature and humidity into one variable. However, this suggests that strength is dependent only on the thermodynamics of the equilibrium between the vapor and condensed phases of water and does not depend on the kinetics of the reaction between water and silica, which is clearly wrong. Armstrong *et al.*<sup>9</sup> examined this question in detail and found that the dew point temperature was not the controlling parameter - therefore the kinetics of the reaction between water and silica does indeed play a role.

In the work presented here, we examine the dependence of both the strength and the fatigue parameters on temperature in water vapor which complements the earlier work of Shiue and Matthewson for liquid water.<sup>5,7</sup> Since interpretation of such results can depend on the mathematical model used to analyze the data, three different models will be used. The models describe how the crack growth rate,  $dc/dt$ , depends on the applied stress intensity factor,  $K_{IC}$

$$\text{Model 1: } \frac{dc}{dt} = A_1 \left( \frac{K_I}{K_{IC}} \right)^{n_1}, \quad (1)$$

which is designated the "power law".<sup>10</sup>

$$\text{Model 2: } \frac{dc}{dt} = A_2 \exp n_2 \left( \frac{K_I}{K_{IC}} \right), \quad (2)$$

which assumes the activation energy for fatigue is reduced by an amount proportional to applied stress intensity.<sup>4</sup>

$$\text{Model 3: } \frac{dc}{dt} = A_3 \exp \left( n_3 \left( \frac{K_I}{K_{IC}} \right)^2 \right), \quad (3)$$

which assumes the activation energy is reduced by an amount proportional to the strain energy release rate, *i.e.* is proportional to the square of the stress intensity. This form may be found by simplifying<sup>10</sup> a more general form proposed by Lawn.<sup>11</sup>

While of different mathematical forms, the three models each involve two parameters which have the same meaning in each model. The  $A_i$  ( $i = 1 \dots 3$ ) are pre-exponential terms which represent the overall crack growth rate and the  $n_i$  which represent how sensitive the rate is to the applied stress. If fatigue is treated as a chemical reaction whose activation energy is modified by the applied stress, then all three models can be incorporated into absolute rate theory to give a general form for  $A_i$ :<sup>5,6</sup>

$$A_i = c_0 \frac{k_B T}{h} f(C) \exp \left( -\frac{\Delta H_0^*}{RT} + \frac{\Delta S_0^*}{R} \right), \quad (4)$$

where  $c_0$  is the increase in crack length per breaking bond at the crack tip,  $k_B$  is Boltzmann's constant,  $T$  is absolute temperature,  $h$  is Planck's constant,  $f(C)$  is some function of the concentration,  $C$ , of the reacting species,  $R$  is the gas constant,  $\Delta H_0^*$  and  $\Delta S_0^*$  are the activation enthalpy and entropy for crack growth in the limit of zero applied stress. The same theory shows that the  $n_i$  parameters can be expressed as a linear function of reciprocal temperature:

$$n_i = \frac{b_H}{RT} + \frac{b_S}{R}, \quad (5)$$

where  $b_H$  and  $b_S$  represent how the activation barrier for the fatigue process is reduced by the effect of stress on the enthalpy and entropy components of the barrier. In general, the function  $f(C)$  will depend on temperature since  $C$  can depend on temperature (for example the partial pressure of water for a vapor environment or the concentration of hydroxyl ions in liquid water) so that if an  $m$ th order reaction is assumed:

$$f(C) = \alpha C^m \quad (6)$$

then the apparent activation energy for fatigue is given by

$$E_a = \Delta H_0^* + m \Delta H^*, \quad (7)$$

where  $\Delta H^*$  is the activation enthalpy for the concentration of the reacting species. This shows that the activation enthalpy (and for similar reasons the activation entropy) depend on the nature of the environment.

While  $b_H$  and  $b_S$  have been determined for liquid environments,<sup>5</sup> they have not been determined for a humid environment. The purpose of this work is to address this issue since it should not be assumed the values of these parameters would be the same in all types of environment.

## 2. EXPERIMENTAL

The specimen used in this study was a dual acrylate coated fiber. The fiber strength was measured at five different faceplate velocities (1, 10, 100, 1000, 5000  $\mu\text{m/s}$ ) using a two-point bending apparatus.<sup>12</sup> The strength was measured by allowing the coated fiber to equilibrate in the appropriate environment, which was  $50\pm 1\%$  humidity at temperatures ranging from 5 to  $55 \pm 1^\circ\text{C}$ . Twenty samples were measured at each speed. At the lower speeds, up to ten specimens could be broken simultaneously by supporting the fibers between multi-grooved faceplates. It has been shown previously that if this fiber is properly equilibrated with the test environment the coating will not perturb the reaction kinetics.<sup>3</sup> Therefore, for convenience only coated fibers are studied here. The equilibration periods utilized depended on the temperature of the test environment and ranged from overnight for the lower temperatures to one hour for the higher temperatures. Numerical integration was used to calculate the fatigue parameters  $n_i$  and  $A_i$  for each model from the data at each temperature. The following parameter values were assumed for this analysis: critical stress intensity factor  $K_{IC} = 0.75 \text{ MPa}\cdot\text{m}^{1/2}$ , crack shape parameter,  $Y = 1.16$ , and initial/inert strength  $\sigma_i = 12 \text{ GPa}$  – while the numerical results depend on these values, the trends are insensitive to them and changing these parameters would not change any of the conclusions.

## 3. RESULTS AND DISCUSSION

Figs. 1 and 2 show the calculated values of  $n_i$  and  $A_i$  respectively as a function of reciprocal temperature. Table 1 presents the values of  $b_H$  and  $b_S$  calculated from the slope and intercepts of the best fit regression lines fitted to the data of Fig. 1 using Eq. 5. For comparison purposes, the results from Ref. 5 for pure water and pH 7 buffer are also shown.

It may be shown that slope and intercept of linear fits to the results shown in Fig. 2 may be used to estimate values for  $\Delta H_0^*$  and  $\Delta S_{0,\text{app}}^*$ :<sup>5</sup>

$$\text{slope} = -\frac{\Delta H_0^*}{R}, \text{ and} \quad (8)$$

$$\text{intercept} = \ln\left(c_0 \frac{k_B T}{h}\right) + \ln f(C) + \frac{\Delta S_0^*}{R}. \quad (9)$$

However,  $f(C)$  is not known (or more particularly, the parameter  $\alpha$  in Eq. 6 is not known even if the order,  $m$ , is known) so the apparent activation entropy in the limit of zero stress can be defined:

$$\Delta S_{0,\text{app}}^* = \Delta S_0^* + R \ln f(C), \quad (10)$$

which can be explicitly calculated from the intercept of the regression lines in Fig. 2.  $\Delta H_0^*$  and  $\Delta S_{0,\text{app}}^*$  are shown in Table 2.

To understand the implications of the current results it is useful to examine the earlier results in pure water and pH 7 buffer. The activation parameters shown in Tables 1 and 2 for these two environments are essentially the same within experimental error. This is because these results were obtained for coated fiber which is an effective diffusion barrier to the large ions in the buffer solution so the glass surface effectively sees the same environment in both cases.\* Assuming a temperature of around 300 K for the pure water and pH 7 results, the effect of stress is primarily felt through its influence on the activation entropy since  $b_H/T$  is small compared to  $b_S$  (model 1) or negative which alone would impede fatigue (models 2 and 3). Turning now to the results for the vapor environment, it is first noted that the confidence intervals are quite large because of the narrower range of temperatures explored in the current work. However, it is clear that the results are significantly different from the liquid environments.  $b_H$  is of more importance whichever model is used to interpret the data, because the values are larger than for the liquid environment. This difference in the

\* Interestingly, when the coating is absent, stripped fibers do show different behavior in the two environments since the pH of water varies with temperature while that of the buffer does not.<sup>7</sup>

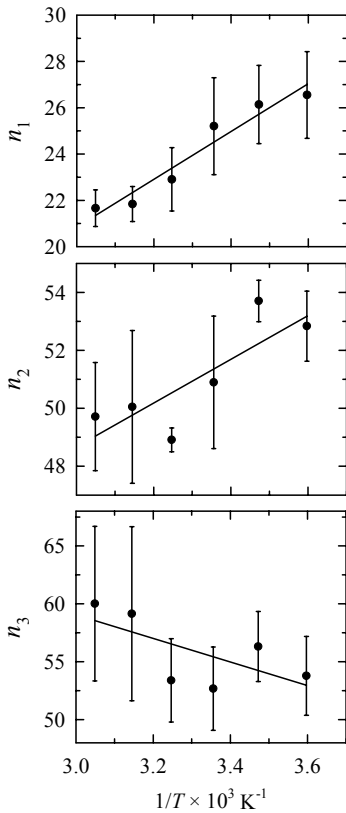


Fig. 1: The fatigue parameter  $n_i$  as a function of  $1/T$  measured at 50% relative humidity.

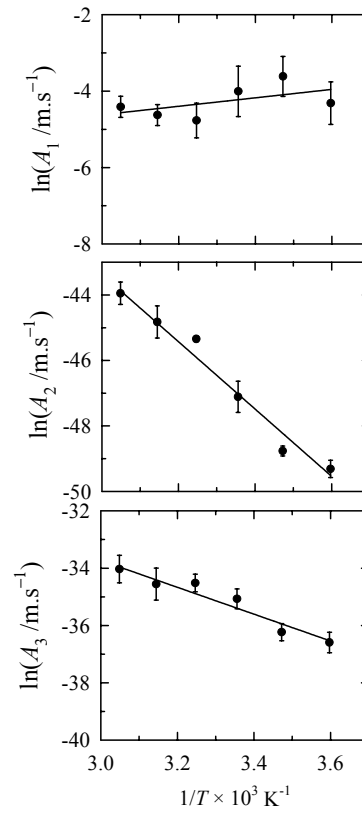


Fig. 2:  $\ln A_i$  as a function of  $1/T$  measured at 50% relative humidity.

sensitivity to stress suggests that the nature of the reactions leading to fatigue are quite different and that the common observation that the strength in liquid is not the same as the strength in humidity in the limit of 100% saturation is not simply explained by a difference in overall fatigue rate, but is actually a different process.

Turning now to the values of the activation enthalpy and (apparent) activation entropy in the limit of zero stress, the entropy plays a bigger role than in the liquid environment, at least for the exponential models. While the above arguments suggest that there is no reason why these parameters should be the same since the reaction paths are different, one expects further differences between  $\Delta S_{0,app}^*$  in liquid and vapor environments because the function  $f(C)$  in Eq. 10 will be different in both cases.

Table 1: Parameters describing the sensitivity of the activation barrier to the applied stress, as defined by Eq. 5. Results for liquid aqueous environments from Ref. 5 are also shown for comparison. The confidence intervals represent a 95% range.

Environment		Model 1	Model 2	Model 3
50% RH	$b_H$ (kJ/mol)	$84 \pm 25$	$53 \pm 29$	$-101 \pm 87$
	$b_S$ (J/mol K)	$-80 \pm 80$	$250 \pm 100$	$800 \pm 280$
Distilled water <sup>5</sup>	$b_H$ (kJ/mol)	$7 \pm 100$	$-130 \pm 30$	$-350 \pm 60$
	$b_S$ (J/mol K)	$180 \pm 30$	$870 \pm 110$	$1700 \pm 200$
pH 7 buffer <sup>5</sup>	$b_H$ (kJ/mol)	$55 \pm 91$	$-110 \pm 40$	$-370 \pm 60$
	$b_S$ (J/mol K)	$160 \pm 30$	$770 \pm 120$	$1700 \pm 200$

Table 2: Activation barrier parameters for fatigue in the limit of zero applied stress. Results for liquid aqueous environments from Ref. 5 are also shown for comparison. The confidence intervals represent a 95% range.

Environment		Model 1	Model 2	Model 3
50% RH	$\Delta H_0^*$ (kJ/mol)	$-9 \pm 16$	$85 \pm 10$	$39 \pm 10$
	$\Delta S_{0,app}^*$ (J/mol.K)	$-80 \pm 55$	$-91 \pm 30$	$-150 \pm 30$
Distilled water <sup>5</sup>	$\Delta H_0^*$ (kJ/mol)	$48 \pm 8$	$-2 \pm 14$	$-4 \pm 9$
	$\Delta S_{0,app}^*$ (J/mol.K)	$50 \pm 30$	$-460 \pm 40$	$-380 \pm 30$
pH 7 buffer <sup>5</sup>	$\Delta H_0^*$ (kJ/mol)	$44 \pm 8$	$5 \pm 16$	$-12 \pm 9$
	$\Delta S_{0,app}^*$ (J/mol.K)	$30 \pm 20$	$-430 \pm 50$	$-400 \pm 30$

#### 4. CONCLUSIONS

Dynamic fatigue measurements on fused silica optical fiber as a function of temperature in a humid environment have been used to characterize the nature of the activation barrier for the fatigue process and how the barrier height is reduced by stress. The results have been found using three different forms for the fatigue kinetics model (the power law and two exponential forms). The values of the parameters are significantly different for each model indicating that care should be taken interpreting fatigue data since the interpretation might depend on the model used. It is therefore recommended that when modeling fatigue and predicting lifetimes for critical applications a range of kinetics models should be considered in order to distinguish general results from results that are merely artifacts of the assumed kinetics model.

The activation parameters in the humid environments are different from the values found in earlier work<sup>5</sup> for liquid aqueous environments. This suggests that the reaction paths in the two environments are different. It is well known that the strength in liquid and high humidity are different – the current results show that this is not simply explained by differences in water concentration/activity but that the fatigue reaction paths are different. It was found in liquid environments that stress decreases the activation barrier primarily via entropic effects (independent of which kinetics model is assumed) and that the effect of stress on the enthalpy component of the activation barrier is either negligible or impedes fatigue. In contrast, we show that while entropy is still important in humid environments, enthalpy also plays a role although the importance of that role depends on which kinetics model is assumed.

The results presented here were obtained for a polymer coated fiber. In other work this fiber was shown to have similar fatigue behavior when stripped of the coating using hot sulfuric acid. In particular the reaction order with respect to humidity was found to be two.<sup>3</sup> However, other coatings may have a significant effect on the fatigue kinetics so caution should be used before applying our results to other coating systems.

The results presented here use short lengths of fiber which have pristine strength and do not contain large defects. For most, though not all, applications reliability is concerned with the behavior of a few large weak extrinsic defects. Since extrinsic defects are different in character from the “defects” in the flaw-free material, the kinetics of fatigue for weak defects should not be inferred from the results for high strength fiber but should be measured directly (see, for example, the paper in this proceedings by Semjonov *et al.* which examines the fatigue behavior of large artificial flaws). However, the approach used here does provide a methodology for interpreting the meaning of fatigue parameters for weak defects.

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