



## Property modelling

## A statistical approach to characterize the viscoelastic creep compliances of a vinyl ester polymer

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## ABSTRACT

The objective of this study was to develop a model to predict the viscoelastic material functions of a vinyl ester (VE) polymer with variations in its experimentally obtained material properties under combined isothermal and mechanical loading. Short-term tensile creep experiments were conducted at three temperatures below the glass transition temperature of the VE polymer, with 10 replicates for each test configuration. The measured creep strain versus time responses were used to determine the creep compliances using the generalized viscoelastic constitutive equation with a Prony series representation. The variation in the creep compliances of a VE polymer was described by formulating the probability density functions (PDFs) and the corresponding cumulative distribution functions (CDFs) of the creep compliances using a two-parameter Weibull distribution. Both Weibull scale and shape parameters of the creep compliance distributions were shown to be time and temperature dependent. Two-dimensional quadratic Lagrange interpolation functions were used to characterize the Weibull parameters to obtain the PDFs and, subsequently, the CDFs of the creep compliances for the complete design temperature range during steady state creep. At each test temperature, creep compliance curves were obtained for constant CDF values and compared with the experimental data. The predicted creep compliances of the selected VE polymer in the design space are in good agreement with the experimental data for all three test temperatures.

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## 1. Introduction

Polymer-matrix composites (PMCs) have gained much interest for structural applications due to their many advantageous properties, such as high strength, good fatigue and durability performance, etc. [1]. The growing use of PMC-based structures is derived in part from the ability to synthesize polymers to achieve the required material properties for a vast range of engineering applications. In some structural applications, PMCs are used in primary and secondary load carrying members where the applied loads are maintained relatively constant throughout the service life of the structure. This constant load produces a time-dependent deformation or creep in the structure. Examples of components which

are subjected to sustained loadings include rotors, turbine blades, pressure vessels, heat engines and other components in power plants and chemical refineries [2].

Creep can lead to a gradual decrease in the effective stiffness of PMC structural members, resulting in unacceptable deformations. Also, exposure of PMC structures to extreme operational environments can result in changes in material properties that translate into structural changes. These can have catastrophic effects on the load-bearing characteristics of composite structures, resulting in creep rupture. Therefore, long-term viscoelastic behavior or creep response under mechanical and thermal loading must be considered in the design of PMC structures.

An additional issue that must be considered for PMC structures is the variability of their viscoelastic properties. In contrast to metals, for which elastic properties are ensured to within  $\pm 5\%$  [3], viscoelastic materials, such as high polymers (polymers with high molecular weight) and PMCs, generally exhibit some variations in their material properties (Young's, shear, and bulk moduli/compliances). Such variability can be of the order of 50–100% and is

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Acronyms and nomenclature			
$C_{1111}$	Creep compliance in longitudinal ( $x_1$ ) direction	$T$	Temperature
$C_{1111}^0$	Instantaneous creep compliance in longitudinal ( $x_1$ ) direction	$t_0$	Initial time of the experiment
$C_{1111}^n$	Prony coefficients in the longitudinal creep compliance Prony series representation, $1 \leq n \leq N_{pr}$	$t_1$	Time at the end of the initial loading phase in creep test
CDF, $F(x w)$	Cumulative distribution function	$t_\infty$	Time at the end of the experiment
DIC	Digital image correlation	$T_g$	Glass transition temperature
$f_\sigma(t)$	Loading function for tensile creep test	$t_m$	Discrete time at which the experimental data is obtained, $1 \leq m \leq M$
$H(t)$	Heaviside unit step function	VE	Vinyl ester
KV	Kelvin–Voigt model	$w$	Distribution parameter
$L(x w)$	Likelihood function	$x$	Observed data
$M$	Total number of sampling points, $1 \leq m \leq M$	$\gamma, \beta$	Weibull distribution shape and scale parameters
MEKP	Methyl ethyl ketone peroxide	$\varepsilon_{11}$	Experimental strain in longitudinal ( $x_1$ ) direction
MLE	Maximum likelihood estimation	$\eta, \omega$	Transformed coordinate system for Lagrange interpolation function
$N_{pr}$	Total number of Kelvin elements (length of Prony series), $1 \leq n \leq N_{pr}$	$\Lambda$	Short-hand notation of the time integral in the Eq. (11)
$N_0$	Number of samples	$\xi$	Integration variable
PDF, $f(x w)$	Probability density function	$\sigma_0$	Constant stress applied for creep loading
PMC	Polymer-matrix composite	$\sigma_{11}$	Applied tensile stress in the loading ( $x_1$ ) direction
$t$	Time	$\sigma_u$	Ultimate tensile stress
		$\tau_n$	Retardation times, $1 \leq n \leq N_{pr}$
		$\psi(T, t)$	Lagrange interpolation function of time and temperature

normally attributed to insufficient quality control in the manufacturing process [3,4]. The stochastic nature of viscoelastic properties of PMCs is primarily due to the viscoelastic response of the polymer matrix [5].

The variability of time-dependent properties of PMCs has been illustrated extensively in the literature. For example, Cook et al. [6] conducted creep tests on seven types of fiber-reinforced composite materials using both thermosets (vinyl ester, polyester, and polyurethane) and thermoplastics (polypropylene) and noted significant scatter (up to 75%) in the creep strain data. Barbero and Julius [7] obtained similar variations of the creep response of thermoplastic polyvinyl chloride, polyethylene blends and a felt-filled thermoset polyester at various temperatures. Additionally, Gnip et al. [8–10] showed substantial experimental variability in compressive creep tests of expanded polystyrene boards. Schwarzl and Zahradnik [11] also observed significant scatter in shear creep compliances in the glass–rubber transition region for both thermoplastic (polyvinyl chloride, polystyrene and polymethylmethacrylate) and thermoset (polyurethane) polymers. Although stochastic models for strength [12–14] and failure [15–17] of PMCs are available in the literature, statistical studies of experimentally obtained viscoelastic material properties, such as creep compliance or relaxation modulus, are very limited. Hilton et al. [18] performed an analytical study to include stochastic processes caused by the variations in linear viscoelastic material properties. However, the experimental data used for statistical analyses in Ref. [12] was generated by assuming a normal probability distribution based on the deterministically obtained relaxation moduli of a solid propellant.

Since the variability of time-dependent properties of PMCs is mainly due to the viscoelastic response of the polymer matrix, this study investigates the creep compliance of a neat polymer matrix. The material of interest is a vinyl ester (VE), which is commonly used as an alternative to epoxies or unsaturated polyesters because of its low cost and high mechanical strength [19]. Thermoset VE resins are typically used in transportation and infrastructure applications such as rail cars, automobiles and bridge decking.

Additionally, due to their superior resistance against a wide range of chemicals, they are also used as a coating in structures such as marine and chemical tanks to protect structures from highly corrosive and thermal environments [19–21].

The objective of this study is to develop a method to predict the creep compliance of a VE matrix with variations in its material properties under combined isothermal and mechanical loading. In subsequent sections of this paper, the experimental program for determining a thermoset VE polymer's viscoelastic properties is described. This includes short-term creep tests that were performed at three temperatures below the glass transition temperature of the polymer. A Prony series representation is used to obtain the time-dependent creep compliances. A statistical approach to determine the Weibull probability density functions for the experimentally derived creep compliances is presented. The Weibull distribution parameters are, subsequently, used to form a predictive model for the selected time and temperature design range.

## 2. Specimen fabrication and creep testing

### 2.1. Material and specimen fabrication

The test articles were fabricated from the commercially available VE resin DERAKANE<sup>®</sup> 441-400 (Ashland Co.) with a styrene content of 33 wt%. The resin formulation included a catalyst promoter, two air release agents and a free radical polymerization inhibitor, as summarized in Table 1. After curing, this formulation

**Table 1**  
Vinyl ester (VE) specimen ingredients.

Ingredient	Weight (g)
DERAKANE <sup>®</sup> 441-400 (vinyl ester resin)	100
Cobalt naphthenate 6% (curing promoter)	0.20
BYK-A 515 (air release agent)	0.20
BYK-A 555 (air release agent)	0.20
MEKP (initiator)	1.00

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