LOCALIZED DIELECTRIC CURE MONITORING THROUGH THE PANEL THICKNESS DURING ORIENTED STRANDBOARD HOT-PRESSING

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ABSTRACT

In wood composite panel hot-pressing, interactions between resin, wood, and moisture complicate the interpretation of dielectric analysis cure monitoring (also known as impedance cure monitoring) signals. In investigating the application of dielectric cure monitoring to oriented-strandboard (OSB) hot-pressing, pairs of fringe-field dielectric sensors were built into resinless laboratory strand mats at various locations through the thickness and hot-pressed with one sensor exposed to a thin layer of phenol-formaldehyde resin. Temperature and gas pressure probes were also implanted into the mat. The experiments thus yielded base comparisons of localized temperature, gas pressure, and dielectric conditions at various locations ranging from the core to the surface, and an indication of the isolated resin curing effect. The results indicate that the dielectric signal is strongly affected by internal temperature and moisture content gradients as well as by the resin polymerization. Speculation regarding a relationship with the thermodynamic energy of the bound water is introduced. Considering this, these experiments advance the understanding and interpretation of dielectric signals, and may subsequently improve the application of such dielectric cure sensors for the optimization of wood composite hot-pressing.

Keywords: Dielectric, heat transfer, hot-pressing, moisture, oriented strandboard (OSB), resin cure, temperature.

INTRODUCTION

Dielectric analysis (DEA) cure monitoring (also known as impedance cure monitoring) is an emerging technology making inroads into hot-press control in the wood-composite panel industry (Magil and Van Doren 2000; Wang and Winistorfer 2003). The technology is based on electrical impedance measurements of dielectric permittivity and loss-factor changes in a wood-composite mat as its adhesive resin cures during hot-pressing manufacture. Non-invasive DEA sensors can be embedded in the platens of the hot-press, thereby giving real-time monitoring of the dielectric properties of the wood-composite with the potential for intelligent feedback control of the hot-pressing process (Magil and Van Doren 2000).

DEA has been successfully used to monitor the cure state of various thermosetting polymers (Pethrick and Hayward 2002). However, in wood-composite panel hot-pressing, interactions

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between the thermosetting adhesive resin, wood, and moisture complicate the interpretation of DEA signals (Wolcott and Rials 1995a,b).

This paper summarizes the results of localized temperature, gas pressure, and dielectric measurements taken in phenol-formaldehyde resin bond-lines at different thickness levels during the hot-pressing of laboratory oriented-strandboard (OSB) mats. The objective is to obtain a better understanding of the interaction of wood moisture content and resin curing on the dielectric properties of wood-composite mats. This would improve the interpretation of the DEA signals as they relate to monitoring and isolating the progress of resin cure during OSB hot-pressing.

BACKGROUND

OSB is a wood-based panel that is widely used in North American residential and commercial construction. Hot-pressing is generally regarded as a key OSB manufacturing step, governing production rates and panel quality. It involves the consolidation of wood flakes or strands, blended with 2% to 7% (by dry wood mass) phenol-formaldehyde or isocyanate-based thermosetting adhesive resin in a heated press. In efforts to optimize the hot-pressing, on-line DEA cure monitoring has gained interest as a method to detect the progression of resin cure in the hot-press and therefore signal the optimal press opening time.

Fringe-field DEA sensors involve applying a low voltage alternating electric field between neighboring electrodes to measure the impedance, thus yielding the localized dielectric relative permittivity and loss-factor of the material between or in the vicinity of the electrodes (Fig. 1). Both the relative permittivity and loss-factor have been related to the material's rheology and ionic conductivity as functions of the responses of charged molecular polar groups, ions, and dipoles to the electric field (Von Hippel 1954; Day 1989; Pethrick and Hayward 2002; Wang and Winistorfer 2003). For heat-cured thermosetting resins, permittivity and loss-factor signals generally show an initial increase, due to the application of heat and its effect on lowering the resin viscosity, followed by an asymptotic decay with the reduction in molecular mobility as the resin cures and solidifies.

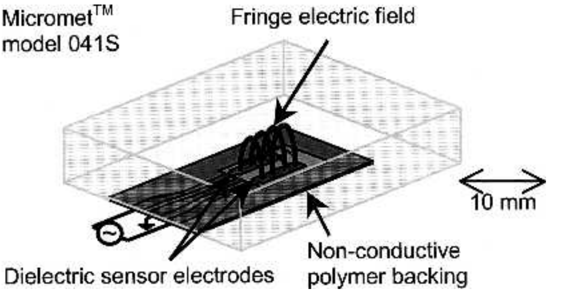

FIG. 1. Fringe-field dielectric ribbon sensor schematic. Fringe electric field through the mat, from a low voltage alternating current.

Applications of DEA in wood-composite hot-pressing have focused largely on non-invasive bulk DEA measurements through the whole mat thickness, with the electrodes situated on the top and bottom hot-press platen surfaces. The results have shown potential in relating gradient changes in the permittivity and loss-factor signals to the panel's dynamic consolidation, modulus of rupture (King and Rice 1996), and internal-bonding properties (Magill and Van Doren 2000; Congleton 2001; Wang and Winistorfer 2003).

Other studies have concentrated on localized fringe-field DEA monitoring of wood adhesives and the wood/adhesive interface under various resin loading, temperature, and moisture content conditions. Analogies between DEA, calorimetric, and spectroscopic techniques have been drawn, with interpretations of the DEA relationship to resin curing kinetics and morphology (Rials 1992; Ballerini 1994; Wolcott and Rials 1995a; Harper et al. 2001a and b). The hot-pressing and controlled temperature environment studies by Wolcott and Rials (1995a and 1995b) highlighted the strong effects of temperature and moisture changes on the dielectric properties of particleboard panels. Hotter temperatures were determined to correspond to stronger dielectric properties. Increases in resin content and moisture content interacted to produce greater DEA signals. Resin was concluded to be a dominant factor, either through the moisture present in certain resin formulations, or through the moisture consumption or formation during the resin polymerization.

EXPERIMENT PROCEDURE

Single-layer, randomly aligned 457- × 457-mm mats of 6.4% moisture content southern yellow pine (primarily loblolly pine, *Pinus taeda*) OSB strands were formed by hand. No resin or wax was added. The mats were hot-pressed at 180°C for 10 min, to an 11-mm thickness and 609 kg/m^3 density. In a subsequent experiment to eliminate the influence of moisture, the method was repeated using dried strands (average moisture content of 2.4% after cooling and mat forming).

During mat forming, two Micromet™ high-conductivity fringe-field DEA ribbon sensors (model 041S) were built into the mat (Figs. 1 and 2). The penetration of the 100 Hz alternating electric field was approximately 1.6 mm (equivalent to the separation of the electrodes; NETZSCH Instruments 2003). One sensor was randomly selected to be exposed to a thin layer of phenol-formaldehyde resin (commercial OSB core resin, 50% solid content, 21.3 Pa · s viscosity at 25°C), lightly brushed onto a wood strand over a 3-cm^2 area.

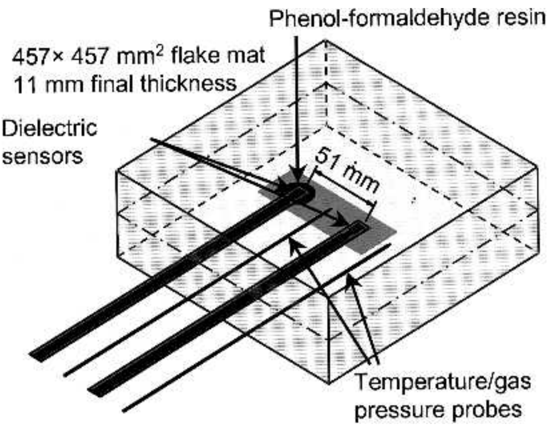

FIG. 2. Experiment setup. Two implanted dielectric analysis sensors (one exposed to a layer of phenol-formaldehyde resin), and two implanted temperature/gas pressure probes.

The sensor set-up was placed at the mat center; either on the bottom surface, or after 50%, 67%, or 83% of the mat's mass had been formed. It is assumed that these correspond to the sensors being at 0%, 50%, 67%, and 83% thickness levels from the bottom surface. Five mats were pressed for each thickness level in a fully randomized experimental order.

Two PressMAN™ thermocouple/gas pressure probes were also positioned about the sensors (Fig. 2). It is assumed that the average temperature and gas pressure readings from the two probes were indicative of the local conditions at the DEA sensor locations, thus allowing correlations of DEA permittivity and loss-factor versus localized temperature and gas pressure conditions.

RESULTS

Dielectric analysis signals without resin

Figure 3 shows the average DEA loss-factor and permittivity signals at different thickness levels for the control sensors with no exposure to resin. The first 60 s of the DEA signals were

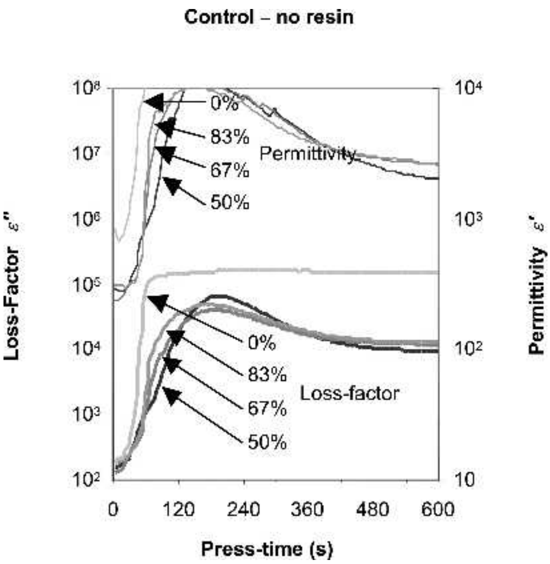

FIG. 3. Average control (no resin exposure) dielectric loss-factor (ε″, log-scale) and permittivity (ε′, log-scale) signals from different thickness levels (0% to 83% thickness).

dominated by press closure with increasing contact between the sensor and wood (Wang and Winistorfer 2003). The signals then displayed a steep initial increase followed by an asymptotic decay, with both DEA signals following similar trends and behavior, but with the permittivity signals capped at 10^4 due to DEA hardware limitations.

The initial trend of steeper DEA signals from the mat core to the surface reflects similar trends in temperature (Fig. 4). This may highlight a relation to heat and mass transfer through the mat while noting the effects of temperature and moisture on dielectric properties (Skaar 1988; Torgovnikov 1993; Siau 1995; Zhou and Avramidis 1999; Kabir et al. 2000 and 2001).

The temperature effect on the mat's dielectric properties is likely to be dominated by its influence on molecular bonding forces and the mobility of charged ions. The 100-Hz frequency of the oscillating electric field is relatively low compared to the relaxation times for dipoles to align or displace in response to the electrical excitation (typically 10^{-6} to 10^{-12} s; Skaar 1988; Torgovnikov 1993; Siau 1995). Thus electrical conductivity, as well as electrolytic and interfacial polarization, is a prominent component of the mat's dielectric properties. High temperatures would imply greater ion mobility, conductivity, and polarization, and therefore high permittivity and loss-factor signals.

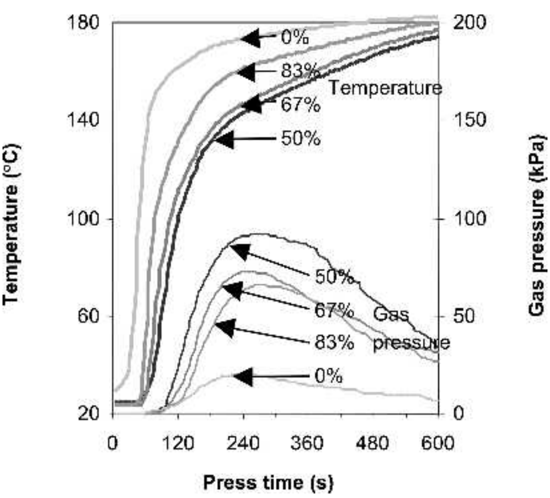

FIG. 4. Average internal temperature and gas pressure readings from different thickness levels.

The association of the DEA signals with conductivity and polarization implies a possible Arrhenius relationship, involving temperature (T) and the thermodynamic activation-energy (E_a) of the dissociation and diffusion of ionic charges (Skaar 1988; Siau 1995):

$$E_a \propto -\frac{\partial \log(\varepsilon' \text{ or } \varepsilon'')}{\partial(1/T)} \text{ or } \log(\varepsilon' \text{ or } \varepsilon'') \propto -1/T \quad (1)$$

Figure 5 plots the logarithm of the average loss-factor (ε'') and permittivity (ε') results versus the reciprocal of the average temperature results. In the roughly linear trend between 30° to 100°C, both the permittivity and loss-factor show approximate logarithmic relationships with the temperature reciprocal, with negative gradients that are largely independent of the thickness level. This leads to speculation that during the initial core heating stage of hot-pressing (50 to 150 s, see Fig. 4), DEA is indicative of increases in thermal energy, primarily of the bound-water,

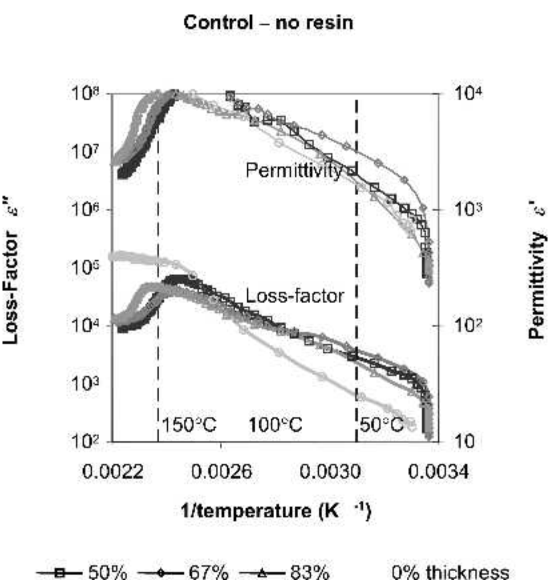

FIG. 5. Average loss-factor (ε'', log-scale) and permittivity (ε', log-scale) versus the reciprocal of the average temperature measurements through the mat thickness with no exposure to phenol-formaldehyde resin.

which would dominate the dielectric properties and ionic conductivity (Skaar 1988; Torgovnikov 1993).

Beyond 100°C, the permittivity and loss-factor signals begin to depart from the Arrhenius relationship (Eq. 1). This departure may highlight the increasing effect of moisture evaporation, thus leading to lower concentrations of dipolar molecules, and reductions in conductivity and dielectric properties. The maximum permittivity and loss-factor measurements would therefore indicate the point at which signal reductions due to moisture loss match the signal gains due to temperature and thermal energy increases. Beyond the maximum permittivity and loss-factor, the decaying signals could thus be more indicative of drying effects.

Although it is not apparent in the results, slight differences between the dielectric responses in the different layers would also be anticipated due to differences in local wood density and the vertical density profile. Also, at the higher temperatures, the wood begins to plasticize and surpass its glass transition temperature (Kelley et al. 1987; Lenth 1999; Lenth and Kamke 2001). These factors could have subtle superimposition effects on the dielectric properties measured.

Dielectric analysis signals with resin

From Fig. 6, resin had the effect of increasing the DEA signal amplitudes and initial gradients, as well as augmenting the variability in the signals between experiment replications. However, apart from at the surface, the DEA sensors exposed to resin showed similar signal trends and behavior as the sensors that were not exposed to resin.

The addition of resin essentially exposed the sensors to increased concentrations of dipoles and ions from the aqueous phenol-formaldehyde solution, thus leading to stronger permittivity and loss-factors. The steeper initial gradient could, therefore, have been a combination of the thermal activation-energy, resin cure byproduct, and decreasing resin viscosity that was superimposed on the underlying increase in dielectric properties from the heating of the wood and bound-water. The increased thermal activation-energy is also apparent in the steeper gradient of Fig. 7. The nonlinearity of the gradients between 30° to 100°C is probably indicative of the superimposition of the resin curing effects.

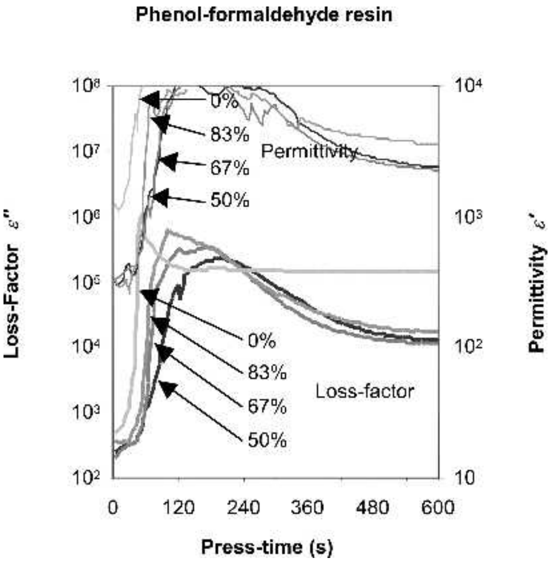

FIG. 6. Average phenol-formaldehyde dielectric loss-factor (ε'', log-scale) and permittivity (ε', log-scale) signals from different thickness levels.

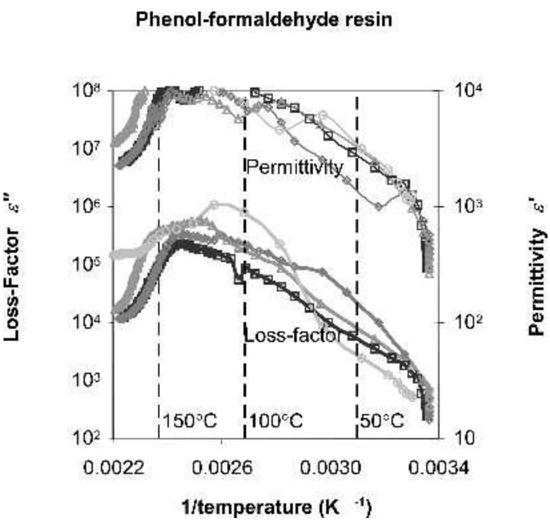

FIG. 7. Average phenol-formaldehyde resin loss-factor (ε'', log-scale) and permittivity (ε', log-scale) versus the reciprocal of the average temperature measurements through the mat thickness. See Fig. 5 for legend.

Considering this, the subsequent asymptotic decay response in Fig. 6 would thus be a combination of the increase in resin viscosity resulting in a reduction of dipole and ionic mobility and the evaporation of the moisture in the resin and wood. Because of the interdependence of these effects, the resin polymerization is difficult to isolate from the moisture effects.

A pure resin effect was apparent only in the 0% thickness loss-factor signal (Fig. 6). The resin signal had a much steeper initial gradient and a peak loss-factor value of 1.1×10^6 that was not present in the resinless signal (Fig. 3). The behavior of the resinless loss-factor reflects the rapid heating and drying of surface flakes. By the time the hot-press closes, the surface flakes are essentially dry, thus yielding signals that are free of the signal decline that would be induced by subsequent moisture loss. The sudden peak and decay in the resin loss-factor are therefore indicative of the resin behavior.

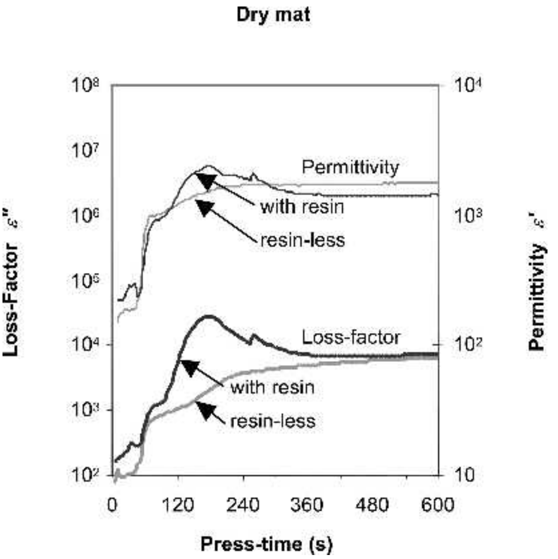

FIG. 8. Average resinless and phenol-formaldehyde dielectric loss-factor (ε″, log-scale) and permittivity (ε′, log-scale) signals at the 50% thickness level for dry mats hot-pressed at 2.4% moisture content.

Dielectric analysis signals from a dry mat

For the oven-dried strands (2.4% moisture content), the DEA signals can be considered as relatively free of the effects of moisture compared to the previous 6.4% moisture content results. This was reinforced by the detection of only small increases in internal gas pressure of less than 4 kPa arising from moisture evaporation during hot-pressing.

In these dry mats, the effects of resin are dominant (Fig. 8) with a marked increase and peak over the resinless DEA signals. Also, the relatively low signal amplitudes (c.f. Fig. 3 and Fig. 6) demonstrate the moisture content susceptibility of the dielectric properties, and the resinless signals lacked the peaks and asymptotic decay seen in Fig. 3.

The subsequent asymptotic decay and approach of the resin DEA signals towards the resinless signals in the late stages of hot-pressing may indicate an association with the resin curing. As seen in the previous 0% thickness level results of Fig. 3 and Fig. 6, in the later stages the resin would have cured and the mat dried, thus approaching the conditions of a dry and resinless mat. However, the similar shape and form of the resin DEA signals of Fig. 8 and the resinless signals of Fig. 3 (c.f. 50% thickness level) could imply a dominating moisture loss effect. Thus, the asymptotic decay of the resin signal of Fig. 8 would be associated with the drying and evaporation of the resin moisture as well as the resin polymerization and cure. The resin polymerization effect is therefore difficult to isolate, but inherently the rates of resin polymerization and moisture evaporation should approximately coincide.

DISCUSSION

In summary, our results reinforce many of the conclusions of Wolcott and Rials (1995a, 1995b). Their observations of particleboard also demonstrated DEA signals to be driven by temperature, moisture flow, and interactions between resin polymerization and the moisture content of the wood and resin formulation. Our experiments and thermodynamic analysis extend their work to OSB, while elaborating on the possible fundamental influence of heat and moisture transfer through the Arrhenius relationship (Eq. 1) of temperature on dielectric properties.

From our findings, during the initial stages of hot-pressing with temperatures increasing to 100°C, DEA signals appear to be driven largely by increases in thermal energy of the water in the resin and wood system. Thus, as also seen by Wolcott and Rials (1995a, 1995b), the initial DEA signal increase is indicative of temperature and heating, with steeper gradients implying greater heat transfer.

Beyond 100°C, the DEA signals begin to depart from the Arrhenius thermodynamic relationship (Eq. 1), highlighting the increasing effect of moisture evaporation combined with resin polymerization. Therefore, the DEA signal peak, which has often been interpreted as an indication of the onset of cure (Wolcott and Rials 1995b; Magill and Van Doren 2000), indicates the balance point at which the signal gain due to temperature and thermal energy increases and equals the signal reduction due to moisture loss and resin cure.

CONCLUSIONS

Local dielectric properties were determined to vary through the mat thickness in relation to the internal mat environment during hot-pressing. Initial DEA cure monitoring signal increases reflected similar trends as seen in internal temperature increases. In the initial core heating stages of hot-pressing, temperature correlated with dielectric properties via a thermodynamic energy relationship (Eq. 1), likely associated with the increasing mobility and diffusion of water molecules with increasing temperature.

Dielectric properties were determined to be susceptible to variations in moisture content, with low moisture content mats having relatively low dielectric properties and DEA signal amplitudes. There was also some indication that the decaying DEA signal in the later stages of hot-pressing was related to moisture evaporation.

Phenol-formaldehyde resin increased the dielectric properties of the mat and DEA signal amplitudes, likely due to the increase in the concentration of ions and dipoles from the aqueous resin solution. The isolated resin effect on DEA showed behavior consistent with moisture loss, and is thus likely to be indicative of the evaporation of the resin moisture combined with effects of resin polymerization and cure.

Regarding heat and mass transfer effects and the general interpretation of DEA cure monitoring signals during OSB hot-pressing; our conclusions imply that the maximum signal reading indicates the point at which the increase in dielectric properties due to heating is equivalent to the decrease due to moisture evaporation and resin cure. Steep initial DEA signals imply rapid heating and heat transfer through the mat. A rapid subsequent signal decay would imply rapid mat drying and resin cure.

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REFERENCES

BALLERINI, A. A. 1994. Cure of aqueous phenol-formaldehyde resin at elevated water vapor pressures. Ph.D. thesis, Department of Wood Science and Forest Products, Virginia Polytechnic Institute and State University, Blacksburg, VA. 141 pp.

CONGLETON, T. G. 2001. Dielectric cure monitoring of composite panels during hot pressing: A fundamental understanding. M.Sc. thesis, Department of Forest Products, Oregon State University, Corvallis, OR. 97 pp.

DAY, D. R. 1989. Dielectric properties of polymeric materials. Micromet Instruments Inc., Cambridge, MA. 51 pp.

HARPER, D. P., M. P. WOLCOTT, AND T. G. RIALS. 2001a. Evaluating cure of a pMDI-wood bondline using spectroscopic, calorimetric and mechanical methods. J. Adhesion 76(1):55–74.

———, ———, AND ———. 2001b. Evaluation of the cure kinetics of the wood/pMDI bondline. Int. J. Adhesion Adhesives 21(2):137–144.

KABIR, M. F., W. M. DAUD, K. B. KHALID, AND A. H. A.

SIDEK. 2000. Equivalent circuit modeling of the dielectric properties of rubber wood at low frequency. Wood Fiber Sci. 32(4):450–457.

———, ———, ———, AND ———. 2001. Temperature dependence of the dielectric properties of rubber wood. Wood Fiber Sci. 33(2):233–238.

KELLEY, S. S., T. G. RIALS, AND W. G. GLASSER. 1987. Relaxation behaviour of the amorphous components of wood. J. Mater. Sci. 22:617–624.

KING, R. J., AND R. W. RICE. 1996. Monitoring resin cure of medium density fiberboard using dielectric sensors. Pages 601–605 *in* M. F. Iskander, J. O. Kiggans, and J. Ch. Bolomey, eds. Proc. Microwave Processing of Materials V. Material Research Society Symposium, Vol. 430, Materials Research Society, Warrendale, PA. 662 pp.

LENTH, C. A. 1999. Wood materials behavior in severe environments. Ph.D. thesis, Department of Wood Science and Forest Products, Virginia Polytechnic Institute and State University, Blacksburg, VA. 122 pp.

———, AND F. A. KAMKE. 2001. Moisture dependent softening behavior of wood. Wood Fiber Sci. 33(3):492–507.

MAGILL, R., AND J. VAN DOREN. 2000. Intelligent monitoring and control of wood panel press cuing using in-situ impedance sensors. Pages 89–96 *in* Proc. 34th International Particleboard/Composite Materials Symposium, Washington State University, Pullman, WA.

NETZSCH INSTRUMENTS INC. 2003. "Micromet series, dielectric sensors," NETZSCH-Geraetebau GmbH, http://www.micromet.com/holometrix/m_sensors.asp (31 May 2003).

PETHRICK, R. A., AND D. HAYWARD. 2002. Real time dielectric relaxation studies of dynamic polymeric systems. Prog. Polym. Sci. 27:1983–2017.

RIALS, T. G. 1992. Cure analysis of phenol-formaldehyde resins by microdielectric spectroscopy. Pages 282–294 *in* W. G. Glasser and H. Hatakeyama, eds. Viscoelasticity of biomaterials. ACS Symposium Series, Vol. 489, American Chemical Society, Washington, DC. 406 pp.

SIAU, J. F. 1995. Wood: Influence of moisture on physical properties. Department of Wood Science and Forest Products, Virginia Polytechnic Institute and State University, Blacksburg, VA. 227 pp.

SKAAR, C. 1988. Wood water relations. Springer-Verlag, New York, NY. 283 pp.

TORGOVNIKOV, G. I. 1993. Dielectric properties of wood and wood-based materials. Springer-Verlag, New York, NY. 196 pp.

VON HIPPEL, A. R. ed. 1954. Dielectric materials and applications; papers by twenty-two contributors. Massachusetts Institute of Technology, Cambridge, MA. 438 pp.

WANG, S., AND P. M. WINISTORFER. 2003. Monitoring resin cure during particleboard manufacture using a dielectric system. Wood Fiber Sci. 35(4):532–539.

WOLCOTT, M. P., AND T. G. RIALS. 1995a. In-situ cure monitoring of isocyanate adhesives using microdielectric analysis. Forest Prod. J. 45(2):72–77.

——, AND ———. 1995b. In-situ cure monitoring of adhesives for wood-based composites. Pages 185–193 *in* Proc. 29th International Particleboard/Composite Materials Symposium, Washington State University, Pullman, WA.

ZHOU, B., AND S. AVRAMIDIS. 1999. On the loss-factor of wood during radio frequency heating. Wood Sci. Technol. 33:299–310.