SHORT-TERM CREEP TESTS ON PHENOL-RESORCINOL-FORMALDEHYDE (PRF) RESIN UNDERGOING MOISTURE CONTENT CHANGES

Lech Muszyński†

Assistant Scientist Advanced Engineered Wood Composites Center University of Maine Orono, ME 04469

Fenghu Wang¹

Professor Northeastern Forestry University 26 Hexing Road Harbin, Heilongjiang 150040, China

and

Stephen M. Shaler[†]

Professor and Assistant Director Advanced Engineered Wood Composites Center University of Maine Orono, ME 04469

ABSTRACT

The objective of the study was to develop an experimental technique that would allow determination of the hygro-mechanical properties of thin uniform resin films undergoing moisture content changes; and to use the technique for assessment of the hygro-mechanical performance of phenol-resorcinol-formaldehyde (PRF) resin.

Creep tests on 6 small specimens of PRF film under constant stress (50% of the short-term ultimate stress level), at room temperature ($23^{\circ}C \pm 2^{\circ}C$) and controlled relative humidity (RH) conditions (drying or wetting) were carried out. Digital images of the specimens were acquired using a CCD camera at discrete time intervals during the experiments. Displacements were then measured by comparing successive images using digital image correlation principles. Separation of strain components from total strain recorded during the creep tests was carried out by using data from two reference tests performed on the same material: 1) free deformations of unloaded specimens during drying or wetting conditions, and 2) creep under equilibrium conditions.

The experimental method developed for the study provided a tool to determine hygro-mechanical properties of thin resin films. Quantitative data on material properties of hygroscopic resins determined by means of the technique may be used for modeling the behavior of adhesive bonds as well as adhesive bonded materials in varying climate conditions. The PRF resin revealed a distinct mechano-sorptive behavior, though it seems to be less significant than that reported for wood in transverse directions.

Keywords: Creep, mechano-sorption, adhesive, resin, phenol-resorcinol-formaldehyde (PRF).

INTRODUCTION

Phenol-resorcinol-formaldehyde (PRF) resin is a commonly used adhesive for bonding structural glulam beams. Compatibility of adhesive systems with wood is an important factor determining durability of the resulting composite. Inadequate response of glue-lines to sustained loads under ambient climate variation will result in additional stress develop-

¹ Formerly Visiting Professor, University of Maine. [†] Member of SWST.

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ment, which may eventually break the glueline and promote bond failure.

When subjected to load and climate variations, wood and wood based products exhibit significant hygro-mechanical behavior. Moisture content level affects the elastic and creep behavior, while varying moisture content induces shrinkage or swelling and mechanosorptive creep (Grossman 1976). The bonding of wood to non-wood materials such as fiber reinforced polymer (FRP) plates combined with glue-laminated timber presents an especially severe condition due to dissimilar material behavior (Gardner et al. 1994; Lopez-Anido et al. 2000). This behavior should be considered while designing new wood and hybrid wood-fiber-reinforced-plastics (FRP) composites for structural use. As such, it is important that compatibility of the hygro-mechanical behavior of wood and the adhesive be carefully examined. Of specific interest in this research was the change in mechanical properties due to changes in value and rate of change in the materials' moisture content.

Although numerous researchers have investigated the hygro-mechanical behavior and environmental durability of adhesive bonded wood based products, less attention has been paid to research on the hygro-mechanical performance of the resins themselves or of assessment of their contribution to the overall performance of the products. Tests on thin uniform resin films are less frequently used because of problems encountered in production of uniformly thin, completely cured, and reproducible films and also because they usually demand more sophisticated instrumentation. An advantage of direct measurements on resin films, however, is the possibility of evaluation of the adhesive or matrix performance without any interaction with the bonded material or fiber reinforcement. This evaluation is particularly important when contribution of adhesive/ matrix performance in wood-adhesive or wood/FRP systems is considered in the context of changing climate conditions (Dinwoodie 1983).

An extensive study on the formaldehyde-

based wood adhesives, including tests on thin resin films was performed in 1987-1991 by Bolton and Irle (Bolton and Irle 1987; Irle and Bolton 1988, 1991). The work focused on creep behavior of resin films in different but constant equilibrium conditions. It did not include responses to varying climate conditions, even though the latter are considered as most destructive for bonded systems. The research evaluated phenol-formaldehyde (two types: powdered PF and low alkali PF) and ureaformaldehyde (UF) resins, which may be expected to perform differently from the PRF resins typically used in the manufacture of glulams and wood FRP composite structural elements.

OBJECTIVES

The objective of the study was:

- 1. To develop an experimental technique that allows determination of the hygro-mechanical properties of thin resin films undergoing moisture content changes.
- 2. To use the method for assessment of hygromechanical performance of thin uniform films of PRF resin.

MATERIALS AND METHODS

The test method selected to determine the hygro-mechanical properties of PRF resin undergoing MC changes was 24-h creep of small specimens of the resin film under constant stress, at room temperature $(23^{\circ}C \pm 2^{\circ}C)$ and controlled relative humidity (RH) conditions. Preliminary experiments indicated that a 24-h exposure of the PRF resin was sufficient to assume that the resulting moisture contents reached equilibrium values.

Total deformation of hygroscopic materials subjected to sustained load under constant temperature and nonequilibrium ambient humidity is believed to be a sum of three theoretically separable components, specifically: viscoelastic (ϵ_{ve})—including the immediate or elastic and the delayed response; free shrinkage or swelling (ϵ_{α}); and mechano-sorptive deformation (ϵ_{MS}). Mechano-sorptive deforma-

	Condtioning			 e.					
Test description	Prior to testing	Testing conditions	Load %UTS*		ε _α	€ _{MS}	 Number of specimens 		
Creep and wetting	62% RH	95% RH	50%	×	×	×	7		
Creep and drying	95% RH	62% RH	50%	×	×	×	7		
Creep in 'wet' cond.	95% RH	95% RH	50%	×			4		
Creep in 'dry' cond.	62% RH	62% RH	50%	×			3		
Free swelling	62% RH	95% RH	Unloaded		×		3		
Free shrinkage	95% RH	62% RH	Unloaded		×		3		

TABLE 1. Sets of specimens, conditioning prior and during the tests, applied loads, and the strain components involved.

* UTS - 21 MPa

tion is defined as that portion of the total strain that cannot be expressed as a simple superposition of the previous two (Dinwoodie 1983). Total strain is thus expressed as:

$$\epsilon(c, t, MC, \Delta MC) = \epsilon_{vo} + \epsilon_{\alpha} + \epsilon_{MS}$$
 (1)

While the viscoelastic behavior is affected by the MC level of the material, free shrinkage/ swelling and the mechano-sorptive deformation depend rather on the magnitude of moisture content change.

Neither viscoelastic creep in response to varying moisture content nor the mechanosorptive strain component can be measured directly. In order to determine the magnitude of these strain components, the total deformation of the loaded specimens undergoing MC changes has to be measured and then the particular components separated from the total response. Experimental techniques to accomplish the separation have been developed mainly for wood and wood products (Hisada 1986) and (Wu and Milota 1996). Most of them, however, neglect the influence of apparent MC level on the elastic and viscoelastic response or even neglect the viscoelastic component entirely. The method used in the study adds some original features to technique developed by Muszyński and Olejniczak (1996).

To enable the separation of the components of total deformation, two additional reference tests were performed apart from the main creep test. These tests were:

1. Creep at constant MC, for two reference equilibrium conditions: "dry" conditions at 62% RH and "wet" conditions at 95% RH. The test enabled determination of the influence of apparent MC on the viscoelastic creep component and separation of this component from total deformation.

2. Free shrinkage/swelling test of unloaded specimens in drying and wetting conditions. During these tests, moisture content changes of unloaded specimens in drying and wetting conditions were also recorded. The purpose of this information was to separate the free shrinkage and swelling component of total strains. The data were also used to evaluate the effect of MC on viscoelastic creep component and to determine the equilibrium MC for the "wet" and "dry" conditions used in the tests.

The conditions prior and during the tests, loads and the strain components involved in the main creep test, and the two reference tests are summarized in Table 1. The outputs of the tests are listed in the diagram in Fig. 1.

Specimens

Resin films were prepared from commercial cold-setting PRF resin (GP 4242 Resorsabond with powder hardener). The resin was mixed with the hardener slurry that was prepared before mixing. The mixture ratio was 70:12:18 of resin, hardener, and water respectively and was degassed before casting.

Formation of the thin uniform film of the PRF resin was performed following the description given by Bolton and Irle (Bolton and Irle 1987) for casting UF resin (with proper modifications needed to account for specific properties of PRF).



FIG. 1. Diagram illustrating the outputs of the three groups of tests and the applied calculation procedure.

The films were cast between two glass plates (150 mm by 150 mm) coated with a thin layer of silicone release agent. The resin was poured onto one glass plate, and then the second glass plate was carefully lowered onto the resin, so that no air was entrapped in the resin. Multiple-folded aluminum foil was used to make spacers providing the desired gap of 0.15 mm between the plates. Finally, the plates were pressed together using medium-size paper binder clips. The film was cured under pressure of the clips for 3 to 4 h. The plates were then opened to cut the specimens (38 $mm \times 5 mm$) while the resin was still pliable and thus presumably not fully cured (the degree of cure was not measured). Next, the cut specimens were covered with glass again and left for further curing of three or more days.

The two sides of resin films fabricated in this fashion have a quite different appearance. The bottom surface, which keeps constant contact with glass surface throughout the film formation process, comes out glossy. The topside, on the other hand, falls apart from the top glass surface as the film shrinks gradually in thickness while curing. As a result, the topside of the film appears matt. The difference is also clearly visible when examined using laser scanning confocal microscope in reflec-

tive mode (Leica TCS-SP2). Figure 2a and b show the surfaces under $20 \times$ magnification. The confocal microscope also enabled the roughness analysis of the observed surfaces. Roughness of the surface was calculated from this noncontact surface measurement. The analysis was carried out on sets of images taken at 60 different focal planes with vertical spacing of about 0.7 µm. The roughness characteristics of the two surfaces are summarized in Table 2 (according to ASME B46.1-1995). Although it is possible that the diffusion properties of the two surfaces are different, it was assumed that no significant impact on the experimental results resulted from the film formation process.

All specimens were conditioned at constant temperature $(23^{\circ}C \pm 2^{\circ}C)$ and humidity for about four weeks. The specimen sets designed for testing in drying conditions and in stable wet conditions were preconditioned at a RH of 95%, while those designated for testing in wetting or equilibrium dry conditions were preconditioned at a RH of 62% (see Table 1).

Equipment and the test method

The experimental test setup was designed to allow for control of RH, apply constant loads



FIG. 2. Two sides of the PRF film specimens cast between two glass sheets as seen under Leica DM RXE confocal microscope (magnification $20\times$). a) the top or matt side; b) the bottom or glossy side.

and monitor specimen shapes through visual interrogation (Fig. 3). Salient details of the unit are explained below:

Climate chamber.—All tests were performed at constant room temperature (23°C \pm 2°C) and a constant relative humidity of 62% or 95%. The specified conditions were maintained in a small climate chamber using tanks with two different saturated salt solutions. Drying and wetting conditions in the chamber were obtained when preconditioned specimens were tested in nonequilibrium conditions.

Loads.—In the 24-h creep tests, the constant tensile stress was applied using a calibrated dead load of 0.80 kg. The applied stress

level of 10.5 MPa represented 50% of the ultimate tensile strength (UTS) as determined by static loading on a matched sample of 8 specimens.

Digital image correlation (DIC) method.— Deformation of the resin films was determined using digital image correlation (DIC). This method has been developed since the early 1980s and used to measure deformation and strains of materials under various loading regimes (Bruck et al. 1989; Ranson et al. 1987; Vendroux and Knauss 1998). The method allowed determination of displacements of selected points on the surface of the deformed specimen by comparing successive digital im-

TABLE 2 Roughness characteristics of the glossy and matt surfaces of the PRF film specimens cast between two glass sheets (Leica TCS SP2 laser scanning confocal microscope).

Surface characteristics*	Unit	Top or rough side	Bottom or glossy side
No. of pixels in sampling area	Pixel	558,450	408,816
Sampling area size, A _s	$(\mu m)_2$	299,576	219,306
Average roughness, AR _a	μm	4.64	4.03
Root mean square (RMS) roughness, AR _a	μm	19.46	15.87
Min. area valley depth (AR_v)	μm	18.32	14.72
Max. area peak height (AR _p)	μm	22.25	14.54

* As defired in ASME B46.1-1995: "Surface Texture (Surface Roughness, Waviness and Lay)."



FIG. 3. Schematic of the experimental setup for the creep tests. In free shrinkage and swelling tests, additionally a sensitive balance was added to trace MC changes.

ages acquired during the test with a reference image of undeformed specimen taken before loading. A numerical correlation technique was used to trace displacements of discrete gray intensity patterns of pixels in direct neighborhood of the selected points (reference areas) with sub-pixel accuracy. Once the displacements of the points and their relationships were determined, components of the strain tensors for areas defined by the points could be calculated (Choi et al. 1991; Muszyński et al. 2000) so that a full field representation of displacements and strains for selected areas could be obtained (Sutton et al. 1991). Another advantage of the DIC is that it is a *no-touch* method and is not limited by the size of the specimen. With the use of a proper optical system, DIC may be virtually applied to determine strain fields on specimens of any sizes. In fact, it has been already successfully applied to determine strains in specimens of different materials and scale: like concrete (Choi and Shah 1997), fiber reinforced plastics (Muszyński et al. 2000), wood and paper (Choi et al. 1991; and Sutton and Chao 1988) as well as in individual wood fibers (Mott et al. 1996). The detailed description of the method may be found in (Bruck et al. 1989; Choi et al. 1991; Choi and Shah 1997; and Ranson et al. Peters 1987).

In the present study, most of the digital images were acquired using a CCD camera of moderate resolution (640×480). However, for the free swelling/shrinkage tests, a highresolution (1296×1024) digital camera was used. The images were acquired at discrete time intervals during the experiments. To enhance the image correlation analysis, paint marks were placed on the specimen's surfaces. Displacements of the marks were then measured by comparing successive images using DIC principles as described above. From this, the linear strains were determined.

Free shrinkage and swelling test.—For the free drying and swelling tests, a sensitive balance $(\pm 0.001 \text{ g})$ was used to record changes in weight of a collection of reference specimens. The recorded data were used for calculation of the moisture content changes, which the resin films were subjected to during the drying and wetting cycles. Three unloaded specimens of the PRF resin film were subjected to a sequence of four cycles, each consisting of a 24-h wetting and another 24-h dry-



FIG. 4. Relative humidity (RH) and the specimens moisture content (MC) cycles in the free swelling/shrinkage test.

ing phase as shown in Fig. 4. Free swelling and shrinkage deformation of the specimens were recorded using a high-resolution (1296 \times 1024) camera.

Calculations

The output of the main creep tests in wetting and drying conditions as well as outputs of the two reference tests are listed in the diagram in Fig. 1. Total deformations of specimens undergoing the creep tests with constant and changing MC, determined using DIC, are shown in Fig. 5. Points in the diagram represent average strain values of all specimens undergoing the same MC regime determined for the same instants of test duration. The deformation₃ recorded during tests with wetting and drying include free swelling/shrinkage and the viscoe astic components. The viscoelastic strains recorded after 24-h creep in equilibrium conditions are given in Table 3. From the data on moisture content changes of the specimens acquired during the free shrinkage/ swelling tests, the equilibrium MC for the resin in the dry (62% RH) and wet (95% RH) conditions used during the tests were determined (Table 3).

The following calculation procedure was applied to separate the strain components:

- 1. General models of the MC changes during sorption and desorption phases of the cycles were determined. The models were then used while processing data from the creep tests where the moisture content changes were not recorded.
- 2. The free swelling and shrinkage strains recorded in the supplementary tests combined with data on MC changes allowed determination of the free shrinkage and swelling rates of the resin (Table 4). The results were used to determine general models of the shrinkage and swelling of the resin specimens during the wetting and drying phases of the creep tests. This enabled subtraction of the free shrinkage and swelling components from the total deformation measured during the creep tests with varying moisture contents.
- 3. The viscoelastic component for transient drying and wetting conditions could not be determined by direct measurement. Instead, it was calculated from the data acquired during the creep tests at equilibrium conditions. Simple power equations of the general form:

$$\epsilon_{\rm ve} = \sigma a t^b \tag{2}$$

were fitted to the data acquired during



FIG. 5. Total deformations recorded during the creep tests with constant and changing MC, determined using DIC. Points are determined as averages of points determined for all specimens undergoing the same MC regime for the same time.

creep tests in equilibrium conditions. Values of the coefficients a and b for the wet and dry conditions were determined (Table 3). The dependence of a(MC) and b(MC) for the intermediate moisture contents were obtained by linear interpolation. The creep curves for changing moisture content were evaluated using the following summation formula:

$$\boldsymbol{\epsilon}_{\rm ve}(t_j,\,{\rm MC}(t_j)) = \boldsymbol{\epsilon}_0 + \sigma \sum_{i=1}^J \Delta J(t_i,\,{\rm MC}(t_i)),\quad(3)$$

where $J(t, MC) = a(MC) \cdot t^{b(MC)}$ is a power creep compliance function.

The resulting viscoelastic creep curves for equilibrium and changing MC are shown in Fig. 6. Strains values obtained after 24-h creep with changing MC are given in Table 5. The creep values were then used to subtract the viscoelastic component from the total deformation measured during the creep tests with varying moisture contents.

4. The strains remaining after subtracting the free shrinkage/swelling and the viscoelastic component represent the mechano-sorptive effect (Fig. 7). The mechano-sorptive deformations are believed to be driven by cumulative moisture content change rather than by time (e.g., Rybarczyk 1973; Ranta-Maunus 1975; Grossman 1976). Therefore, to obtain the mechano-sorptive compliance for the resin, the mechano-sorptive strains

TABLE 3. Total elastic and creep strains after 24 h loading in equilibrium conditions (constant MC).

Testing Equilibrium MC conditions for PRF resin	Equilibrium MC	Total clastic and creep	VE model coefficients (Eq. 2)			
	for PRF resin	strains after 24 hours	a	b		
Dry (RH 62%)	14.6%	0.366%	0.7×10^{-3}	0.1480		
Wet (RH 95%)	20.3%	1.260%	4.7×10^{-3}	0.0869		

TABLE 4. Shrinkage/swelling rates and mechano-sorptive compliances for specimens subjected to moisture content change.

TABLE 5.	Compone	nts	of	the	total	stra	in	determ	ined _.	for
specimens	subjected	to	24	h	load	and	m	oisture	cont	ent
change.										

Testing; conditions	Swelling/ shrinkage rate (%/%)	MS compliance I/GPa	Correlation coefficient (R ²) for m
Wetting (RH 95%)	0.148	9.97	0.650
Drying (RH 62%)	0.153	9.44	0.929

Testing conditionsSwelling/
shrinkageTotal elastic
and creepMechano-
sorptive
creepWetting (RH 95%)0.914%1.57%0.626%Drying (RH 62%)0.816%1.15%0.546%

* All specimens tested in drying conditions failed within the first 10 hours of loading.

have to be plotted against the absolute value of the related moisture content change (Fig. 8). A simple linear model equation for mechano-sorptive creep proposed by Rybarczyk (Rybarczyk 1973) and Ranta-Maunus (Ranta-Maunus 1975) was used in order to determine the mechano-sorptive compliance:

$$\dot{\boldsymbol{\varepsilon}}_{\rm MS} = \boldsymbol{m}\boldsymbol{\sigma} | \boldsymbol{\dot{\boldsymbol{u}}} | \tag{4}$$

where m is the mechano-sorptive compliance, u is the cumulative MC change and the dotted symbols represent time derivatives.

For monotonic MC changes, the expression with cumulative MC change |u| may be substituted by $|\Delta MC|$. In addition, when constant stress is used the model equation has the following solution:

$$\epsilon_{\rm MS} = m\sigma |\Delta {\rm MC}| \tag{5}$$

Mechano-sorptive compliances for loaded resin specimens undergoing wetting and



FIG. 6. Viscoelastic creep components of the total deformation: strain data acquired from the experiment for stable wet and dry conditions (gray square and black square), model curves for stable conditions (thin solid gray line and thin solid black line), and model curves for changing MC (thick solid gray line and thick solid black line).



FIG. 7. Mechano-sorptive creep as it appears after subtraction of the viscoelastic strain components.

drying were calculated by fitting the linear model equation to the extracted data. The resulting curves are shown in Fig. 8. The determined mechano-sorptive compliance values are given in Table 4.

The four-step calculation procedure is summarized in the diagram in Fig. 1.



FIG. 8. Mechano-sorptive creep vs. absolute change in MC.

RESULTS AND DISCUSSION

The hygroscopic changes of the cast PRF films were significant with an equilibrium moisture content (EMC) of the film averaging 14.6% and 20.3% at RH conditions of 62 and 95%, respectively. Extrapolation of the resin film EMC to 100% results in values in the range of the fiber saturation point (25-30%) reported for many species of solid wood (Rijsdijk and Laming 1994). Also, the free swelling and shrinkage rates determined in the tests are very similar to those quoted for most of softwood species: 0.15%/% for the PRF resin compared with 0.10%/% to 0.11%/% and 0.25%/% to 0.35%/% in radial and tangential directions respectively for different subspecies of pines (Rijsdijk and Laming 1994).

Creep curves for equilibrium conditions shown in Fig. 6 suggest that after 24 h the PRF films loaded at stress level of 50% UTS approach the viscoelastic creep limit. The observed influence of MC level on creep behavior of PRF resin was much stronger than that reported for UF and PF resins (Bolton and Irle 1987). The creep limit observed for the resin at a MC of 20% was almost three times higher



UF = urea formaldehyde LAPF = lov/ alkali phenol formaldehyde PPF = powered phenol formaldehyde

FIG. 9. Creep of PRF compared with results reported for UE LAPE, and PPF resins for similar conditions (1): a) PRF tested at 62% RH vs. UF and PFs tested at RH of 55%; b PRF tested at 95% RH vs. UF and PFs tested after immersion in water.

than that observed at MC of 15%, while the largest difference reported in (Bolton and Irle 1987) was only ²/₃ of the "dry" value (Fig. 9). The h gh hygroscopicity of the PRF resin as well as the high sensitivity of its mechanical properties to the MC level when compared to other formaldehyde-based resins, may be attributed to presence of cellulosic filler, which is added to the PRF resins with the hardener and to a smaller degree to higher reactivity of resorcinol itself (Dinwoodie 1983; Marra 1992; Pizzi 1983).

The specimens tested under changing MC revealed a significant mechano-sorptive effect. The observed mechano-sorptive deformations

were linear with respect to the absolute value of the MC changes experienced by the resin during the experiment. This linearity of the mechano-sorptive behavior makes it apparently very similar to mechano-sorptive behavior observed by Muszyński and Bąk (1998) on clear wood specimens tested in tension in transverse direction. However, the magnitude of the mechano-sorptive compliance determined here for PRF resin is approximately 10 times lower than that reported for European pine (*Pinus silvestris*) in tangential direction (Muszyński and Bąk 1998).

The proportion of the particular components of total deformations after 24-h testing and a single moisture content change of $\pm 5\%$ (free swelling/shrinkage : viscoelastic : mechanosorptive) appears to be approximately 3:5:2 while wetting and 4:5:2.5 when drying. This result reflects the capability of the PRF adhesives to comply with hygro-mechanical performance of wood without developing excessive stresses, which is important for durability of a bond.

No information on equilibrium moisture contents, shrinkage/swelling coefficients or mechano-sorptive compliances for other formaldehyde-based resins was located in the literature. Similar tests on the resins could enable further comparison of their hygro-mechanical properties.

CONCLUSIONS

The above discussion leads to the following conclusions:

The experimental method designed for the study provided a proper tool to determine hygro-mechanical properties of thin resin films. Particularly, DIC appeared a promising method for determination of deformations for thin resin films. Quantitative data on material properties of hygroscopic resins determined by means of the technique may be used for modeling the behavior of adhesive bonds as well as adhesive bonded materials in varying climate conditions.

The PRF resin revealed a distinct mechano-

sorptive behavior, though it seems to be less significant than that reported for wood in transverse directions.

Despite the disproportion in the mechanosorptive response, the hygro-mechanical properties of PRF resin seem to correspond fairly well with those of wood, which may explain the durability of bonds in glulams and woodcomposite structural elements exposed to varying climate conditions.

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