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Intelligent Processing of PMR-15

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INTELLIGENT PROCESSING OF PMR-15

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Sean Michael Hart

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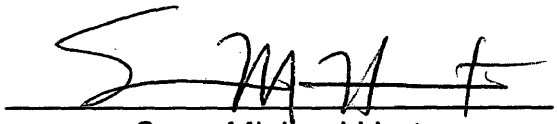

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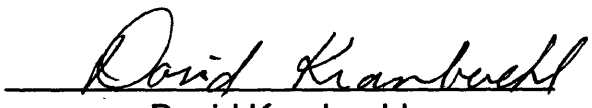

David Kranbuehl

Gary Rice

William Starnes

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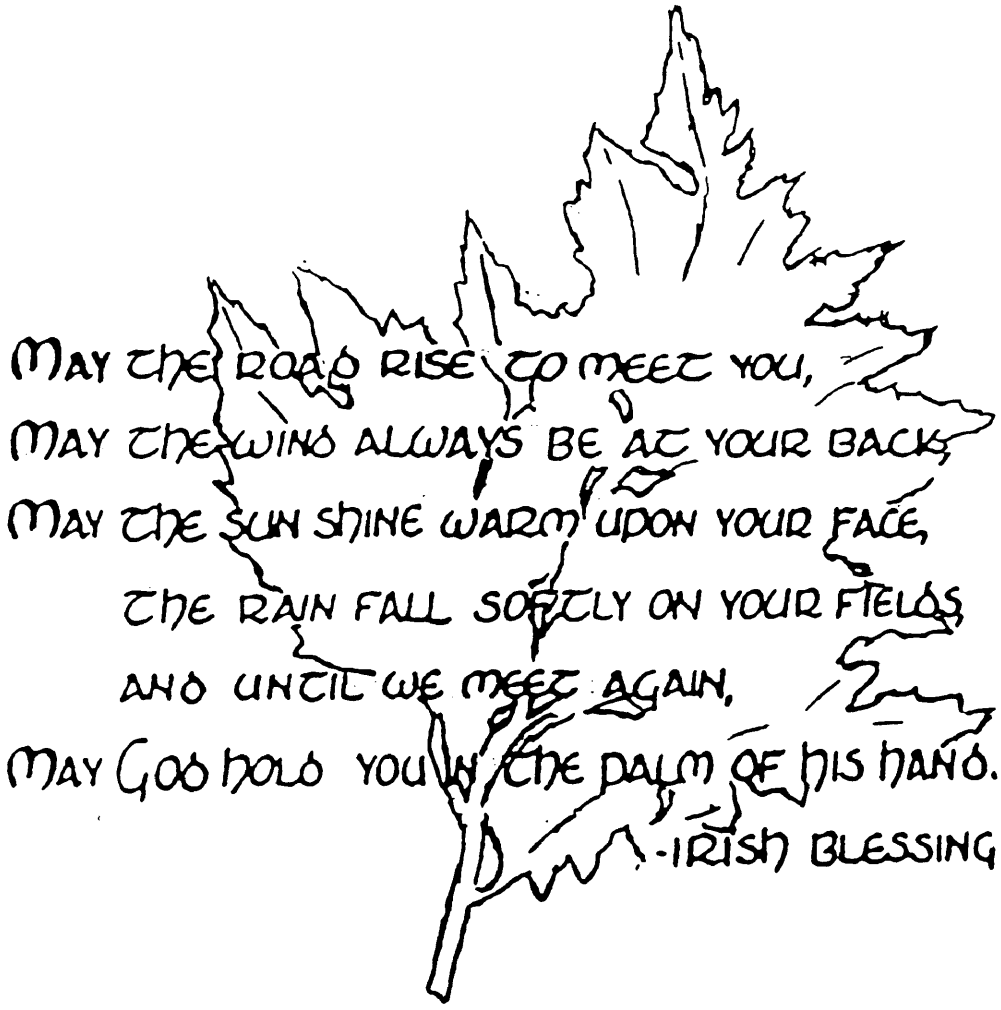
DEDICATION

This work is dedicated to my parents, Grace and Jim Hart. Their love and support have helped me to reach this point in my life, and they will continue to provide strength as tomorrow becomes today. A love of life and of the joy of discovery have been given to me; may God grant me the opportunity and the purity of heart to share these with others.

To you who now study this work, welcome to my small part of the world of science and learning. I hope that it provides you with the knowledge for which you seek. If it does not, I wish you luck in your search.



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Intelligent Processing of PMR-15

ABSTRACT

An intelligent, automated composite cure control system has been developed and used to control the cure of PMR-15/graphite prepreg in the autoclave and the thermal press. The expert system uses Frequency Dependent Electromagnetic Sensing (FDEMS), results from the Loos processing model for PMR-15, and the Qualitative Process Automation Language (QPAL) developed for the United States Air Force by Frances Abrams, Wright-Patterson Air Force Base, Ohio, USA in conjunction with the Universal Technology Corporation, Dayton, Ohio, USA. Pmr-15 polyimide resin is one of the leading candidates for aerospace applications involving long-term service at *circa* 300°C use temperatures.

The Loos processing model for PMR-15 has been used to predict and to optimize the cure process. Included are the extent of reaction, resin flow and part consolidation. The FDEMS sensing technique monitors in situ the removal of solvent, changes in viscosity, reaction advancement and completion of cure in the tool continuously throughout the cure process. The FDEMS sensor information is compared in real time with the optimum processing goals determined by the Loos processing model and user definition.

The QPAL component of the intelligent cure control system provides a language for making decisions regarding the continuation of the cure process. It allows comparisons between the FDEMS sensor data and the processing goals to be broken down into a series of discrete, easily understood steps. By monitoring the achievement or the avoidance of these milestones in the PMR-15 cure process, the expert system discussed here has been used to control and to optimize the cure process of fresh and aged PMR-15/graphite prepreg.

INTELLIGENT PROCESSING OF PMR-15

INTRODUCTION

The ability to fabricate high quality composite structures using a variety of thermosets and thermoplastics provides a continuing challenge to the aerospace industry. Consistently manufacturing good composite structures with different resin systems at best currently requires many trials with a minimum of errors. The use of intelligent, closed loop processing provides the scientist and the engineer with a cost-effective method for consistently producing high quality, void-free composite structures for use in a variety of applications. By monitoring and evaluating in situ the changing state of the curing matrix resin, real time adjustments may be made to the remainder of the fabrication process to ensure successful part production. Thus variations in resin batch, moisture uptake, out time in part preparation and incidental or intentional advancements in the degree of cure may be taken into account.

CHAPTER 1

EXPERT SYSTEMS

Intelligent processing of composites involves the use of an empirically developed expert system coupled with a real time method for determining the changing state of a curing composite structure. Expert control systems are designed to acquire knowledge, to inform about the current reality, and to make decisions that will optimize the ability to achieve a desired outcome. As such they are systematic, thorough users of the collective expertise of the human experts who constructed them. The three major components of an expert system are:

(1) the appropriate human-machine interface,
(2) the problem-solving and inference engine, and
(3) the knowledge base of facts and heuristics.[1]

A fourth important feature of expert systems is that they are *data*, not procedure, *driven*.[2] In other words, rather than following a preset procedural path to a final point, the intelligent expert system will instead *choose* the best possible path available to achieve the desired goal. In composite manufacturing this focus on decision making based on real time data rather than a preset recipe for cure allows the intelligent, closed loop cure control system to minimize part loss due to variations in cure as a function of tool, temperature, pressure, environmental conditions and resin/prepreg precure state.

Let us consider in more detail the major components of an expert system. The first is the appropriate *human-machine interface*. Ideally, this interface would allow the human expert to pass the complete breadth of his or her knowledge to the expert system as well as the ability to reason and to adapt to unforeseen circumstances.[1-3] In reality, however, the expert system can only adapt to variations within the set of limitations that its creator has used to define its 'thought' processes. This interface typically consists of a computer keyboard, a monitor screen and an auxiliary printing device which allow the human expert to interact with his or her mechanical computer counterpart. It is here that the intelligent machine expert is created and enhanced, and it is also from here that the human expert receives feedback from his or her creation.

The second component, the *problem-solving and inference engine*, is the specific structure and its related language and symbolism created in the programming environment of choice.[3] This is probably the most

important component of the functional intelligent control system. Simply put, without a well-written, comprehensive and flexible means of control, the expert system will not be able to replace its human creator in everyday scenarios. The expert system is constructed to acquire new knowledge in the form of real time data and to make decisions based on those inputs via the *knowledge base of facts and heuristics* which comprises the third major component of its logical structure. The portion of the knowledge base that is previously defined expert knowledge allows the intelligent control system to understand current data inputs and to make correct decisions regarding the remainder of the composite cure process based on a series of heuristics, or rules, designed to achieve a certain goal.

Finally, the expert system must have an automated means for acquiring real time data concerning the current state of the curing composite material. Two commonly used methods for monitoring the progress of a reaction rate are:

(1) the temperature measurement of the macroscopic changes in temperature as a function of resin reaction and tool cure temperature profile, and

(2) the measurement of the microscopic changes of the state of the reacting system based on the changing dielectric nature of the curing matrix resin.

A schematic representation of the intelligent, closed loop expert control process is shown in Figure 1.1.

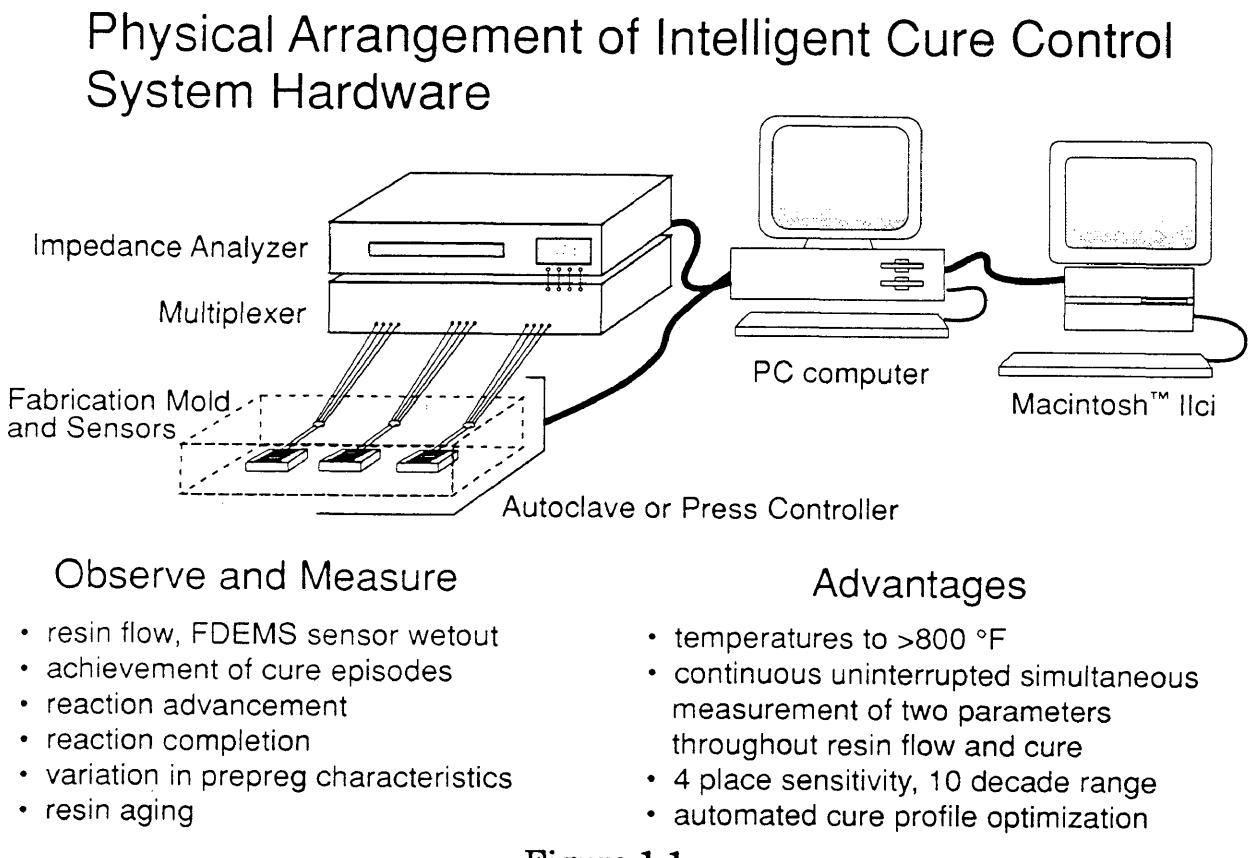

Figure 1.1

Possible Methodologies

The simplest method of controlling the cure of a composite part is to follow a procedure-driven manufacturer recommended cure cycle for the matrix resin of interest. This procedure makes adjustments to the curing environment to predetermined ideal settings regardless of the actual state of the curing material. Typical manufacturer recommended cure cycles are very conservative and require longer times to reach completion than those provided by optimized cure schedules.

A second means of determining the cure cycle for a given composite part is through the use of trial and error fabrication runs of the part of interest. However, trial and error determinations of an optimum cure cycle are extremely difficult to reproduce due to the variations in materials and tooling/curing environment response which tend to make virtually every run unique.[4] Additional problems with the trial and error approach are:

(1) that they cannot necessarily be generalized to parts that are of different thicknesses, geometries, and materials, and

(2) that they are expensive and time consuming.[5]

Several workers have developed expert control systems for polymer processing. These systems are based on the use of various methods for continuous, in situ measurement of the cure process. Among these methods, the use of temperature and dielectric measurements has proven successful. Gluyas[6], Kranbuehl[7-10] and others[11,12] have shown that the ionic conductivity or mobility of a resin can be related to its macroscopic viscosity and degree of cure. Additionally, the literature contains many references which discuss the relationship of temperature to reaction advancement. Thus it is not surprising that these techniques have moved to the forefront of intelligent cure control efforts.

Kranbuehl, Loos and colleagues have demonstrated closed loop control of the autoclave cure process. Their approach uses in situ, continuous dielectric sensing techniques and cure process modelling for closed loop control of the autoclave. This work involves the use of dielectric sensors to monitor the variations in reaction advancement as a function of changing matrix resin viscosity within a thick graphite-epoxy laminate. The experimental viscosities were compared to ideal values generated by the Loos-Springer cure process model. In the subsequent autoclave run, the outputs from four dielectric sensors placed strategically within the thick laminate were used to control the autoclave environment, resulting in an optimized cure cycle for the laminate.[13-15] One benefit of this approach is that it compensates for the occurrence of localized exotherms within the thick laminate. This system is limited, however, because of its resin specific nature and its exclusion of other possibly significant milestones in the matrix resin cure process.

Ciriscioli and Springer have also developed a smart cure process for the autoclave. Their system selects and controls in real time the cure conditions (that is, the autoclave temperature and pressure) during the cure of thermoset matrix composites. This system monitors the following parameters during cure:

(1) the autoclave temperature,

(2) the surface temperatures of the composite,

(3) the midpoint temperature of the composite,

(4) the dielectric properties at one point inside the composite,

(5) the thickness of the composite, and

(6) the autoclave pressure.[16]

Although this particular smart cure control system requires no prior knowledge of the material properties of the composite being fabricated, it does have its limitations. Experience has shown that simply knowing the temperature of a material at a given time provides only a gross indication of the actual state of the curing material. Additionally, unless the expert system is capable of monitoring virtually every square inch of the composite material being cured, localized exotherms which would damage the part are likely to be discovered too late. Also, the Ciriscioli smart cure process is only suitable for composites which are made with thermosetting matrices; no provision is made for monitoring the formation of parts from thermoplastic or other matrix resin materials.

One feature of the Ciriscioli smart autoclave cure process is its limited use of dielectric measurements of the curing matrix resin. This expert system also uses knowledge from one point inside the composite to control the compaction process as well as to determine the end of cure.[16]

Abrams and coworkers have developed a third approach to the intelligent cure of composite materials. It differs from the approach used by Ciriscioli in that it uses a generalized software shell as the programming and decision making environment for controlling the cure process. This Qualitative Process Automation Language (QPAL) provides the expert system with a set of actor definitions (*i.e.*, heater, sensor), a library of available methods (*i.e.*, IF ... THEN rules), and a library of behavioral actions (*i.e.*, turning off or on a heater, reading a sensor) which allow for the control of any properly interfaced tooling curing device.[4]

The second component of the QPAL system describes the process of interest. This descriptive knowledge base may or may not be material specific depending upon the desired outcome. This description of the cure process does not include step-by-step instructions. Rather, it includes only a list of possible process episodes and the goals and history of the current process. Thus the QPAL system offers a declarative method for describing a process which differs from the traditional procedural approach in that the order of the process steps are not specified.[4] Another advantage of the Abrams QPAL system is the portability of a given knowledge base (with minor modifications) to other QPAL-based expert systems using the same general sensor control format. Finally, QPAL has the capability of testing a newly modified knowledge base either internally through a user-defined trace or externally from previously acquired real data without having to run a series of time consuming, expensive real composite cures.

Like the Ciriscioli smart autoclave cure system, the Abrams expert

system relies heavily on temperature data. As previously mentioned, changes in temperature provide only macroscopic indications regarding the state of the curing resin and often occur too late for decisions based on them being able to prevent destructive exotherms or other thermally induced damage to the composite part. The Abrams QPAL autoclave control system also used dielectric sensors. However, their use is limited to monitoring the buildup in matrix modulus and to determining the ability of the matrix to flow for purposes of compaction.[4]

A fourth expert system for composite manufacture has been developed by Johnson and Roberts at The General Dynamics Corporation/Pomona Division for a material based on the Monsanto Skybond 703 polyimide resin. In contrast to the two previously described intelligent cure control systems, the Johnson/Roberts closed loop control system focuses on the changing dielectric properties of the curing resin. By monitoring these changes as well as changes in part and tool temperatures, this expert system looks for the achievement of precisely three user-defined critical windows or stages in the cure of said composite part based on the changes in the dielectric signal of the curing matrix resin.[17]

The Johnson/Roberts expert systems' use of in situ dielectric measurements of the curing matrix resin allows it to probe the microscopic state of the resin system. It is limited, however, by the fact that it constrains itself to a previously determined optimum cure cycle requiring the occurrence of three critical stages in the cure process. While this closed loop control system may be adjusted for different resin systems, its controlling logic does not allow for processes/resins that require more or fewer than three critical windows in the control process. Additionally, because it has been developed for a specific manufacturing process, adaptation to different fabrication requirements may prove difficult and time consuming.

A fifth expert system has been developed by Kingsley, Williamson and others in our laboratory at William & Mary for control of the resin transfer molding (RTM) process in the thermal press. It uses in situ dielectric and temperature measurements to monitor and control the cure of both the liquid injection and the film infusion RTM processes for several commercially available epoxy resin systems. These measurements are made continuously throughout the fabrication process and determine the completion of various stages in the cure process. For example, the dielectric sensor is used to determine full wet out of the dry fiber preform and to determine the end of cure based on the changing dielectric signal

during the final cure hold.[18,19] One major drawback to this system is the complexity of its controlling logic. A second is that transfer from one curing environment to another requires the user to rewrite portions of the controlling software in order to correctly interface with the new oven, press or autoclave. The above descriptions do not provide an exhaustive history of the field of smart composite cure processes. They do, however, serve as a good encapsulation of the major developmental thrusts being tried today.

The final method of intelligently controlling the cure of a composite part to be discussed is the focus of this paper. Our intelligent, automated sensor-model closed loop control system uses the Abrams QPAL shell, dielectric measurements similar to those found in both the Johnson/Roberts and Kingsley/Williamson expert systems, and generalized temperature measurements common to all of the above expert systems. Its major departure from these methods lies in its coupling of the object oriented QPAL structure with the acquisition of continuous, in situ Frequency Dependent Electromagnetic Sensing measurements of the cure process. In doing so it monitors the microscopic state of the matrix resin and uses this information to evaluate the process history and to make decisions regarding the remainder of the cure process. Thus each individual cure is optimized.

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CHAPTER 2

FREQUENCY DEPENDENT ELECTROMAGNETIC SENSING

Frequency Dependent Electromagnetic Sensing (FDEMS) provides a convenient, non-destructive means of monitoring the cure process of polymer systems. Impedance measurements, made over a wide frequency range in the hertz to the megahertz region, are an effective means of characterizing a wide variety of resin systems.[1]

Polarization

Polarization can be conceptualized as the displacement of charges over a relatively short distance within a molecule. Although a polar molecule is electrically neutral overall, it possesses regions of permanently localized positive and negative charges which are separated by a distance of molecular dimensions. A dipole of charges $+q$ and $-q$ separated by a distance d has a dipole moment, μ, where

$$\mu = q * d \qquad [1]$$

The application of an electric field induces an intramolecular charge shift, resulting in the polarization of the molecule. In non-polar molecules, this polarizability results from only two molecular responses: *Electronic polarization* and *Atomic polarization.* Electronic polarization (P_E) results from the shift of electron clouds with respect to the positive charge centers of the molecule. Atomic polarization (P_A) results from the shift of atomic nuclei within the polymer molecule, as exemplified by carbon-hydrogen bond stretching in response to an applied electric field. Polar molecules, in addition to electronic and atomic polarizability, also possess a dipole polarization (P_O) which results from the orientation of permanent dipoles with the applied electric field. Thus the total polarization of the molecule may be defined:

$$P_T = P_E + P_A + P_O \qquad [2]$$

P_E and P_A result from the relative displacements of positive and negative charges within the molecule, and are referred to as *distortion polarizations.*[2,3]

The amount of localized charge built up in response to the applied electric potential is time dependent. As the field is suddenly changed, the charge surges toward a new equilibrium distribution proportional to the new electric field. This rate of change in equilibrium depends upon the rates of change of the electronic, atomic and dipole polarizations. For P_E and P_A, this corresponds to the electronic and vibrational modes of the molecule and is very rapid. The reorientation of the permanent dipoles, on the other hand, is delayed by their external environments; thus an equilibrium with the applied field is reached in a relatively long time.

Dielectric Measurements

The examination of the properties of a simple parallel-plate condenser provides an easily understood model of how impedance measurements are made. Consider a parallel-plate capacitor in a vacuum, with the plates of large area separated by a small gap so that edge effects are negligible.[4] When a potential difference V is applied across the condenser plates they acquire charges $+Q$, $-Q$ per unit area. The capacitance of a condenser measures its ability to store charge and is defined:

$$C_0 = Q/V \qquad [3]$$

If the gap separating the plates is filled with a dielectric material, the surfaces of the dielectric material will experience charge localizations $-P$, $+P$ per unit area. The condenser can now hold a charge $(Q+P)$ (see Figure 2.1) at an applied potential V.

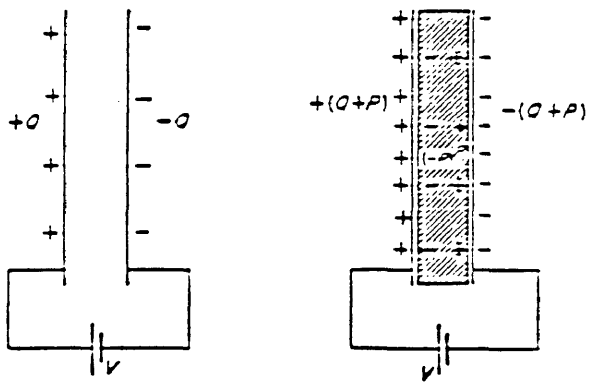

Figure 2.1

Thus the capacitance per unit area has increased to:

$$C = (Q+P)/V \qquad [4]$$

The relative permittivity of the material is defined as the ratio of the capacitance of the condenser containing the dielectric material to that of the empty condenser under vacuum:

$$\mathcal{E}_0 = C/C_0 = (Q+P)/Q \qquad [5]$$

This permittivity is related to the total polarization and can be expressed in terms of the polarization P produced by an applied electric field E:

$$\mathcal{E}_0 = 1 + 4\pi P/\mathcal{E}E \qquad [6]$$

where $\mathcal{E}$ is a constant which depends on units.[5]

Polarizability

The three polarization mechanisms can also be expressed in terms of a molecular physical quantity called the *polarizability,* α. Polarizability is defined as the average molecular polarized dipole moment induced by an

electric field of unit strength. Under the action of the local electric field of strength E', the sum of the individual molecular dipole moments produces an average polarized dipole moment μ. The field E' will normally differ from the applied electric field E because of the polarization of the surrounding dielectric medium. Thus, for a general case, μ is proportional to E', or

$$\mu = \alpha_T E' \qquad [7]$$

where the polarizability constant α_T is the total polarizability of the molecule and is given by:

$$\alpha_T = \alpha_E + \alpha_A + \alpha_O \qquad [8]$$

α_E, α_A and α_O are the *electronic*, the *atomic*, and the *dipolar* contributions to the polarizability.

If a unit volume of a polymer contains N molecules, then after polarization the total polarized dipole moment per unit volume is $N^*\mu$, which is equal to the polarized charge density P. Thus,

$$P = N\mu \qquad [9]$$

and substitution from Equation [7] gives:

$$P = N\mu_T E' \qquad [10]$$

This is the general expression of the value of the polarization and it links the macroscopically measurable dielectric constant to three molecular parameters: the number N of contributing elementary entities per unit volume, their polarizability α, and the local electric field strength E'.[2] After substituting P from Equation [10] back into Equation [6], the relative permittivity becomes

$$\mathcal{E}_0 = 1 + (4\pi N\alpha_T E')/(\mathcal{E}E) \qquad [11]$$

Each component of the total polarizability coefficient is a function of the frequency of the applied electric field. Suppose an alternating electric field is applied to the parallel-plate condenser filled with a polar material. As the field shifts, the orientation of the polar molecules must change. When the frequency of the applied electric field is sufficiently low, all three

components of the polarization reach the values that they would obtain under the influence of a steady field (Figure 2.2).[6]

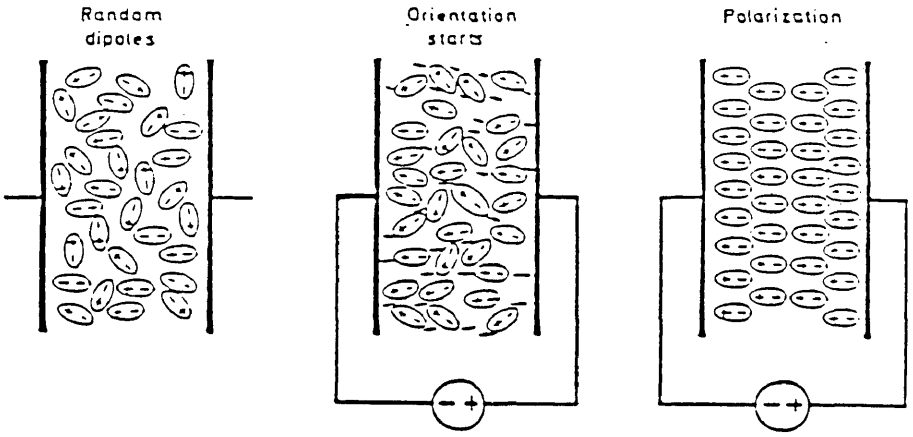

Figure 2.2

As the frequency of the applied field increases, the total polarization can no longer obtain its static field value. The first component of the total poalrizability affected is α_O. Thus at relatively higher frequencies the total polarization becomes:

$$\alpha_T = \alpha_E + \alpha_A \qquad [12]$$

The frequency at which the loss in orientation polarization occurs varies from very low frequencies on the order of 100 Hz for polymer chains, to 10^{10} to 10^{12} Hz for small molecules. The contributions of α_E and α_A remain unchanged at frequencies where dielectric relaxation (*i.e.,* dipolar resonance with the applied field) takes place because the distortion polarization of a molecule equilibrates with the applied electric field much sooner than does α_O. At frequencies comparable to the natural vibrational frequencies of the atoms in the molecule, α_A fails to reach its static field equilibrium value and further dispersion regions in signal will appear in the infrared region of the electromagnetic spectrum. The loss of the electronic polarization α_E occurs at even higher frequencies which correspond to the electronic transitions between different energy levels in the atom (the visible to X-ray region of the electromagnetic spectrum).[4] Figure 2.3 shows the region of the electromagnetic spectrum used in FDEMS characterization.

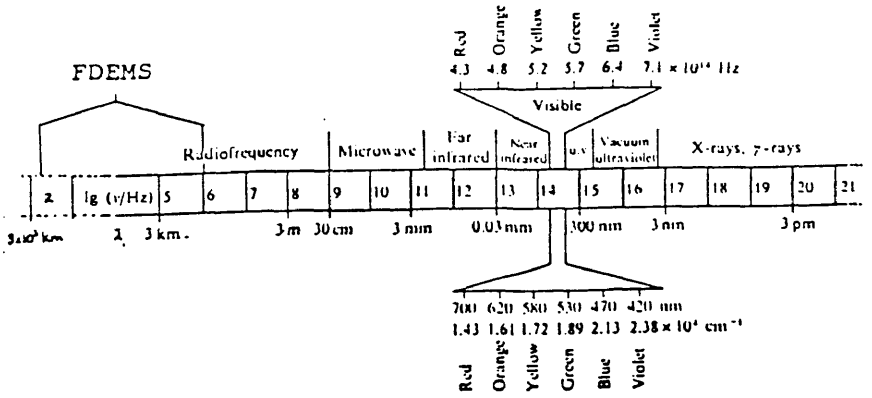

Figure 2.3

Experimental Theory

In making dielectric measurements, the material to be characterized is placed between the plates of a capacitor. The *impedance Z* at a frequency *f*, of a material is given by:

$$Z^{-1} = G + i2\pi fC \qquad [13]$$

Measurements of the *capacitance C* and the *conductance G*, both dependent on sample geometry, are used to calculate the intensive geometry independent complex permittivity:

$$\mathcal{E}^* = \mathcal{E}' + i\mathcal{E}'' \qquad [14]$$

Using the electronic relationships

$$I = i2\pi fCV \qquad [15]$$

and

$$C = \mathcal{E}^* C_0 \qquad [16]$$

the following derivations define the behavior of the dielectric between the plates of the parallel-plate capacitor in terms of a resistor and a capacitor in parallel[5] (Figure 2.4).

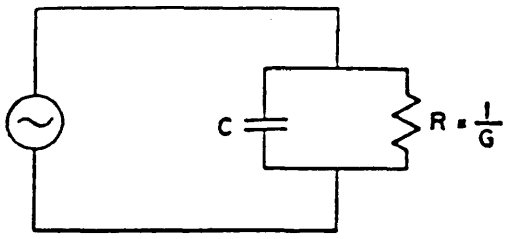

Figure 2.4

Substituting Equation [16] into Equation [15],

$$I = i2\pi f \mathcal{E} C_0$$

$$I = i2\pi f(\mathcal{E}' + i\mathcal{E}'')C_0 V$$

$$I = 2\pi f C_0(\mathcal{E}'' + i\mathcal{E}')V$$

$$I = 2\pi f C_0 \mathcal{E}'' V + i2\pi f C_0 \mathcal{E}' V \qquad [17]$$

The real portion is in-phase and the imaginary portion is out-of-phase with the applied voltage. Substituting into Ohm's Law, $V=IZ$, where Z is the impedance, or total resistance, of the circuit, Equation [17] yields:

$$Z^{-1}=(2\pi f C_0 \mathcal{E}'' V + i2\pi f C_0 \mathcal{E}' V) / V$$

$$Z^{-1}=2\pi f C_0 \mathcal{E}'' + i2\pi f C_0 \mathcal{E}' \qquad [18]$$

Setting Equations [13] and [18] equal gives:

$$2\pi f C_0 \mathcal{E}'' + i2\pi f C_0 \mathcal{E}' = G + i2\pi f C$$

$$0 = 2\pi f C_0 \mathcal{E}'' - G + i2\pi f C_0 \mathcal{E}' - i2\pi f C \qquad [19]$$

Separating the real and the imaginary parts of Equation [19] yields the dielectric components $\mathcal{E}'$ and $\mathcal{E}''$.

$$2\pi f C_0 \varepsilon'' - G = 0 \qquad i2\pi f C_0 \varepsilon' - i2\pi f C = 0$$
$$2\pi f C_0 \varepsilon'' = G \qquad i2\pi f C_0 \varepsilon' = i2\pi f C$$

$$\varepsilon'' = G/(C_0 2\pi f) \qquad \varepsilon' = C/C_0 \qquad [20]$$

ε' is the previously described dielectric permittivity and ε'' is the dielectric loss factor resulting from the energy loss associated with the highly time-dependent orientation polarization and bulk charge conduction.[9]

The real and imaginary components of ε^* have both a *dipolar* and an *ionic* component.

$$\varepsilon' = \varepsilon'_D + \varepsilon'_I \qquad [21]$$

$$\varepsilon'' = \varepsilon''_D + \varepsilon''_I$$

The dipolar portion results from the rotational diffusion of bound charges and molecular dipole moments. The frequency dependence of the dipolar component may be represented by the *Cole-Davidson function*:

$$\varepsilon^*_D = (\varepsilon_r - \varepsilon_u)/(1+i2\pi f\tau)^{\beta} + \varepsilon_u \qquad [22]$$

where ε_r and ε_u are the limiting low and high frequency values of ε^*_D, τ is the characteristic relaxation time, and β is the Cole-Davidson distribution parameter ($0<\beta<1$), which measures the distribution in relaxation times. It is this dipolar component which dominates the dielectric signal at high frequencies and in highly viscous media.[1]

The ionic component often dominates ε^* at low frequencies, low viscosities and/or elevated temperatures. The presence of mobile ions gives rise to localized layers of charge near the electrodes. Johnson and Cole derived empirical equations for the *ionic contribution* to ε^*:

$$\varepsilon'_I = \varepsilon_u + \frac{\varepsilon_r - \varepsilon_u}{1+(2\pi f\tau)^2} \qquad [23]$$

$$\varepsilon''_I = \frac{\sigma}{2\pi f \varepsilon_0} + \frac{(\varepsilon_r - \varepsilon_u)\,2\pi f\tau}{1+(2\pi f\tau)^2} \qquad [24]$$

where ε_0 is the permittivity of a vacuum ($8.85*10^{-14}$ Farads/cm) and σ is the conductivity ($ohm^{-1}cm^{-1}$), an intensive variable, as compared to the

conductance G (ohm^{-1}), which is dependent on both cell and sample size. The first term in Equation [24] describes the conductance of ions translating through the medium. the second term results from the electrode polarization and makes impedance measurements difficult to interpret and use as the frequency at which the measurements are made becomes significantly lower.

Rearrangement of Equation [24] yields:

$$\varepsilon''_I 2\pi f = \frac{\sigma}{\varepsilon_0} + \frac{(\varepsilon_r - \varepsilon_u)((2\pi f)^2 \tau)}{1+(2\pi f \tau)^2} \qquad [25]$$

The second term on the right-hand side becomes negligible when the following inequality is satisfied:

$$\frac{(\varepsilon_r - \varepsilon_u)((2\pi f)^2 \tau)}{1+(2\pi f \tau)^2} << \frac{\sigma}{\varepsilon_0} \qquad [26]$$

At frequencies where this expression is satisfied, the ionic conductivity is related to the loss factor by the expression[4]:

$$\varepsilon''_I 2\pi f = \frac{\sigma}{\varepsilon_0} \qquad [27]$$

Electrode polarization is a significant and often difficult to account for factor at frequencies below 10 Hz and/or for high values of ionic mobility S usually associated with a highly fluid resin state. It results from the generation of a 'mini-capacitance' by the extremely rapid movement of electrons between the obverse and reverse surfaces of the plates of the capacitor. Thus the dielectric material experiences a net capacitance slightly decreased from the theoretical. Frequencies have been found for which sensor electrode polarization and dipole effects are negligible. The frequency dependence of ε^* due to dipolar mobility is generally observed in the kilohertz to megahertz regions of the electromagnetic spectrum. For this reason the hertz to megahertz range is optimal for measuring the ionic mobility parameter σ and the dipolar mobility parameter σ^{-1} as represented in Figure 2.5.[1]

Two Molecular Probes

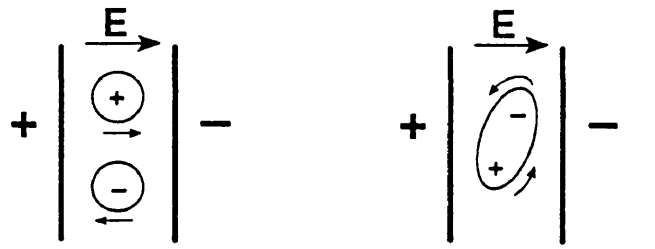

Figure 2.5

QUALITATIVE PROCESS AUTOMATION LANGUAGE

The *Qualitative Process Automation Language (QPAL)* was developed at the U.S. Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, by Frances Abrams and coworkers. Further development and commercialization of QPAL has been done by the Universal Technology Corporation, Dayton, Ohio. The commercial version of QPAL is known as the *Composite Cure Control System (CCCS).*

QPAL is a highly structured derivative of the standard FORTH programming language for the Macintosh™ that has been tailored for composite cure processing. QPAL contains the material or process specific acquired knowledge and goals of the human expert(s) who develop it in the form of events or states or *episodes*. These episodes represent chemical or physical occurrences which have been previously determined to have significance to the ultimate goal of fabricating a given composite structure. The goals of the intelligent cure control system are defined in terms of the achievement or avoidance of these states. As a result, the QPAL based expert system can detect and respond to changes in the ongoing cure process without the need for human intervention. A schematic representation of the PMR-15 expert cure control process using QPAL is shown in Figure 2.6.

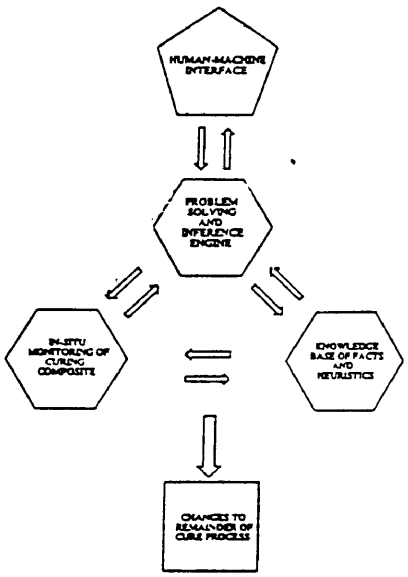

Figure 2.6

The human-machine interface is simply the ability of the expert system creator to modify the FDEMS data acquisition and transmission software and the QPAL knowledge base logic software via computer keyboard. The heart of the intelligent composite cure control system is contained in the three components represented by the hexagons in Figure 2.6. The problem solving and inference engine consists of the logical process realized in the QPAL knowledge base. It is the portion of the expert system that ultimately makes decisions regarding the flow pathway of the overall composite cure process. The knowledge base of facts and heuristics is that portion of the QPAL logic software that contains the facts and the rules that are used by the problem solving and inference engine to interpret the ongoing fabrication process. Finally, the in situ monitoring of the curing composite is the previously described use of FDEMS sensing to continuously monitor the cure process. The information gathered via this technique is then passed to the QPAL subsystem where it becomes the basis for decisions made by the controlling knowledge base logic. The functions of these three components within the expert system are highly interdependent, as indicated by the arrows in Figure 2.6. The net result of this 'thinking' process is to make changes to the remainder of the composite cure control process when appropriate, thus optimizing composite part fabrication.

The four main components of QPAL described below comprise the problem solving and inference engine and the knowledge base of facts and heuristics as well as a portion of the user-machine interface found in the schematic representation of the closed loop expert control process shown in Chapter 1.

QPAL is an *object oriented* programming environment in which the heuristics, actions and goals of the overall control system are created and saved as an expert *knowledge base*. As a result, *concrete* physical objects such as dielectric sensors and autoclave controllers are easily represented and defined within the knowledge base. Similarly, *abstract* concepts such as 'composite cure complete' can also be defined as a discrete object within the QPAL environment. The net result is a full description of the cure process, from start up to shutdown, as a list of possible process episodes or states and the goals and the history of the current process. Thus QPAL offers a *declarative* method for describing a process which differs from, and is more easily interpreted than, a more traditional rule based procedural approach in that the order of occurrence of the process steps is

not predefined. Let us consider in more detail the major parts of the QPAL knowledge base.[7]

Cure Schedule

The brains of the QPAL knowledge base lies in the *Cure Schedule*. From the cure schedule, decisions are made regarding the actual cure plan to be followed, whether the expert system should shut down, and whether specific conditions exist which warrant aborting the entire fabrication process. Additionally, explanations may be made to the QPAL user regarding the current and previous states of the cure process; these explanations may be tailored to require either a passive or an active response from the operator.

One should note the syntax used in the above description. The use of the word *and* rather than *or* in the discussion of the role of the cure schedule notifies the reader that all of these things are done simultaneously. This is also true of the evaluation of the various possible states of the cure process which will be discussed shortly. As a result, it is possible to consider simultaneously several different possible responses of the intelligent cure control system. Thus achievement of certain conditions by the curing composite part will trigger subsequent changes in the processing profile to be followed.

Sensor Definitions

The QPA Language allows the knowledge base developer to represent chemical or physical changes in the state of the curing composite or its curing environment as *discrete sensor elements*. For example, changes in the matrix resin as it reacts may be described by changes in dielectric signal at a given frequency or by changes in local temperatures or both. Similarly, the overall state of the curing environment may be described by the applied pressure, tool temperature, active heating or passive cooling. The types of physical quantities which can be represented by sensor definitions are limited by two things:

1) the imagination of the knowledge base developer and
2) his or her ability to collect and transmit that data to the

QPAL system.[7]

Other important aspects of the sensor definitions are the freedom to define rates and accelerations as the user desires, and to define the source, whether internal or external to the knowledge base, of the raw data for a given sensor. Of course, the knowledge base developer must keep in mind the second limitation described above.

Episodes

This section of the QPAL knowledge base is the most important, and, generally, the most complicated part of the QPAL system. It is here that the human expert makes concrete the processes of composite part fabrication. These critical *episodes* or states in the cure process capture the possible control strategy flow pathways which will result in the fabrication of the desired composite structure. By separating the various parts of the overall cure process into discrete episode objects, several things are accomplished.

First, complicated reaction/fabrication processes are broken down into specific subtasks which relate to the overall cure plan. As a result, specific decisions regarding the attainment or the avoidance of certain processing events may be made. Second, the successful completion of these various episodes depends on the conditions required for a given segment to begin and for a specific segment to end. It is important to realize that these episode starting and ending conditions are not based on the passage of time. Rather, they depend entirely on events sensed during the curing process. Finally, decomposition of the gross fabrication process into a series of comprehensible critical episodes allows conflicting process goals to coexist. For example, the general process for curing a thermosetting composite requires an increase in temperature until a final, cured structure has been fabricated. Within that general 'heat to full cure' process, there may exist situations when active cooling of the tool is required. By separating these processes into independent episodes, conflict can be avoided.
And, since QPAL monitors the active or inactive nature of each episode simultaneously, rapid changes in the curing composite result in the appropriate expert system adjustments to the autoclave or thermal press controller(s).[7]

Abrams and coworkers present the following simple description of

the difference between a procedure-driven process and an event-driven process.

> The QPA System offers a 'declarative' method for describing a process which differs from the traditional procedural approach in that the order of the process steps are not specified. As an example, a step-by-step set of instructions for baking a cake might include:
>
> 1-preheat oven to 350 degrees
> 2-bake for 20 minutes
>
> This set of instructions does not include the possibility that the chemical reactions and physical processes involved in the transformation of a batch of dough to a cake (the baking process) may not occur as expected under the environmental conditions and the specific ingredients used.
>
> A different approach is to ***declare*** the constraints on the baking process, and let the QPA System conduct the exercise:
>
> 1-maximum temperature=350 degrees
> 2-done when cake "springiness"=some value
>
> Here we introduce a notion any baker already uses: even though the instructions originally read to bake for a certain time, most bakers quit when the cake springs back when poked. So, we invent some springiness value, locate a sensor capable of monitoring that value, declare the limits of the process, and establish a process instance in the QPA System process library which uses the primitive behavior "turn up the heat" to control the process.[8]

Controller Actions

The final piece in the knowledge base is the block of external behaviors providing control over the curing environment. These *controllers* output a series of commands to the hardware which controls the curing environment. The exciting or inhibiting action of the knowledge base controllers depends on decisions made within the episodes. As a

result, the knowledge base *optimizes* the curing process in order to efficiently achieve the overall goals of the fabrication process.

SYNERGISM BETWEEN FDEMS AND QPAL

The combination of the Dek Dyne, Inc. Frequency Dependent Electromagnetic Sensing System and the Abrams Qualitative Process Automation Language is the sensor-model intelligent, closed loop automated control system for composite cure developed by the author. Continuous, in situ monitoring of the microscopic state of the curing composite matrix electrical state and of several part/tool temperatures is performed by the specially modified FDEMS system. Select portions of this dielectric and temperature data is then passed to the Macintosh™-based QPAL system where the appropriate user developed knowledge base evaluates the data, makes decisions, and sends the resultant controller setpoint command changes back to the FDEMS system. The FDEMS system then uses its specifically developed interface with the autoclave or thermal press controller(s) to update the status of the controller(s). Thus the feedback loop for intelligent composite cure closes.

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CHAPTER 3

PMR-15

PMR-15, or Polymerization by Monomeric Reactants with a formulated molecular weight of the imidized polymer of 1,500 grams per mole, has a bright future in aerospace applications. Of the several types of matrix materials (*e.g.*, thermoplastic toughened epoxies, bismaleimides, polyimides) being considered for use in advanced composites, PMR-15 is the frontrunner. It is the leading resin system for applications involving an extended service life at temperatures up to 300°C.[1]

The PMR approach was developed by Serafini and coworkers at the NASA Lewis Research Center, Cleveland, Ohio, USA in the early 1970's. This approach involves the formation of a low boiling solution of monomers which is then used to impregnate the desired reinforcing fibers.[1] This solution has a very low viscosity and thus ensures a fairly uniform deposition of resin on the desired prepregging fibers.

Chemistry

PMR-15 in its conventional formulation is a mixture of the monoalkylester of 5-norbornene-2, 3-dicarboxylate (NE), the dimethyl ester of 3, 3', 4, 4'-benzophenone tetracarboxylate (BTDE), and 4, 4'-methylene dianiline (MDA) in the molar ratios 2(NE):(n+1)(MDA):n(BTDE) where n=2.087, dissolved in methanol. The NE and BTDE are first formed by dissolving their respective anhydrides in the methanol solvent. MDA is then added; this route prevents premature formation of the polyamide acid prepolymer. The resultant solution of monomers can be maintained as a homogeneous, low viscosity mixture from which the low boiling solvent can be easily removed. These particular molar ratios have been determined by NASA to provide the best overall balance of processing characteristics and composite thermomechanical and physical properties with 316°C thermo-oxidative stability for use in high temperature applications.[1-8, 10]

The monomeric solution has the methanol solvent removed at low temperatures. At temperatures between 121°C and 232°C, the monomers

undergo *in situ* cyclization to form a norbornene-end capped low molecular weight imide prepolymer. Addition polymerization of the maleimide end caps occurs at high temperatures without the evolution of significant volatile reaction by-products. The general reaction sequence for the polymerization of PMR-15 is shown in Figure 3.1.

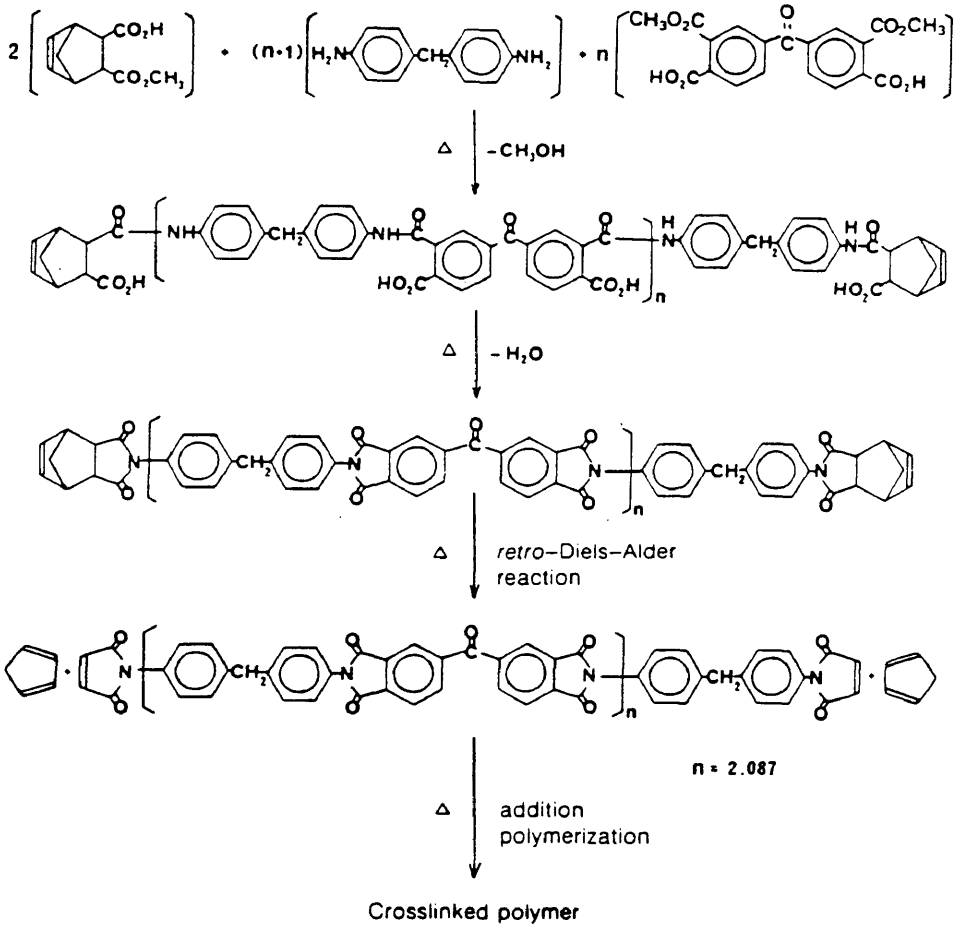

Figure 3.1

The first step shows the mixture of the unreacted NE, MDA and BTDE monomers as they exist in solution. The second shows the addition of heat to temperatures near 200°C and the concurrent loss of methanol. This results in the addition of the monomeric substituents to form the low

molecular weight polyamide acid. Previous theories on the mechanism of the formation of the prepolymer assumed this key step to be a direct nucleophilic displacement of the methoxy groups by amines, as seen in Figure 3.2.

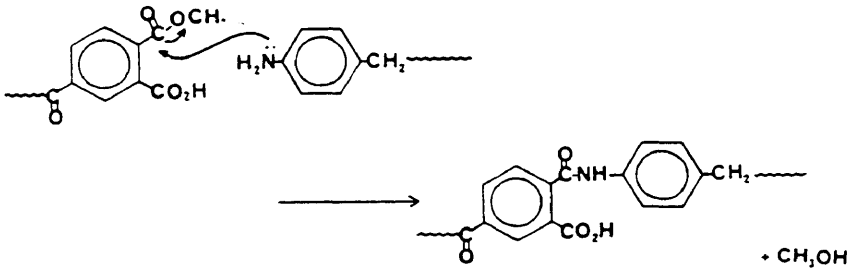

Figure 3.2

Similar aminolysis reactions of simple esters, however, are very slow under similar conditions (100-150°C). Johnston and coworkers[10] have demonstrated that an alternative mechanism involving the formation of an anhydride intermediate is more plausible (Figure 3.3).

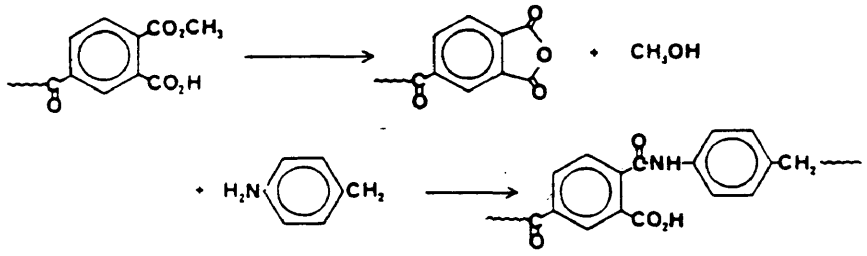

Figure 3.3

Two factors favor this pathway over simple aminolysis. In the first step of Figure 3.3, the intramolecular ring closing process resulting in the loss of methanol is entropically more favorable than the bimolecular aminolysis process. In the second step of Figure 3.3, the carboxylate anion leaving group is a much weaker base than the methoxy anion leaving group found in the direct aminolysis process. Thus the ring opening pathway in the addition of MDA to the anhydride further supports arguments for the existence of an anhydride intermediate in the early stages of the polymerization of PMR-15.

On heating, acid esters undergo a reverse reaction to form cyclic anhydrides and methanol. These anhydrides then react rapidly with amines to form the polyamide acids. Methanol appears to act as a masking agent for the highly reactive anhydrides, thus preventing premature reaction.[1] Lauver[3] and Garcia and Serafini[8] have substantiated the existence of an anhydride intermediate during the course of PMR-15 processing via the use of infrared spectroscopy. Robillard[9] in our lab at William and Mary has also substantiated the formation of the anhydride intermediate through the use of NMR spectroscopy on model compounds of those found in the imidization reaction. Additionally, she has demonstrated a temperature dependence of the reactions between the diester and the amine and between the nadic ester and the amine. These results could prove significant in future expert system developments for PMR-15 where careful control of the thermal history could preferentially 'choose' one imidization pathway over another, leading to varied mechanical properties in the final composite part.

The third step in Figure 3.1 shows the *in situ* cyclization of the polyamide acid to form the norbornene endcapped imide prepolymer. While the final cure reaction is very complex as a result of the many possible side reactions to the imidization process and therefore not well understood, the fourth step shows the conventionally accepted *retro*-Diels-Alder reaction of the imide prepolymer to form maleimide terminated prepolymer and cyclopentadiene. Step 5 is the final crosslinking process. This involves the limited copolymerization of the maleimide end groups with the double bonds of the cyclopentadiene and the unreacted norbornene imide prepolymer end groups to produce a final crosslinked structure with minimal evolution of residual pentadiene.[1]

Prepregging

Commercial prepregging of PMR-15 can follow several routes. Woven fabric prepreg is prepared by a solvent-impregnation method using a 50% by weight solution of the three monomers in methanol. After the actual impregnation, the fabric is heated at low temperatures to reduce the solvent methanol content to 5-10% by weight. The residual methanol provides tack to the prepreg, thus making it relatively easy to lay the prepreg plies in the desired pattern. On completion of the prepregging process, the rolls of prepreg are interleaved with polyethylene film and stored at -18°C. This is to prevent any reaction between the monomers which should still be unreacted at this stage. Unfortunately, experience has shown that the resin reaction process will advance slowly even under these conditions, leading to a limited shelf life similar to that observed with most resins.

Unidirectional fiber prepreg or tape is difficult to prepare by the solvent impregnation route due to the tendency of the fiber tows to separate. Instead, a pseudo hot melt process is usually used.[1] This involves the careful melting of an 80-90% by weight solution of monomers in methanol. The fiber tape is then passed through a chamber containing the monomer melt and then rapidly cooled prior to being interleaved with polyethylene film and taken up on a spool. Fabrication of composite structure using PMR-15 unitape often proves difficult due to the tendency of the fiber tows to separate and the lack of tack resulting from the increased loss of methanol during the hot melt process relative to solution prepregging.

A third PMR-15 prepregging technique involves coating the fibers with the powdered oligomer of the carefully prestaged and vacuum dried monomeric solution by proprietary techniques.

Considerations

The making of any type of prepreg material, including PMR-15, requires close control on the production process. Several important considerations are given below.

1) The 'tightness' of the fiber fabric can hinder the impregnation process.

2) Lack of stability in stored prepregging solution can lead to the formation of undesirable by-products.
3) Lack of precise control over the thermal profile of the resin during the prepregging process leads to unwanted by-products.
4) The amount of residual methanol present in the prepreg can adversely affect a standard processing/cure cycle.
5) Poor handling and storage of the prepreg may lead to water adsorption, resin aging, and damage to the material.

While it may prove to be impossible to satisfactorily process prepreg suffering from any of the above problems (as well as other problems which may exist at other stages in the fabrication process), the use of an intelligent expert system capable of adjusting to a range of precure material properties would greatly increase the probability that a given prepreg sample could be used.

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CHAPTER 4

EXPERIMENTAL METHODS

FDEMS Iinstrumentation

FDEMS measurements were made using a Hewlett-Packard 4192A LF Impedance Analyzer controlled by an IBM compatible 286 or 386 personal computer containing a National Instruments IEEE-488 General Purpose Interface Bus (GPIB) communications card. GPIB control of the below peripheral devices completed the FDEMS measurement system. Multiplexing of sensor and temperature measurements were accomplished through computer control of a Keithley 7067 4-wire scanner card and a Keithley 7057A thermocouple card, respectively, inserted in a Keithley Instruments 705 Scanner mainframe. Coaxial 50 ohm cable completes the FDEMS circuit and Type J iron-constantan thermocouple wire completes the temperature circuit. Temperature measurements were obtained by connecting the thermocouple card to a Keithley Instruments 199 System DMM (a voltmeter). Control of a Baron-Blakeslee autoclave and of a Carver 12-ton thermal laboratory press with special modifications for remote temperature control was also accomplished via IEEE-488 communications.

Measurements of conductance and capacitance were taken at frequencies ranging from 5 Hz to 1 MHz were taken at regular intervals throughout the cure process. These values were then converted to the desired components of the complex permittivity, $\mathcal{E}^* = \mathcal{E}'\text{-}i\mathcal{E}''$, using Dek Dyne FDEMS Data Acquisition Software. The data acquisition software has been modified by the author to allow for communication with the QPAL component of the intelligent, automated cure control system that is the focus of this paper.

Accurate dielectric measurements were made possible by the use of disposable, planar, geometry-independent microsensors developed by Kranbuehl. This FDEMS sensor (Figure 4.1) consists of a fine array of interdigitated comb electrodes deposited on an inert substrate. The electrodes are constructed from gold and other noble metals while the substrate is composed of high temperature ceramics.

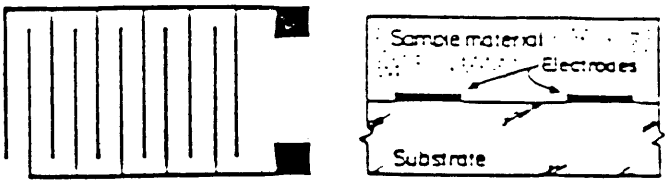

Figure 4.1

This sensor is designed to withstand curing temperatures in excess of 400°C, pressures up to 1000 psi, and oxidative conditions during processing. It is capable of continuously monitoring the entire range in magnitude (usually 10^{-2} to 10^{8}) of both the real and the imaginary components of the complex permittivity described above.

QPAL Instrumentation

The Qualitative Process Automation Language (QPAL) runs on a Macintosh™ IIci computer. The PMR-15 specific knowledge is loaded into the QPAL shell and then run. This collection of rules, heuristics and actions reads the dielectric and the temperature data sent by the FDEMS system and then makes decisions regarding the remainder of the cure process. Adjustments to the autoclave or the press controller(s) are made via the FDEMS interface with said controller. The transfer of information between the PC-based FDEMS system and the Macintosh™-based QPAL system occurs via a specialized RS-232 9 pin serial-to-modem computer cable.

Preset Time-Temperature Profile

Much of the processing of today's composite materials is accomplished by following a 'manufacturer's recommended cure cycle'. This approach provides a standard recipe for any cure using a specific matrix resin, regardless of its precure history. While this approach to

composite fabrication will generally result in a usable part, several noteworthy difficulties are often encountered.

The first is that most recommended cure cycles are quite conservative, often involving a complicated series of ramps and extended holds to ensure that the resin achieves the same chemical states from part to part. Reality shows, however, that many times the resin completes various stages in its reaction process, including reaching 'full cure', sooner or later than the recommended cure cycle expects. Thus the fabrication process and the bottom line of ultimate costs could benefit from a run to run optimization of the cure profile.

A second difficulty in composite part fabrication lies in the variability of the raw materials used. These variations may be as gross as intentionally prestaging, or advancing of the cure reaction, of the resin or as subtle as the uptake of a few weight percents of moisture in a freezer environment. The literature is filled with examples of the effects of resin batch variability, material out time and subsequent reaction advancement, moisture adsorption, and other prefabrication factors which directly affect the behavior of a given monomeric/polymeric material during cure.

For example, Ahn, *et. al.*, provide data regarding the relationship between epoxy prepreg aging and material tack. Tack may be defined as "the ability to assume and hold shape during lay-up as well as in consolidation processing".[1] They studied the tack of a commercially available carbon fiber/epoxy prepreg system as a function of aging at several temperatures as well as elevated moisture levels. The results of this study showed a general trend of decrease in prepreg tack with increasing reaction advancement of the matrix resin. This trend is complicated, however, by their observations that up to a certain, variable temperature, prepreg tack may be improved despite aging of the prepreg due simply to the increase in resin fluidity and resulting interply interaction as a result of the increased temperature.[1]

A final, and often significant, difficulty which arises in the fabrication of composite parts is the variation in the thermal responses/profiles of different tools and curing environments. In fact, even a single autoclave may experience uneven heating throughout its cavity, leading to localized hot and cold spots where one expects temperature uniformity. Similar variability may also be observed in the heating platens of a thermal press. The net result of these temperature variations is the nonuniform cure of a given composite, unless allowances are made for the variations. The use of a well-developed, intelligent processing control

system which incorporates this knowledge and can adjust accordingly should minimize these problems.

Thus we can see that the standard, preset recipe approach to composite cure may make it impossible to satisfactorily process materials which lie outside an often narrow window of acceptability. Since the severity of these variations from the norm is usually unknown prior to material use, the use of an intelligent cure control system seems to be the logical choice to maximize productivity and, ultimately, profits.

QPAL Knowledge Base Development

As is the case with the elucidation of any 'ideal' process, the development of a resin specific *knowledge base* for intelligent cure control requires significant effort. The first step in developing a knowledge base is to perform various chemical and mechanical analyses of the cure of the resin. The typical information gathered relates to the buildup in the resin degree of cure as a function of time and temperature, as well as information regarding the viscosity of the curing material prior to gel. Examples of instrumental techniques useful for gathering such information are Differential Scanning Calorimetry (DSC), dynamic rheology and Nuclear Magnetic Resonance (NMR), and various mechanical tests for desired final composite properties. From these (and other analyses which may, for example, provide insight on desired mechanisms for specific molecular structure formation) studies, the basic desired cure profile for the material may be created. Kranbuehl has shown that Frequency Dependent Electromagnetic Sensing (FDEMS) can provide valuable information regarding the microscopic state of a curing resin continuously throughout the cure process. This experimental technique has been used to aid in the determination of an ideal cure time/temperature/pressure profile for PMR-15 prepreg. A final method for studying resin cure is to use experimental kinetic information regarding changing degree of cure and viscosity to model the effects of variations in chemical composition and/or temperature and pressure on composite cure and consolidation. This methodology provides the opportunity to quickly and easily determine the most desirable overall cure profile, saving much of the considerable costs involved in experimentally evaluating these permutations vial real runs. Modelling work by Loos and Kranbuehl provided the basis for the 200°C hold and the 'ideal' 265°C crosslinking and

compaction hold found in the PMR-15 knowledge base created for this project.

One advantage of the QPAL system is its ability to perform both internal and external simulations of the knowledge base as it is being developed. The QPAL simulator allows the user to create sample 'experimental' traces of knowledge base sensor data and then run this data with the developing knowledge base. While these traces do not possess the subtleties and the complexity associated with the cure of a real resin, they are able to imitate the major transitions associated with resin cure. Various approximations of critical dielectric and temperature data were used in developing the PMR-15 knowledge base.

The second type of simulation possible with the QPAL system uses previously collected experimental dielectric and temperature data. This data exists as a series of preset time-temperature cure profile runs as well as optimized cure runs obtained using earlier versions of the PMR-15 knowledge base. Using a modified version of the FDEMS data analysis software, this experimental data may be used in conjunction with the QPAL system to test a given knowledge base. (These modifications to the FDEMS Data Manipulation Software were made by the author and Bruce Hinds, formerly with the Phillips Laboratory, Edwards Air Force Base, California.)

The second type of simulation proved especially useful for this project. We could test the ability of a given knowledge base heuristic to respond to experimentally observed variations in the cure process. For example, this procedure made it possible to fine tune the procedure to determine the decreasing rate of change in the dielectric signal (*i.e.*, the 'folding over') as the PMR-15 resin reached its minimum viscosity temperature for the *circa* 265°C CrossLinking Hold. This process was also very effective for testing the limits of the knowledge base in determining the minimum value of the change in slope of the FDEMS signal $((d\varepsilon''/dt)/\varepsilon'')$ used to signal completion of the imidization process during the 200°C Imidization Hold and completion of the final crosslinking process during the 320°C Final Cure Hold. Without this capability, we would have had to perform quite a few fabrication runs to determine the effects of resin variation as well as instrument noise levels on these criteria. As can be seen, the use of the simulation capabilities of the QPAL system speeds knowledge base development. By coupling it with the judicious performance of experimental fabrication test runs, the user can create the desired knowledge base in the most efficient manner possible.

Part Fabrication

The first step in making a composite using the intelligent, sensor-model automated cure control system is the layup. The ceramic FDEMS sensor(s) with the attached gold wire leads is placed at the desired location(s) on the bottom metal plate of the mold or tool. The FDEMS sensor active surface is protected from the electrically conductive graphitic environment of the prepreg by a thin, protective fiberglas filter material. Next a second, thin (*i.e.*, <1/8") metal plate with a space for the FDEMS sensor is put in place to protect the sensor from cracking due to too much pressure. Because this shim has a space for the FDEMS sensor, the sensor 'sees' an uninterrupted flow of PMR-15 as the prepreg is heated, thus *wetting out*. The desired number of PMR-15/graphite prepreg plies, cut to specification, are then stacked over the sensor. For the 1/8" thick panels that make up the majority of the panel fabricated for this project, this corresponds to 6 prepreg plies. Thermocouple #1 is placed at the midpoint of the ply stack, providing the experimental part midpoint temperature used in the QPAL knowledge base. Thermocouple #2 is placed on top of the last ply to provide a record of the temperature history seen by the upper extreme of the composite part. The third thermocouple is located outside of the composite on the metal base plate; this provides the lower temperature profile seen by the curing composite part. A cork material is then used to create a dam around the completed prepreg layup in order to minimize resin loss. During the actual cure process, the preform is compressed down to this dam by the application of pressure with the thermal press. A release/bleeder material is placed on op of the prepreg stack and the entire layup is covered with a metal top plate. Finally, the layup should be bagged and a vacuum pulled. The part is then placed in the thermal press. Finally, the power to the controller and the heating platens is turned on. For autoclave runs the above procedure is similar, with pressure, heat and vacuum being applied remotely through the autoclave controller.

The PMR-15 prepreg used was supplied by ICI Fiberite, Tempe, Arizona.

Once the part preparation is completed, the user runs the appropriate PC and Macintosh™ software. The FDEMS Data Acquisition/transmission Software used in the expert system runs from the Microsoft™ Quickbasic PC environment. The Macintosh™ IIci runs the QPAL component of the intelligent cure control system. From the QPAL environment, the user loads the desired knowledge base for use in the

smart cure process. From here the expert system initialization is menu driven. Once completed, the PMR-15 composite fabrication process will proceed to completion without the need for operator interaction.

NOTES FOR CHAPTER 4

1. Ahn, K.J., L. Peterson, J.C. Seferis, D. Nowacki and H.G. Zachmann, *J. Appl. Poly. Sci. (vol. 45)*, 399-406 (1992).

CHAPTER 5

RESULTS AND DISCUSSION

The QPAL Knowledge Base

Kranbuehl, Loos and others have examined the relationship between changes in viscosity and the progression of the PMR-15 reaction process as it undergoes and completes initial maximum flow, imidization, maximum flow for part consolidation, and crosslinking.[1] Based on this process modelling work, one expects the curing prepreg matrix resin to have its highest flow at the viscosity minimum occurring around 80°C, just prior to the onset of the imidization reaction. The imidization reaction should reach completion at the end of a 200°C hold; that is, the 'imidization temperature'. A slow ramp to approximately 265°C shows a second viscosity minimum. A hold at this second viscosity minimum temperature allows for the addition of pressure to consolidate the part and the driving off of residual volatiles. Finally, a ramp to and a hold at 320°C, the final crosslinking temperature, allows 'full cure' to be achieved.[2]

The current QPAL knowledge base incorporates this information into a series of processing milestones, or episodes, for PMR-15 prepreg composite part fabrication. The knowledge base contains ten episodes which control the cure of the PMR-15 based on the achievement or avoidance of a series of reaction subgoals to the final goal of curing the composite. It is the interpretations made by and the actions of these *cure episodes* which optimize the overall cure process.

Knowledge Base Cure Episodes

Pre-Imidization Max Flow

The first episode in the cure of PMR-15 determines whether the resin achieves full part wet out (*i.e.*, maximum flow) and thus full wet out of the FDEMS sensor as the part is being slowly heated to approximately 80°C. FDEMS sensor wet out is defined by the dielectric signal $Log(\mathcal{E}''*\omega)$ (where $\omega = 2\pi*frequency$) for frequencies 5 kHz, 25 kHz and 50 kHz achieving a

value greater than 6.4 for three consecutive data acquisition cycles. Additionally, the ionic mobility, or s, at 5 kHz in the region where the frequency lines overlap, must be decreasing. The three consecutive readings requirement eliminates the possibility of a spurious value leading to an interpretation of successful completion of the episode, while the decreasing sigma requirement ensures that the solvent elution process has progressed enough to allow continuation of the cure cycle. Alternatively, the Pre-Imidization Max Flow cure episode requirements may be satisfied by the FDEMS signal $Log(\varepsilon''*\omega)$ for frequencies 5 kHz, 25 kHz and 50 kHz achieving a value greater than 7.4 for two consecutive data acquisition cycles as well as having a decreasing sigma. Finally, in the case of no sensor wet out in the expected *circa* 80°C temperature range, the Pre-Imidization Max Flow episode continues to slowly increase the temperature of the part with the hope that a higher temperature will cause the resin to reach a low viscosity and thus wet out the FDEMS sensor. If sufficient resin flow and thus sensor wet out has not occurred before the part reaches 150°C, the cure process is terminated.

Imidization Onset (Nadic)

The second cure episode for PMR-15 has three requirements. During a hold at the temperature determined by the completion of the previous episode, the value of $Log(\varepsilon''*\omega)$ for frequencies 5 kHz, 25 kHz and 50 kHz must be less than 8.0 for three consecutive readings. This rule ensures that prepreg samples containing high percentages of solvent elute enough to minimize the formation of voids by relatively large volumes of trapped volatiles later in the cure process. Second, the magnitude of the FDEMS signal at 25 kHz must be less than 0.9 times the maximum value for the signal for two consecutive readings. This maximum value in $Log(\varepsilon''*\omega)$ is determined at the beginning of the episode. Third, the change in FDEMS signal $((d\varepsilon''/dt)/\varepsilon'')$ at frequency 25 kHz must be less than zero (that is, decreasing) for four consecutive readings. the last two rules signal the onset of the nadic imidization process in PMR-15. Finally, a thirty minute backup hold time exists in the event that the above criteria are not met.

Imidization Ramp

This stage is simply a slow 2-3°C per minute ramp to the 200°C imidization temperature. FDEMS data is collected continuously during this time.

Imidization Hold

This episode determines completion of the PMR-15 imidization reaction. It checks for a 'flattening' in the FDEMS signal which indicates completion of the imidization reaction process. Reaction completion is indicated by the absolute rate of change $((d\varepsilon''/dt)/\varepsilon'')$ of the FDEMS signal at either 5 kHz or 25 kHz being less than 3.0×10^{-4} for four consecutive data acquisition cycles. In addition, the actual part temperature must be at least 193°C before these criteria may be considered, thus ensuring that the required model-determined imidization reaction temperature is reached. Finally, there is a maximum hold time of three hours in the event that the above criteria are not met. This conservative safety feature ensures completion of the imidization reaction in all but the most unusual, and unpredictable, of cases without making the cost of processing prohibitive.

CrossLinking Min Viscosity (look for viscosity minimum)

The fifth episode slowly ramps the curing composite to a temperature near the model-determined 265°C ideal for part consolidation. During this time the knowledge base checks the incoming FDEMS data to determine if a minimum in viscosity has been achieved. this rule requires the changing FDEMS signal at 25 kHz to be less than 0.6 times the slope maximum determined during the ramp for four consecutive data acquisition cycles. Once this decreasing sigma criterion is met, the episode terminates. In the event that the decreasing sigma does not occur prior to the part reaching 280°C, the episode is terminated when 280°C is reached. This last rule ensures that the PMR-15 reaction process does not advance significantly into the final crosslinking reaction temperature regime before part consolidation and residual volatile evolution can occur.

CrossLinking Hold (hold at minimum viscosity temperature)

This episode holds the composite part at the minimum viscosity temperature just determined until the change in FDEMS signal $((d\varepsilon''/dt)/\varepsilon'')$ at either 5 kHz or 25 kHz is less than 3.0×10^{-4} for four consecutive data acquisition cycles. If this flattening of the FDEMS signal does not occur, the hold will end after a maximum of two hours.

Final Cure Ramp

This part of the knowledge base ramps the curing composite at 2- 3°C per minute to the final cure temperature of 320°C. During this ramp the crosslinking reaction between polymeric chains progresses.

Final Cure Hold (complete crosslinking)

This episode holds the curing composite part at 320°C until the final crosslinking reaction of PMR-15 reaches completion. The knowledge base determines that the cure is complete when the rate of change in the FDEMS signal, $((d\varepsilon''/dt)/\varepsilon'')$, at either 5 kHz or 25 kHz is less than 3.0×10^{-5} for four consecutive FDEMS readings. Once this user-defined criterion for '100% cure' is reached, the knowledge base begins to shut down the autoclave or the thermal press. If the change in slope of the FDEMS signal never achieves the above value for four consecutive readings, the episode, and thus the cure process, will terminate after four hours. Again, this backup hold time attempts to salvage the part by employing a conservative maximum time for the hold. The user hopes that this will allow the part to be used rather than having wasted the considerable costs of the materials, labor and processing time used to bring the composite to this point in the fabrication process.

Cool Down

The penultimate episode ends the active heating process and waits for the composite to cool to the desired temperature. (This cool down temperature is 295°C in the case of the William & Mary thermal press.)

Shut Down

The final knowledge base cure episode terminates the intelligent cure control process by signalling the conclusion of the cure process to the FDEMS Data Acquisition Software. Thus the fabrication process is completed.

For the following analyses, the above episodes shall be labelled as follows:

Region A = PreImidization Max Flow
Region B = Imidization Onst (Nadic)
Region C = Imidization Ramp
Region D = Imidization Hold (complete imidization)
Region E = CrossLinking Min Viscosity (look for viscosity minimum)

Region F = CrossLinking Hold (hold at minimum viscosity temperature)
Region G = Final Cure Ramp
Region H = Final Cure Hold (complete crosslinking)
Region I = Cool Down
Region J = Shut Down

Typical Preset Time-Temperature Profile

The problem of identifying the times of occurrence of each stage in the PMR-15 reaction process can be eliminated through in situ FDEMS sensor measurement of the resin's real time state in the processing tool.[2] Figure 5.1 shows the FDEMS output for a typical fresh PMR-15 prepreg (ICI

Fiberite Lot# 10502S) cure using a preset time-temperature cure profile incorporating the results of the previously mentioned process modelling work of Loos and Kranbuehl.[1] Region A shows FDEMS sensor wet out on a slow 2°C/minute ramp to 80°C and the achievement of a maximum in fluidity (a viscosity minimum) for the system. Region B is a one hour hold at 80°C to allow for solvent elution and maximum part wet out. Region C shows the onset of imidization as characterized by a drop of over four decades in the ionic mobility, the $(\mathcal{E}''*\omega)$ overlapping lines, and thereby the rise in the viscosity of the resin. Region D shows the continuation of the imidization reaction as it is held for one hour at 200°C. Normally one would want to hold at this temperature until the imidization reaction is completed as evidenced by the change in slope of the FDEMS signal $((d\mathcal{E}''/dt)/\mathcal{E}'')$ approaching zero. We can see here that the use of FDEMS sensor feedback would have shown the imidization reaction to be incomplete and therefore the 200°C hold would have been extended by the intelligent, automated cure control system that is the subject of this work. Regions E and F show the viscosity minimum achieved during a ramp to 270°C and the subsequent decrease in the FDEMS signal as the part is consolidated and the residual volatiles are evolved during the hold. Finally, Regions G and H show the ramp to and hold at 320°C, the final crosslinking reaction hold temperature. One should note that after approximately 75 minutes in the 320°C hold the change in slope of $\mathcal{E}''$ (that is, $((d\mathcal{E}''/dt)/\mathcal{E}'')$) is small and close to zero, and 'full cure' based on user criteria is reached. Had the FDEMS/QPAL intelligent, automated cure control system controlled this cure process using the criteria of final cure being completed when $((d\mathcal{E}''/dt)/\mathcal{E}'')$ for frequencies 5 kHz and 25 kHz is less than 3.0×10^{-5}, it could have ended the run after a total cure time of only 384 minutes, thus saving 96 minutes of the 480 minute total cure time shown. This criteria for the achievement of final cure was determined empirically from a series of preset cures of several different 'fresh' samples of PMR-15 prepreg. Some of these fresh and moderately aged (maximum of three days at room temperature) samples achieved the chosen value at various points during the final preset 320°C hold while others did not. It was determined that this value, where $((d\mathcal{E}''/dt)/\mathcal{E}'')$ was essentially unchanging within the noise level of the instrumentation, was acceptable when the variations inherent within the prepreg samples were taken into consideration. Thus one sees the potential power of such an intelligent, closed loop cure control process.

Intelligent, Automated Cure

Figure 5.2 shows the dielectric and temperature output for the intelligent sensor-model automated cure of fresh PMR-15 prepreg, ICI Fiberite Lot# 11769S. Region A, PreImidization Max Flow, shows the panel as it warms from room temperature to approximately 80°C. FDEMS sensor wet out is indicated by the sharp increase in the magnitude of the $Log(\varepsilon''*\omega)$ signal at 81°C. Region B, Imidization Onset, is a hold near 80°C to allow for solvent elution prior to beginning a slow ramp to the imidization hold temperature. Region B requires that the magnitude of $Log(\varepsilon''*\omega)$ at 500 Hz, 5 kHz and 25 kHz be less than 8.0 for four consecutive FDEMS readings, that the change in FDEMS signal $((d\varepsilon''/dt)/\varepsilon'')$ at 25 kHz be decreasing (*i.e.*, negative) for four consecutive FDEMS readings, and that the magnitude of $Log(\varepsilon''*\omega)$ at 25 kHz decrease by ten percent from the maximum value determined at the beginning of the hold. Based on these criteria, Region B is an eleven minute hold at 81°C. Region C, Imidization Ramp, is the slow 2-3°C per minute ramp to the 200°C imidization hold temperature. Region D shows the imidization hold at 200°C lasting only 12 minutes before meeting the criterion that the magnitude of $((d\varepsilon''/dt)/\varepsilon'')$ at 25 kHz be less than 3.0×10^{-4} for four consecutive FDEMS data acquisition cycles. This short dwell time indicates the virtual completion of the imidization reaction prior to entering the QPAL knowledge base Imidization Hold for this particular prepreg sample. Region E, CrossLinking Min Viscosity (look for viscosity minimum) and Region F, CrossLinking Hold (hold at minimum viscosity temperature) are the ramp to the minimum viscosity consolidation temperature and the preset 30 minute hold at that temperature, respectively. The QPAL knowledge base determined the minimum viscosity temperature for this run to be 260°C based on the criterion that the ionic conductivity of the resin as seen in the low frequency FDEMS signal lines be decreasing (*i.e.*, the $Log(\varepsilon''*\omega)$ signal is 'folding over'). This value for the secondary viscosity minimum is almost identical to the 'ideal' model generated 265°C hold temperature for part consolidation. Region G, Final Cure Ramp, shows the slow ramp to the 320°C final crosslinking cure temperature and Region H, Final Cure Hold (complete crosslinking), shows the achievement of full cure, based on the criterion that the magnitude of $((d\varepsilon''/dt)/\varepsilon'')$ at 25 kHz be less than 3.0×10^{-5} for four consecutive FDEMS data acquisition cycles, after 115 minutes in the 320°C hold. Regions I and J show system cool down and shut down, respectively.

The total cure time for this fresh PMR-15 prepreg processing run was 338 minutes.

Figure 5.3 shows the intrabatch variability in a second fabrication process using fresh PMR-15 prepreg from ICI Fiberite Lot# 11769S. The first 3 regions for Figures 5.2 and 5.3 are very similar. In Region D of Figure 5.3, however, the imidization hold is much longer, lasting 105 minutes as opposed to the twelve minutes in Region D of Figure 5.2. Region E shows the ramp to and the determination of the minimum viscosity temperature for part consolidation at 235°C by the QPAL knowledge base. This temperature is thirty degrees lower than the 'ideal' value based on the Loos modelling work.[1] New for this run is the criterion that the hold at this minimum viscosity temperature terminate when $((d\varepsilon''/dt)/\varepsilon'')$ at 25 kHz be less than 3.0×10^{-4} for four consecutive FDEMS readings. This occurred after 28 minutes in the Region F CrossLinking Hold. Region G again is simply the slow 2°C per minute ramp to 320°C. Region H, the Final Cure Hold, for this run lasted 240 minutes, the maximum allowed backup hold time. This happened because the change in magnitude of the FDEMS signal $((d\varepsilon''/dt)/\varepsilon'')$ at 25 kHz did not drop below 3.0×10^{-5} for four consecutive FDEMS data acquisition cycles. regions I and J are the expert system cool down and shut down, respectively. As a direct result of the extended hold time at the 320°C crosslinking final cure temperature, as well as the significant dwell at the 200°C imidization hold temperature, this fabrication run required 625 minutes to reach completion.

Figure 5.4 shows the intelligently controlled cure of a PMR-15 prepreg sample aged four months under freezer conditions (ICI Fiberite Lot# 10645S). Region A shows FDEMS sensor wet out based on the criterion that $Log(\varepsilon''*\omega)>7.4$ for two consecutive readings during the slow ramp to approximately 80°C. (Recall that the actual temperature for this hold is based on the temperature at which the preceding episode reached completion.) As previously discussed, Region B is a hold near 80°C to allow for solvent elution and maximum part wet out. For this run, the Region B imidization Onset hold at 82°C is 42 minutes in length. Region C is the slow ramp to the 200°C imidization hold temperature. The 200°C hold seen in Region D lasted only seven minutes because the imidization reaction reached completion based on the criterion that the magnitude of $((d\varepsilon''/dt)/\varepsilon'')$ at 25 kHz be less than 3.0×10^{-4} for four consecutive FDEMS readings. Region E is th slow ramp to the CrossLinking Min Viscosity temperature of 280°C as determined by a decrease in the rate of change of of the FDEMS signal, while Region F is a thirty minute hold at that part consolidation temperature. Following the Region G ramp to the final cure

temperature, the expert system determined full cure of the composite part after 110 minutes in the Region H 320°C Final Cure Hold. Again, the criterion for full cure at this hold temperature is that the magnitude of $((d\varepsilon''/dt)/\varepsilon'')$ at 25 kHz be less than 3.0×10^{-5} for four consecutive FDEMS data acquisition cycles. Region I is part cool down and Region J is expert system shutdown. Important points to note are the extremely short dwell time at the 200°C Imidization Hold, the 280°C instrumentally determined secondary hold temperature for part consolidation, and the total cure time of 380 minutes.

Figure 5.5 shows the intelligent cure of a PMR-15 prepreg sample from the same batch as the cure seen in Figure 5.4 (ICI FIberite Lot# 10645S) after aging for 6 months under freezer conditions. Note the variations in the holds seen in Regions B, D and H when compared with Figure 5.4. Despite the fact that they differ significantly, the overall cure times for each run are essentially the same. Of course, other questions such as the effects of these different cure profiles on the final composite mechanical properties, for example, are unknown without formal studies of the issues. However, the possibility exists that further investigations of the temperature dependence of the nadic versus the BTDE imidization reactions (work on the imidization reaction mechanism and kinetics is ongoing in our laboratory at William & Mary) could be incorporated into a higher level intelligent cure control system which would selectively control this and other similar effects on the final composite properties.

Figure 5.6 shows the intrabatch variability in the expert cure of the six month freezer aged ICI Fiberite Lot# 10645S PMR-15/carbon prepreg. Again, the most dramatic differences between the fabrication runs lie in the three holds seen in Regions B, D and H. Note that the Region B Imidization Onset hold near 80°C is still short; the Region D Imidization Hold at 200°C, however, has now decreased from 60 minutes in Figure 5.5 to only 5 minutes in Figure 5.6. Since these runs were done within 72 hours of each other, the most satisfactory explanation for this difference is that significant intrabatch variability exists within the prepreg samples in addition to the already accepted interbatch variability. Another important point to note is the unusual noise in the FDEMS signal in Regions F and H. While it had no effect on the control of the cure during the preset 30 minute hold at the secondary minimum viscosity temperature, it did have a negative effect on the determination of the end of cure. Because of the noise in the FDEMS signal, the expert system was never able to determine end of cure based on the criterion that the magnitude of $((d\varepsilon''/dt)/\varepsilon'')$ at 25 kHz be

less than 3.0 x 10^{-5} for four consecutive FDEMS data acquisition cycles. Instead the intelligent, automated PMR-15 cure control system terminated the cure process after a maximum allowed 320°C hold time of two hours. One possible explanation for this noise is that carbon fibers from the prepreg migrated to the sensor active surface with the flowing PMR-15 resin and that as the material approached full cure the electrically conductive fibrils became the source of the electrical noise in the FDEMS signal. A second possibility is that as the composite approached full cure, it partially lifted off the sensor active side, thus leading to the unusually noisy FDEMS signal.

Figure 5.7 is the FDEMS output for the sensor-model expert system's control of a sample of ICI Fiberite Lot# 10502S PMR-15/carbon prepreg after aging for seven months under freezer conditions. Region A shows FDEMS sensor wet out as determined by $Log(\varepsilon''*\omega)>7.4$ for two consecutive readings during the slow 2°C per minute ramp to 83°C. Region B requires that the temperature be greater than 80°C and that the magnitude of $((d\varepsilon''/dt)/\varepsilon'')$ at 25 kHz be less than 3.0 x 10^{-4} for four consecutive FDEMS data acquisition cycles. These criteria are met after 15 minutes in the Region B Imidization Onset hold. The Region C Imidization Ramp shows a four and one half decade decrease in the ionic mobility of the PMR-15 (as shown by the overlapping ε'' low frequency lines) and thus a corresponding increase in viscosity as the imidization reaction process proceeds. By the time the standard imidization hold temperature of 200°C is reached, the $Log(\varepsilon''*\omega)$ signal for this aged PMR-15 prepreg has already met the criterion that the magnitude of $((d\varepsilon''/dt)/\varepsilon'')$ at 25 kHz be less than 3.0 x 10^{-4} for four consecutive FDEMS data acquisition cycles (Region D). Thus the expert system continued heating the curing composite part on an almost continuous ramp (Region E) to the high temperature part consolidation hold as determined by a 'folding over' (*i.e.*, a decrease in the rate of change) of the FDEMS signal. Region F shows the changing dielectric signal approaching a constant value at the end of a thirty minute preset hold at this minimum viscosity temperature. This hold allows for evolution of residual volatiles resulting from the reaction as well as part consolidation to eliminate voids before heating to the final crosslinking cure temperature. Region G is a slow 2°C per minute ramp to the final crosslinking cure temperature of 320°C. Region H, the Final Cure Hold at 320°C, successfully ended after 75 minutes when the magnitude change in ε'' signal $((d\varepsilon''/dt)/\varepsilon'')$ at 25 kHz was less than 3.0 x 10^{-5} for four consecutive FDEMS data acquisition cycles. Regions I and J are system cool down and shut down, respectively. A comparison of the total

processing time for this intelligently controlled cure of seven month aged PMR-15/carbon prepreg with the preset time-temperature cure of fresh PMR-15 prepreg seen in Figure 5.1 shows that the expert cure required 200 minutes less time than the recipe cure. Thus it can be seen that curing 'non-ideal' material using the standard preset time-temperature recipe approach often leads to an inefficient and expensive composite cure process.

Figure 5.8 shows what happens when the PMR-15 matrix resin never becomes fluid enough to full wet out the FDEMS sensor. Obviously, if the sensor(s) which is/are being used to monitor in situ the real time state of the curing material never wets out, it will be impossible to intelligently control the cure process. Indeed, if the FDEMS microsensor does not wet out, it is also likely that the prepreg layers of the composite part also do not fully wet out. Thus it would be pointless to continue with an expensive and time consuming cure process.

In Figure 5.8 we see an attempt to cure a PMR-15/carbon prepreg panel using ICI Fiberite Lot# 11769S (the same material seen in Figures 5.2 and 5.3) after aging for three months under freezer conditions. Because the material did not wet out the FDEMS sensor before reaching a part temperature of 150°C (Region A), the intelligent, automated cure control system terminated the cure process after reaching the process abort value required in Region J. One should remember that processable PMR-15 prepreg should *always* be expected to fully wet out the FDEMS sensor.

Clearly PMR-15 prepreg exhibits significant variations in processing due to the effects of batch variation, aging, and material handling history. These factors, as well as universal concerns regarding heat transfer characteristics of thicker and/or more geometrically complex parts and in different molds/tooling, make the use of an expert cure control system highly desirable. An FDEMS/QPAL intelligent, automated sensor-model cure control system responds to these variations and improves the consistency of processing, resulting in product consistency and a potentially significant cost savings in processing times.

Experimental Trends with Respect to Prepreg Age

As prepreg ages, a variety of processes occur. Included in this list are solvent loss, premature reaction advancement and moisture adsorption. Due to the initial intrabatch and interbatch variability within the prepreg, the effects of solvent loss over time is difficult to trace in the above data. Another factor helping to obscure the results of this phenomenon is the differential exposure of prepreg plies to the environment. Put simply, some plies are on the outside in direct contact with the storage environment while others are on the inside of the stack or roll. Similarly, the effects of accidental moisture adsorption during storage are difficult to quantify.

One area where we should be able to observe a trend with respect to prepreg age is in the effects premature reaction advancement over time has on the optimized cure profile. An examination of the length of time required to complete the 200°C imidization hold shows positive correlation with the known age of the prepreg under freezer storage conditions. In general, the length of time at 200°C in Figures 5.2 through 5.7 decreases with increasing prepreg age with the exception of Figure 5.5. (The longer hold time seen in Figure 5.5 is most likely a result of the normal variations within prepreg samples.) Thus one may hypothesize that, due to advancement of the imidization process during storage, the imidization reaction in aged samples is much closer to completion by the time the Imidization Hold is actually reached, resulting in significantly decreased Imidization Hold times when compared to the fresh PMR-15 prepreg material.

A second point during the cure cycle which shows positive correlation with the age of the prepreg is the minimum viscosity temperature for part consolidation determined by the expert system for each optimized cure. Recall that the expert system determines the temperature at which the matrix resin reaches a secondary viscosity minimum (the first viscosity minimum being the point of maximum flow seen in Region A) by finding the point at which the FDEMS signal shows a decreasing ionic conductivity (*i.e.*, the FDEMS signal 'folds over'). This then becomes the hold temperature for the Region F CrossLinking Hold for part consolidation previously discussed. For the intelligently cured fresh PMR-15/carbon prepreg, the CrossLinking Min Viscosity temperature ranges from 235°C to 250°C (Figures 5.2 and 5.3). For Figure 5.4, the sample aged for four months under freezer storage conditions, this minimum viscosity temperature rises to 280°C. In Figures 5.5 and 5.6, the

FDEMS data for the samples aged six months under freezer storage conditions, this temperature remains constant at approximately 280°C. Finally, Figure 5.7 shows the PMR-15 prepreg which has aged for seven months under freezer storage conditions reaching is viscosity minimum at 290°C. Thus we see that the prepreg age does affect the overall cure process although the exact mechanisms leading to these variations are not currently understood. Despite these facts, the use of the FDEMS/QPAL intelligent, automated sensor-model cure control system can optimize the PMR-15 cure process.

Although one could design endless tests to quantify the many permutations in PMR-15 (or any other material for that matter), the bottom line is this: in real life, significant variations may exist between materials which are nominally identical. However, through the use of the FDEMS/QPAL intelligent, automated sensor-model cure composite cure control system that has been the focus of this work, we can optimize the cure of the majority of these materials regardless of their inherent variability.

NOTES FOR CHAPTER 5

1. Kranbuehl, D., T. Prettyman, K. Robillard, J. Smith, A. Nicoletti, S. Hart, A. Loos and J. Koury, *Int. SAMPE Symp. Ser. 36,* 1161-1171 (1991).

2. Hart, S., D. Kranbuehl, A. Loos, B. Hinds and J. Koury, Int. *SAMPE Symp. Ser. 37,* 224-230 (1992).

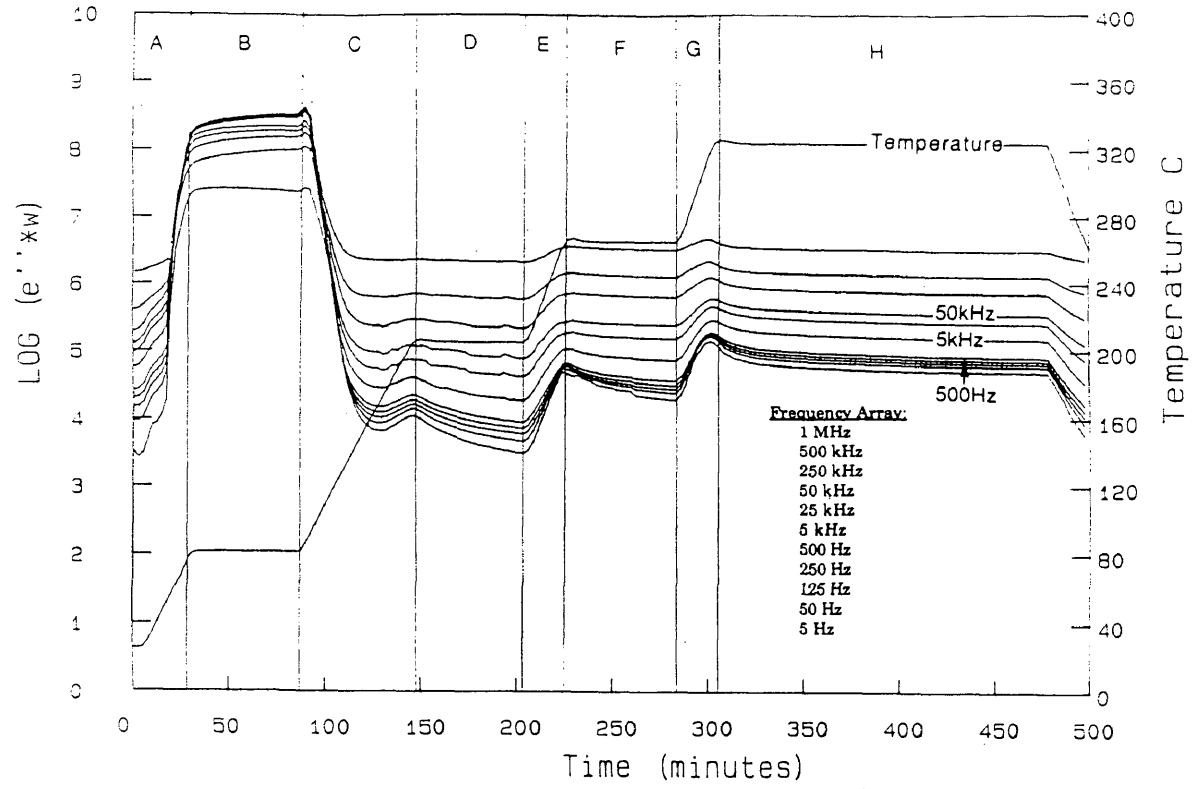

Figure 5.1: Values of temperature and ε'' multiplied by frequency for a "standard" cure of fresh PMR-15 prepreg. (Lot #10502S)

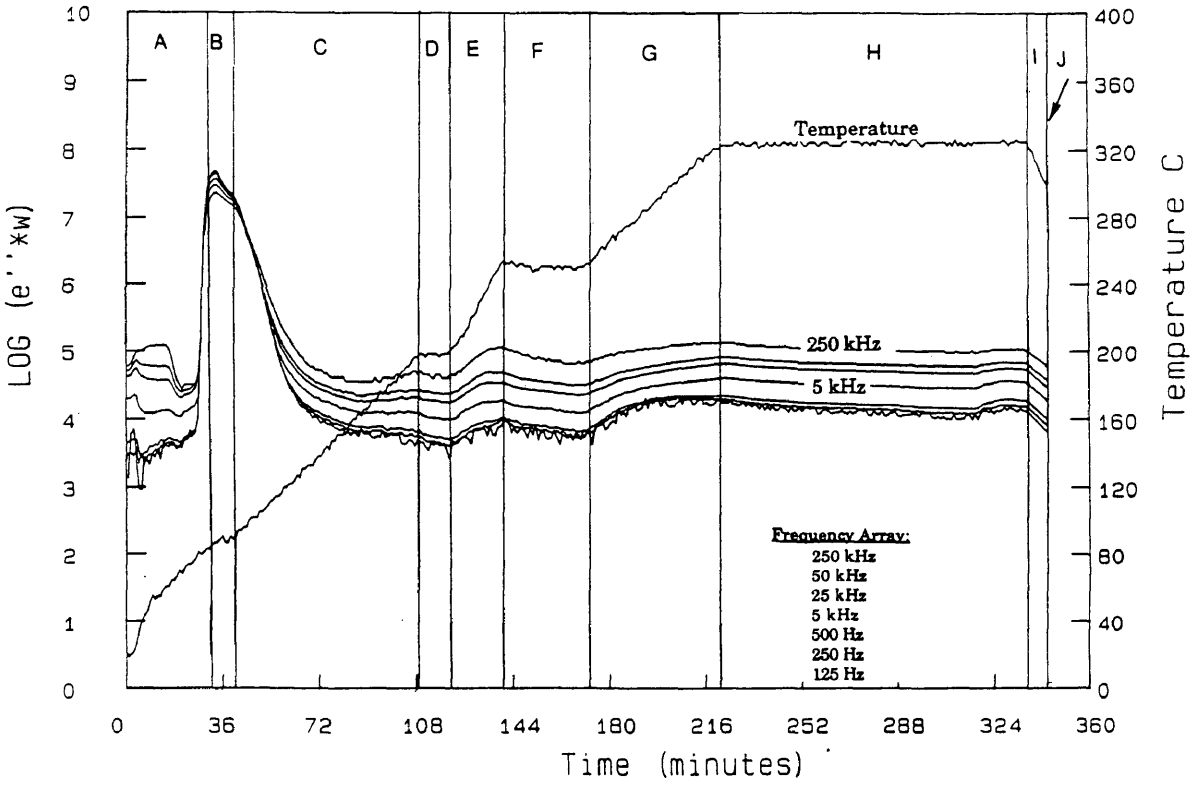

Figure 5.2: Values of temperature and ε'' multiplied by frequency for an intelligent, automated cure of fresh PMR-15 prepreg. (Lot #11769S)

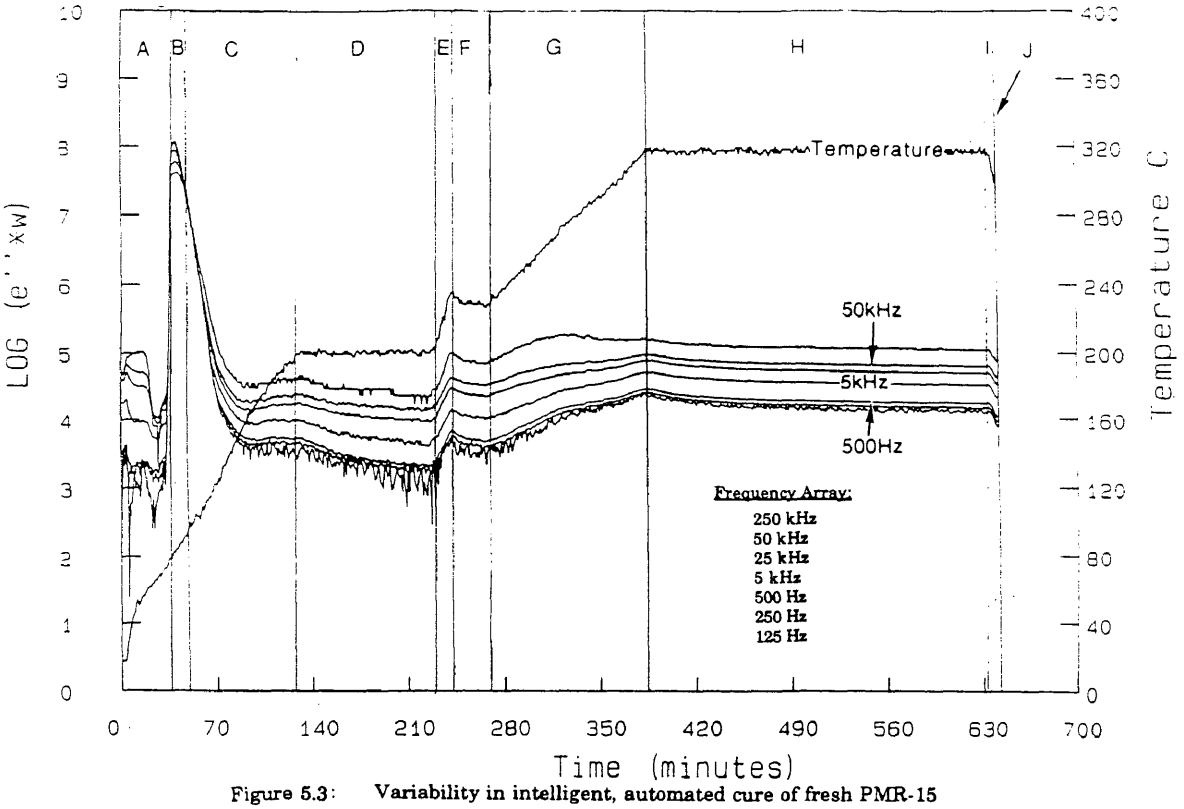

Figure 5.3: Variability in intelligent, automated cure of fresh PMR-15 prepreg (Lot #11769S)

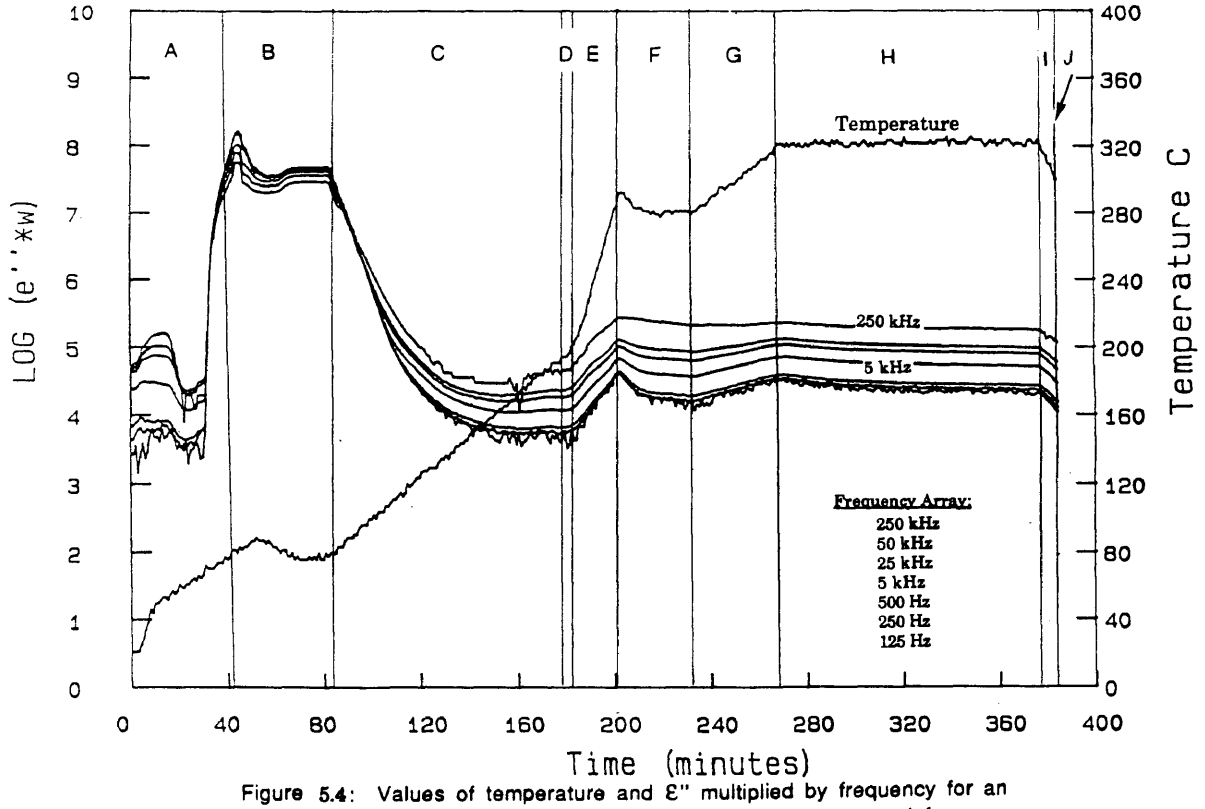

Figure 5.4: Values of temperature and ε" multiplied by frequency for an intelligent, automated cure of PMR-15 prepreg aged four months under freezer conditions. (Lot #10645S)

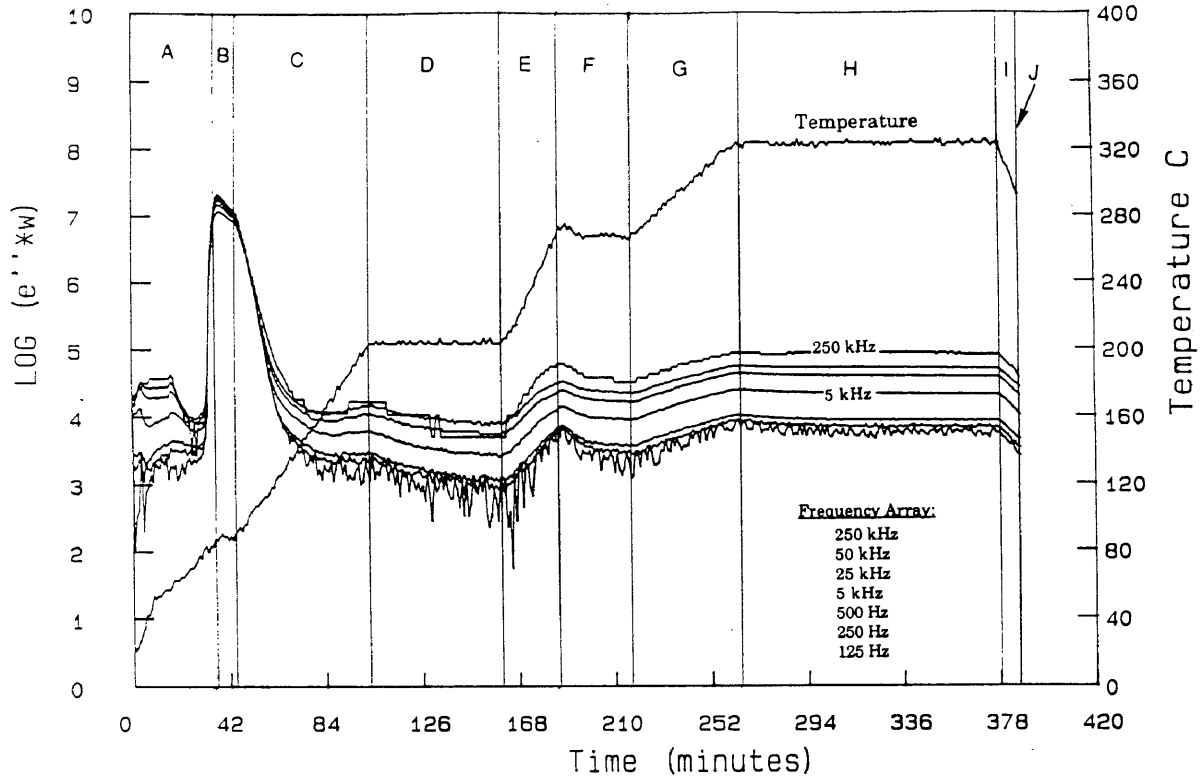

Figure 5.5: Values of temperature and ε'' multiplied by frequency for an intelligent, automated cure of PMR-15 prepreg aged six months under freezer conditions. (Lot #10645S)

Figure 5.6: **Variability in intelligent, automated cure of PMR-15 prepreg aged 6 months under freezer conditions (Lot #10645S)**

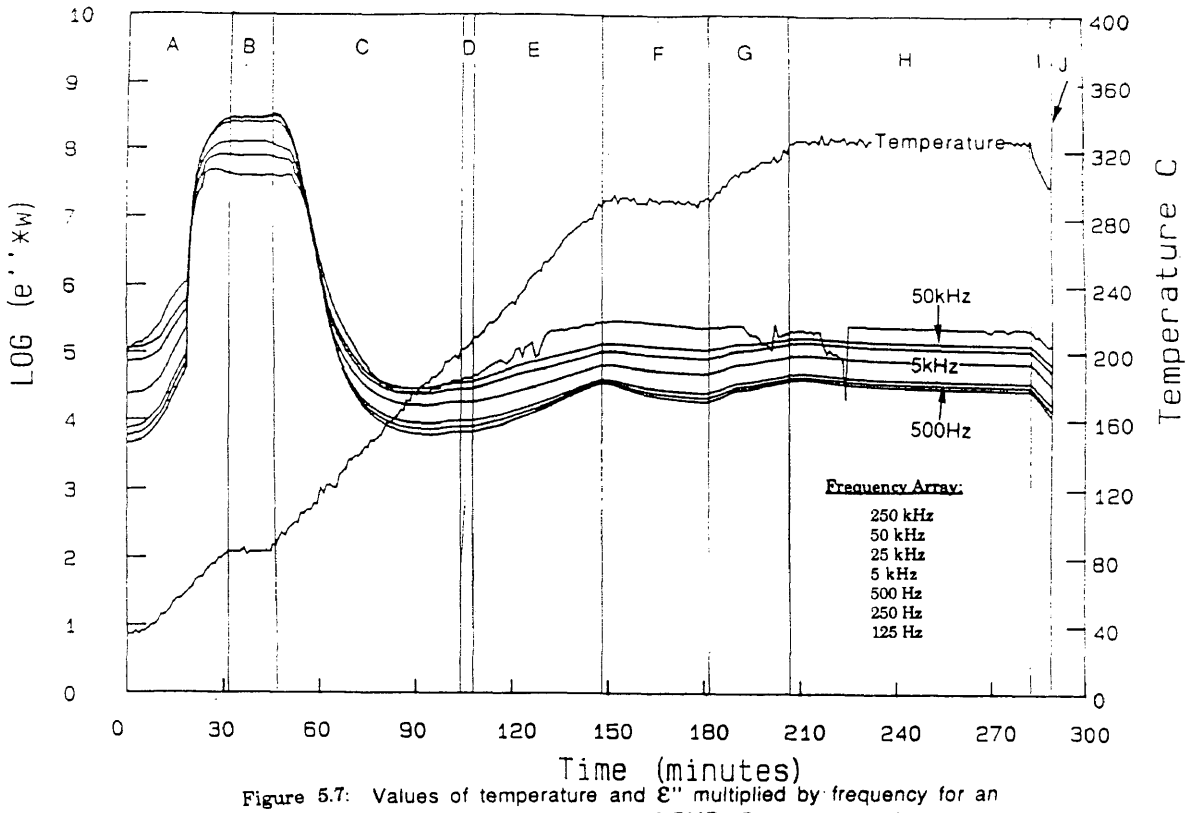

Figure 5.7: Values of temperature and ε'' multiplied by frequency for an intelligent, automated cure of PMR-15 prepreg aged seven months under freezer conditions. (Lot #10502S)

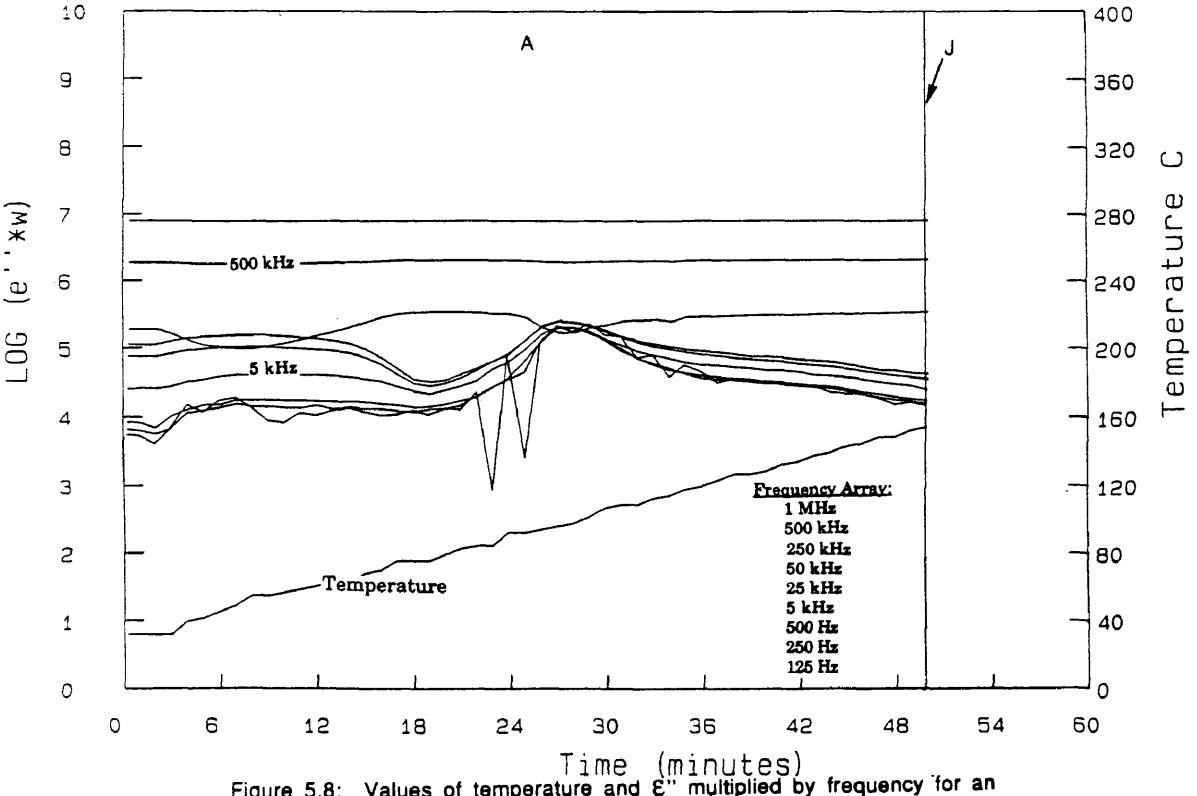

Figure 5.8: Values of temperature and ε'' multiplied by frequency for an intelligent, automated controlled shutdown of the cure of PMR-15 prepreg aged three months under freezer conditions. (Lot #11769S)

CHAPTER 6

CONCLUSIONS

An intelligent, automated composite cure control system has been developed and used to control the cure of PMR-15/graphite prepreg in the autoclave and the thermal press. The expert system consists of the synergistic coupling of the user modified Dek Dyne, Inc. in situ Frequency Dependent Electromagnetic Sensing system and the Abrams Qualitative Process Automation Language and resin-specific knowledge base. The expert system uses predictions from the Loos thermo-chemical processing model for PMR-15 to help define the 'ideal cure' process.

This intelligent cure control system has been used to fabricate a series of flat, high performance PMR-15/graphite composite panels that followed optimized cure profiles based on real time in situ FDEMS sensor output. QPAL provides a flexible environment for making decisions regarding the control process.

The expert system has shown that the optimum cure profile can vary significantly as a function of resin/prepreg batch, moisture adsorption, out time in part precure preparation, and incidental or intentional advancements of the degree of cure. The FDEMS-QPAL sensor-model intelligent, automated cure control system responds in real time to these variations, improving the consistency of the material's processing properties, often with a significant cost savings in processing time.

SUGGESTIONS FOR FURTHER WORK

While the development of expert systems for composite cure control has come a long way in the last decade, much remains to be done to take greater advantage of this powerful approach to composite fabrication. The FDEMS-QPAL sensor-model intelligent, automated cure control system developed for this project has many possibilities for future development. With respect to PMR-15, the scale up to large part fabrication for further evaluation and subsequent use is a short term goal. A mid term goal might take advantage of continuing investigations into the precise monomeric/oligomeric/polymeric reaction mechanisms, providing new information regarding the selective control of the PMR-15 imidization and crosslinking reactions. This information could then be incorporated into future QPAL knowledge bases. One example of the benefit of this advancement would lie in possibly being able to preferentially control the temperature dependent growth or termination of long chains prior to crosslinking the material,thus 'toughening' the composite. A second set of mid range goals should take advantage of the flexibility of this expert system and expand its application to a wide variety of matrix resins and processes.

A long term goal would be the development and hardening of the FDEMS-QPAL sensor-model intelligent, automated cure control system for use in the manufacturing sector. Given the emphasis on maximizing profits, the use of an expert cure control system that minimizes both part loss and processing time over the long haul should be highly desirable.

APPENDIX

PMR-15 KNOWLEDGE BASE

```
( ************************************************************************** )
( *                                                                        * )
( * PMR 15 Knowledge Base.                                                 * )
( * Created at William and Mary College                                    * )
( * By: Sean Hart and Bruce Hinds                                          * )
( * Jan 14-17, 1991                                                        * )
( * Revised May 24-August 9,1991 by S. Hart                                * )
( * Revised 2 SEP 1991 by S. Hart                                         * )
( * Revised November 1991-October 1992 by S. Hart                         * )
( * Revised June 1993 by S. Hart                                          * )
( *                                                                        * )
( * Sensors, Controllers, and Parts section originally written by:         * )
( * Universal Technology Corporation                                       * )
( * 4031 Col Glenn Hwy                                                    * )
( * Dayton, Ohio  45431                                                   * )
( * Created By: Frances Abrams, Simon Insley, Rick Matejka, Dick Warnock   * )
( * Creation Date:  17 Oct 88                                             * )
( * Completion Date:                                                      * )
( * Revision Dates:     26 JUL 89, 20 SEP 89                              * )
( *                                                                        * )
( ************************************************************************** )

\ This Knowledge Base is written for the cure of simple thin composite parts with
\ PMR-15 polyimide resin.  This knowledge base does not take into consideration part
\ geometry, thus part thickness is not accounted for.  Rather we are concerned with
\ the state of the resin during various stages of the cure.
\ The cure of PMR-15 can be catagorized into three major stages: 1) solvent elution and
\ part consolidation 2) Imidization 3) Cross linking.  In this KB we have states\
                                                                        associated
\ with each of the major stages.  For the the sovent elution and part consolidation the
\ following states are associated: Pre-Imidization Max Flow, and Imidization Onset.
\ For the Imidization stage the Imidization states is associated.  For the cross\
                                                                            linking
\ stage the associated states are: CrossLinking-Min-Viscosity, CrossLinking Hold, and
\ Final Cure Ramp and Hold.

\ This simple Knowledge base uses only ONE FDEMS sensor to be placed in the middle of\
                                                                        the part


\ 3/16/92 by SMH at W&M:
\ The changes made in the setting of hold temps in STATE: Imidization Onset and STATE:\
                                                            Crosslinking Hold so
\ that the outtemp Heat-Ctrl value is the last integer part-1-mid-temp value plus 5C\
                                                        should ensure that the part
\ does not cool significantly as may happen if set the outtemp to the actual part temp\
                                                            which usually lags
\ behind the outtemp to the Heat-Ctrl significantly in the press.  When running in an\
                                                        autoclave where there
\ is not as great a temp lag problem, one would want to return these sets to the value\
                                                            of Part-1-Mid-Temp 0
\ as the hold is entered.

Start KnowledgeBase

System( UpdateTime 60 )
SetScale( 510 )
2 Places


( ************************************************************************** )
( ************************************************************************** )
( *                  PMR-15 Composite Cure Control Knowledge Base          * )
( ************************************************************************** )
```

```
( ************************************************************************** )
( *                                Sensors                                 * )
( ************************************************************************** )
( ************************************************************************** )
( ************************************************************************** )

\  The autoclave controller sensors are INACTIVE for KB use at William & Mary with the\
                                                          small Carver thermal press.
\  SENSOR: Autoclave-Pres


\   %FullScale  IS 100E0
\   Rate        FUNCTION:   \ ***** The rate is divided by time to give true rate as\
                                                                  with accel 7-5-91
\                   Result := { ( Magnitude( MYSELF 0 ) - Magnitude( MYSELF 1 ) ) /\
                                                                SampleTime( MYSELF )
\                              }
\                   ENDFUNCTION

\   Acceleration    FUNCTION:
\                       Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                              SampleTime( MYSELF ) }
\                       ENDFUNCTION

\  *** This function initializes serial communications and would be used in real runs

\   SensorInit  FUNCTION:
\       BAUD( Modem 9600 )
\       DATABITS( Modem 8 )
\       STOPBITS( Modem 1 )
\       BUFFERSIZE( Modem 255 )
\       RESET( Modem )
\       ENDFUNCTION

\ *** the actual channel input from the IBM to be used in a real run
\   Channel     FUNCTION:
\                       READ( modem )
\                       Result := { GET( modem ) }
\                       Write( modem " pressure " )
\               ENDFUNCTION


\   EndSensor



\ SENSOR: Autoclave-Temp
 SENSOR: SmallPress-Temp
    %FullScale  IS 500E0
    #POINTS IS 9
    Rate        FUNCTION:
                    Result := { ( Magnitude( MYSELF 0 ) - Magnitude( MYSELF 1 ) ) /\
                                                            SampleTime( MYSELF )
                              }
                    ENDFUNCTION
    Acceleration    FUNCTION:
                        Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                          SampleTime( MYSELF ) }
                        ENDFUNCTION

    SensorInit  FUNCTION:
        BAUD( Modem 9600 )
        DATABITS( Modem 8 )
        STOPBITS( Modem 1 )
```

```
        ENDFUNCTION

    Channel     FUNCTION:
                        READ( modem )
                        Result := { GET( modem ) }
                        Write( modem " ATemp " )     \ ++The sensor definitions use\
                                                              dummy strings to complete
                                                     \ ++handshaking of communications\
                                                                with FDEMS software.
                ENDFUNCTION

    EndSensor


SENSOR:  Part-1-Mid-Temp
    %FullScale  IS 500e0       \ ++++6-19-91++++500E0
    Rate        FUNCTION:
                    Result := { ( Magnitude( MYSELF 0 ) - Magnitude( MYSELF 1 ) ) /\
                                                                  SampleTime( MYSELF )
                                }
                ENDFUNCTION
    Acceleration    FUNCTION:
                        Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                                SampleTime( MYSELF ) }
                        ENDFUNCTION
    Channel     FUNCTION:
                        READ( modem )
                        Result := { GET( modem ) }
                        Write( modem " dude " )
                ENDFUNCTION


    EndSensor


SENSOR: Part-1-Top-Temp
    %FullScale  IS 500E0

    Rate        FUNCTION:
                    Result := { ( Magnitude( MYSELF 0 ) - Magnitude( MYSELF 1 ) ) /\
                                                                  SampleTime( MYSELF )
                                }
                ENDFUNCTION
    Acceleration    FUNCTION:
                        Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                                SampleTime( MYSELF ) }
                        ENDFUNCTION
\   Channel     FUNCTION:
\                       Result := { SetPoint?( Heat-Ctrl ) }    \ ****This is for\
                                                                          simulation
\               ENDFUNCTION

    Channel     FUNCTION:
                        READ( modem )
                        Result := { GET( modem ) }
                        Write( modem " dude " )
                ENDFUNCTION


    EndSensor

\ ***** The FDEMS sensors would probably read a log value.  The rates have generally\
                                                                        been normalized
\ ***** by dividing by the value of the sensor.
```

```
SENSOR: Part-1-E2f4
    VARIABLE: e2 := 0e0
    %FullScale  IS 10E0
    #POINTS IS 9

    Rate    FUNCTION:
            Result := { ( ALOG( Magnitude( MYSELF 0 ) ) - ALOG( Magnitude( MYSELF 1 ) )\
                                                                                ) /
                        ( ALOG( Magnitude( MYSELF 0 ) ) ) / SampleTime( MYSELF )  }    \

                        \ this is dE2/E2/dt

                ENDFUNCTION
    Acceleration    FUNCTION:
                        Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                                SampleTime( MYSELF ) }
                        ENDFUNCTION

    Channel     FUNCTION:
                        READ( modem )
                        e2 := { GET( modem ) }
                        e2 := { e2 MAX 1e-8 }   \ ** won't let e2 be < 1e-8 for\
                                                                log(zero) error
                        Result := { LOG( e2 ) }
                        Write( modem " dude " )
                ENDFUNCTION

    EndSensor

SENSOR: Part-1-E2f5
    VARIABLE: e2 := 0e0
    %FullScale  IS 10E0
    #POINTS IS 9

    Rate    FUNCTION:
            Result := { ( ALOG( Magnitude( MYSELF 0 ) ) - ALOG( Magnitude( MYSELF 1 ) )\
                                                                                ) /
                        ( ALOG( Magnitude( MYSELF 0 ) ) ) / SampleTime( MYSELF )  }    \

                        \ this is dE2/E2/dt

                ENDFUNCTION
    Acceleration    FUNCTION:
                        Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                                SampleTime( MYSELF ) }
                        ENDFUNCTION

    Channel     FUNCTION:
                        READ( modem )
                        e2 := { GET( modem ) }
                        e2 := { e2 MAX 1e-8 }   \ ** won't let e2 be < 1e-8 for\
                                                                log(zero) error
                        Result := { LOG( e2 ) }
                        Write( modem " dude " )
                ENDFUNCTION

    EndSensor

SENSOR: Part-1-E2f6
    VARIABLE: e2 := 0e0
    %FullScale  IS 10E0
    #POINTS IS 9

    Rate    FUNCTION:
```

```
                ENDFUNCTION
    Acceleration    FUNCTION:
                        Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                                SampleTime( MYSELF ) }
                        ENDFUNCTION

    Channel     FUNCTION:
                        READ( modem )
                        e2 := { GET( modem ) }
                        e2 := { e2 MAX 1e-8 }   \ ** won't let e2 be < 1e-8 for\
                                                                log(zero) error
                        Result := { LOG( e2 ) }
                        Write( modem " dude " )
                ENDFUNCTION

    EndSensor

\ **** The e**w sensor values are calcualted from the e* sensor values from above (done\
                                                                        in log)

SENSOR: Part-1-E2wf4        \ ***checking of KB on 6-13-92 and realized that these e2*w\
  calcs had never been adjusted for the 9 freq array used since AUG 91 changes made in\
                                                the FDEMS program at the Phillips Lab.
    %FullScale  IS 10E0     \ ***I have elected to alter the KB so that freq4=5000Hz,\
                                        5=25kHz and 6=50kHz as in the FDEMS program.
    #POINTS IS 9

    Rate    FUNCTION:
            Result := { ( ALOG( Magnitude( MYSELF 0 ) ) - ALOG( Magnitude( MYSELF 1 ) )\
                                                                                ) /
                        ( ALOG( Magnitude( MYSELF 0 ) ) ) / SampleTime( MYSELF ) }
                        \ this is dE2/E2/dt

                ENDFUNCTION
    Acceleration    FUNCTION:
                        Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                                SampleTime( MYSELF ) }
                        ENDFUNCTION
    Channel         FUNCTION:
                        Result := { Magnitude( Part-1-E2f4 0 ) + LOG( 5000e0 ) + LOG(\
                                        6.283185307e0 ) } \ gives e**w where w=5khz
                    ENDFUNCTION

    EndSensor


SENSOR: Part-1-E2wf5
    %FullScale  IS 10E0
    #POINTS IS 9

    Rate    FUNCTION:
            Result := { ( ALOG( Magnitude( MYSELF 0 ) ) - ALOG( Magnitude( MYSELF 1 ) )\
                                                                                ) /
                        ( ALOG( Magnitude( MYSELF 0 ) ) ) / SampleTime( MYSELF ) }
                        \ this is dE2w/E2w/dt

                    ENDFUNCTION
    Acceleration    FUNCTION:
                        Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                                SampleTime( MYSELF ) }
                        ENDFUNCTION
    Channel         FUNCTION:
                        Result := { Magnitude( Part-1-E2f5 0 ) + LOG( 25e3 ) + LOG(\
                                        6.283185307e0 ) } \ gives e**w where w=25Khz
```

```
    Rate    FUNCTION:
            Result := { ( ALOG( Magnitude( MYSELF 0 ) ) - ALOG( Magnitude( MYSELF 1 ) )\
                                                                                    ) /
                        ( ALOG( Magnitude( MYSELF 0 ) ) ) / SampleTime( MYSELF ) }
                        \ this is dE2/E2/dt

                ENDFUNCTION
    Acceleration    FUNCTION:
                        Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                            SampleTime( MYSELF ) }
                        ENDFUNCTION
    Channel         FUNCTION:
                        Result := { Magnitude( Part-1-E2f6 0 ) + LOG( 5e4 ) + LOG(\
                                        6.283185307e0 ) } \ gives e**w where w=50Khz


                    ENDFUNCTION

    EndSensor

\ *** The e' values are to be read from the IBM

SENSOR: Part-1-E1f4
    %FullScale  IS 10E0
    #POINTS IS 9
    VARIABLE: e1 := 0e0

    Rate    FUNCTION:
            Result := { ( ALOG( Magnitude( MYSELF 0 ) ) - ALOG( Magnitude( MYSELF 1 ) )\
                                                                                    ) /
                        ( ALOG( Magnitude( MYSELF 0 ) ) ) / SampleTime( MYSELF ) }
                        \ this is dE1/E1/dt

                ENDFUNCTION
    Acceleration    FUNCTION:
                        Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                            SampleTime( MYSELF ) }
                        ENDFUNCTION

    Channel     FUNCTION:
                        READ( modem )
                        e1 := { GET( modem ) }
                        e1 := { e1 MAX 1e-8 }   \ ** won't let e1 be < 1e-8 for\
                                                                log(zero) error
                        Result := { LOG( e1 ) }
                        Write( modem " dude " )
                ENDFUNCTION

    EndSensor


SENSOR: Part-1-E1f5
    VARIABLE: e1 := 0e0
    %FullScale  IS 10E0
    #POINTS IS 9

    Rate    FUNCTION:
            Result := { ( ALOG( Magnitude( MYSELF 0 ) ) - ALOG( Magnitude( MYSELF 1 ) )\
                                                                                    ) /
                        ( ALOG( Magnitude( MYSELF 0 ) ) ) / SampleTime( MYSELF ) }
                        \ this is dE1/E1/dt

                ENDFUNCTION
```

```
    Channel     FUNCTION:
                        READ( modem )
                        e1 := { GET( modem ) }
                        e1 := { e1 MAX 1e-8 }   \ ** won't let e1 be < 1e-8 for\
                                                                    log(zero) error
                        Result := { LOG( e1 ) }
                        Write( modem " dude " )
                ENDFUNCTION

    EndSensor



SENSOR: Part-1-E1f6
    VARIABLE: e1 := 0e0
    %FullScale  IS 10E0
    #POINTS IS 9

    Rate    FUNCTION:
            Result := { ( ALOG( Magnitude( MYSELF 0 ) ) - ALOG( Magnitude( MYSELF 1 ) )\
                                                                                ) /
                        ( ALOG( Magnitude( MYSELF 0 ) ) ) / SampleTime( MYSELF ) }
                        \ this is dE1/E1/dt

                ENDFUNCTION
    Acceleration    FUNCTION:
                        Result := { ( Rate( MYSELF 0 ) - Rate( MYSELF 1 ) ) /\
                                                            SampleTime( MYSELF ) }
                        ENDFUNCTION

    Channel     FUNCTION:
                        READ( modem )
                        e1 := { GET( modem ) }
                        e1 := { e1 MAX 1e-8 }   \ ** won't let e1 be < 1e-8 for\
                                                                    log(zero) error
                        Result := { LOG( e1 ) }
                        Write( modem " dude " )
                ENDFUNCTION

    EndSensor

( ************************************************************************** )
( ************************************************************************** )
( ************************************************************************** )
( *                              Controllers                               * )
( ************************************************************************** )
( ************************************************************************** )
( ************************************************************************** )

\ *** It is important to note that these sensors have same names as Susan Ferrer's KB.

 CONTROLLER: Heat-Ctrl
    Priority {{ INCREASE Lowest STABILIZE DECREASE Highest }}
    Initial     IS 60E0     \ new start value =60C b/c no flow until >70C 3/12/92 smh
    MaxValue    IS 450E0
    MinValue    IS 70E0     \ was 80
    %FullScale  IS 500E0
    Feedback    IS SmallPress-Temp
\   Feedback    IS Autoclave-Temp   \  **Change this to ACTIVE and line above to\
                                                        INACTIVE for autoclave runs.
    LogStatus   IS FALSE
    Write( modem " HEllO " )
    VARIABLE: SP-Int
```

```
        Channel FUNCTION:
            SP-Int := Integer( Result )
            SP := String( SP-Int )
            Write( modem SP )
        ENDFUNCTION
    EndController


\ CONTROLLER: Pressure-Ctrl
\   Priority {{ INCREASE Lowest STABILIZE DECREASE Highest }}
\   Increment   IS  5E0
\   Initial     IS  0E0
\   MaxValue    IS  100E0
\   MinValue    IS  0E0
\   %FullScale  IS  100E0
\   Feedback    IS Autoclave-Pres
\   VARIABLE: SP-Int
\   VARIABLE: SP
\       Channel FUNCTION:
\           SP-Int := Integer( Result )
\           SP := String( SP-Int )
\           Write( modem SP )
\       ENDFUNCTION
\   EndController



( ********************************************************************************* )
( ********************************************************************************* )
( ********************************************************************************* )
( *                                  Parts                                        * )
( ********************************************************************************* )
( ********************************************************************************* )
( ********************************************************************************* )


PART:   Part1
    PartNumber IS " Part 1"
    Sensors {{
            Part-1-Mid-Temp
            Part-1-Top-Temp
            Part-1-E2f5
            Part-1-E2wf5
            }}
    EndPart

PART:    SmallPress
\ PART:      Autoclave
    RunNumber   IS " PMR"
    PartNumber IS " SmallPress"
\   PartNumber IS " Autoclave"
    Sensors {{
            SmallPress-Temp
\           Autoclave-Temp
\           Autoclave-Pres
            }}
    EndPart

( ********************************************************************************* )
( ********************************************************************************* )
```

```
( ************************************************************************ )
( *                          States and StateTypes                       * )
( ************************************************************************ )
( ************************************************************************ )
( ************************************************************************ )



( ************************************************************************ )
( *                          STATE   Pre-Imidization Max Flow            * )
( ************************************************************************ )

\ **** This state is to get the viscosity min before imidization sets in.  We have\
                                                                        found
\ **** that 80C is a good temperature.  This state is really a temperature ramp.  A\
                                                                      further
\ ** consideration is that sometimes 80C is not hot enough to get the resin to flow\
                                                                     onto the
\ ** sensor, hence we're willing to heat up to 90C until there is FDEMS sensor\
                                                                   response.

\ **2/3/92**It has been noted for some time that instead of assuming an "ideal" temp\
                                                                 for max flow that
\ (S.Hart,  the KB should utilize the concept of a decreasing sigma (as is used in the\
                                                                 state Cross-
\  W&M)     Linking-Min-Viscosity) to indicate termination of the initial ramp and the\
                                                                 hold temp for \
\           this state of maximum fluidity.  The decreasing sigma is calculated over 5\
                                                                 historical values.

STATE:  PreImidizationMaxFlow

\   VARIABLE:  HoldTemp1 := 70e0    \ +++(1/20/92,W&M)--lowered from 80C to 75C in case\
                                                          max flow reached before 80C
    VARIABLE:  MaxTemp1 := 90e0 \ 5-22-92**SET TO MAX PART TEMP OF 95C TO LET SENSOR\
                                                 DETERMINE EXCEPT IN THE VERY WORST CASES
                                \ 7-5-92**SET TO MAX PART TEMP OF 90C TO LET SENSOR\
              DETERMINE EXCEPT IN THE VERY WORST CASES b/c I feel that 95C is too high.

\ **2/3/92,S.Hart,W&M**The previous 2 lines were removed since they are not necessary\
                                                        for decreasing sigma criterion.
    VARIABLE:  TimeInterval := 0e0
    VARIABLE:  RampRate := 4e0  \ Deg/min   **use 2e0 for real runs**
                                \ changed to 4C/min on 3/18/92 by smh b/c the press\
          does not heat as rapidly as I would like due to low gain setting of current\
                                                                  calibration
    VARIABLE:  SetPnt := 60e0   \ +++(6/11/91)Must initially have heat set to 30C as in\
                                                                  reality.+++
    VARIABLE:  SetPntINT := 60  \ +++(1/20/92,W&M)--bumped up to 50C to ensure that W&M\
                                                 small press heating platens come on
                                \ 3/11/92 by smh. Changed to 60C b/c I want it to move\
                               thru the initial warming where no flow occurs more quickly
    VARIABLE: DecreasingSigma? := 0e0
    VARIABLE: SlopeMx := 0e0
    VARIABLE: e2/heatRateA := 0e0
    VARIABLE: e2/heatRateB := 0e0
    VARIABLE: e2/heatRateC := 0e0
    VARIABLE: e2/heatRateD := 0e0
    VARIABLE: AbortRun? := 0e0 \ **7-22-92
    VARIABLE: AbortRunTemp := 150e0 \ **7-22-92**This is the maximum temp that the KB\
                                                                 will allow in
                                    \ trying to achieve sensor wetout.  If the sensor\
                                                               has not fully wetout
                                    \ by this temp then we will terminate the run b/c\
```

```
    VARIABLE:  TimeFactor := 1.0e0

\  **The variable time factor is used for simulations reading actual PMR data.  Since\
                                                                                in
\ a simulation the QPAL will cycle every 15 seconds and the data was obtained every 2-3\
                                                                              min
\ there could be problems with temp ramp rates and e" rate criteria.  The time factor\
                                                                            is the
\ ratio of actual data time interval divided by the QPAL time interval.  It is used\
                                                                           whenever
\ a temperature ramp is used or when comparing to FDEMS sensor rates.  A time factor of\
                                                                                1
\ would be used in real runs.

\ **7-22-92**In a 2 ply test of the knowledge base WMFDBAKKB7692, the sensor did not\
                                                                      show full wetout
\ until after the KB had passed into the second episode.  The result is that the KB\
                                                                       proceeded as if
\ this was correct information.  The net result is that the flatteneing associated with\
                                                                       the 2nd episode
\ was taken to be the end of the 200C hold and thus the control system was completely\
                                                                         out of sorts.
\ Thus the changes tagged with this date were made to ensure that if the sensor does\
                                                                       not show wetout
\ before reaching the desired maxtemp of 90C theKB will continue heating the press\
                                                                      until an ultimum
\ maximum temperature of 150C might be reached.  If this AbortTemp part temp is reached\
                                                                        and the sensor
\ has still not wetout, then the KB outtemps 27C to shutdown.
    STATECONDITIONS
        {{
\ **2/3/92,S.Hart,W&M** Added check for frequencies 4 and 6 to this criterion.

            CONDITION: 80CrampCondition
                TRUEIF: {    ( WetOut? = 1e0 ) AND          \ as of 12-07-92 wetout a\
                                                                    subtask check
                             ( DecreasingSigma? = 1e0 ) OR

\ **7-22-92**Added the requirement that before the max temp of 90C can be checked for\
                                                                       the sensor must
\ show full wetout.  Otherwise the system will be allowed to continue ramping to a\
                                                                     maximum part temp
\ of 150C to try and force wetout.  After this point the temp control will be shutdown.
                             ( WetOut? = 1e0 ) AND
                             ( Magnitude( Part-1-Mid-Temp 0 ) > ( MaxTemp1 ) )    \\
                                        3/9/92--added -5C so do not overshoot as much

                         }
            ENDCONDITION,


        }}
\ +++If bottom sensor wet-out has not occurred by the time the 80C hold is reached, the\
                                                                          temperature
\ +++should continue to increase slowly until a hold temp. sufficient to achieve\
                                                                  wet-out is achieved.

    EXCITORS
        {{
            RULE: Excite80Cramp
                IF:      { ( ABS( Magnitude( SmallPress-Temp 0 ) - Magnitude(\
 Part-1-Mid-Temp 0 ) ) < 8e0 ) AND      \ **make this absolute value of the difference\
                                                        between these temps 10/2/91
```

```
\ increasing due to teperature rising thus we must normalize the increase with the
\ increase in temperature by dividing the e* rate by the temp rate.  This is the\
                                                                          variable
\ e2/heatRate.  If the temp rate happens to be zero at the beginning of the state there\
                                                                            will
\ be a division by zero error.  To avoid this the rules NonZeroRate and ZeroRate were\
                                                                          written
\ to assign values to e2/heatRate.  If temp rate is > .003 deg/min then the value will\
                                                                             be
\ calaculated but if it is less than .003 then the dummy value -666 is assigned.  The\
                                                                        HeatRate
\ values are also kept historic with e2/heatRateA being most current and e2/heatRateD\
                                                                       the oldest.
\ 5-28-92, SMH***added in requirement that the value of e**w for freq 5 be greater than\
                                                          or equal to 5e0 on a log scale
\               for each of the determinations below.  It still takes three\
                                                            consecutive decreasing sigma
\               calculations for the DecreasingSigma value to be true and the stage\
                                                                  terminate as per
\               the state rules.

          RULE: NonZeroRate          \ **7-9-91 changed to frequency 5 instead of 3\
                                                                          for sim.
              IF: { Rate( Part-1-Mid-Temp 0 ) > 1e-3 AND
                    ( Magnitude( Part-1-E2wf5 0 ) >= 5.5e0 ) }
              THEN:  \ e2/heatRateD := e2/heatRateC
                     e2/heatRateC := e2/heatRateB
                     e2/heatRateB := e2/heatRateA
                     e2/heatRateA := { Rate( Part-1-E2f5 0 ) / Rate( Part-1-Mid-Temp\
                                                                          0 ) }
          ENDRULE,

          RULE: ZeroRate
              IF: { Rate( Part-1-Mid-Temp 0 ) < 1e-3 AND
                    ( Magnitude( Part-1-E2wf5 0 ) >= 5.5e0 ) }
              THEN:  \ e2/heatRateD := e2/heatRateC
                     e2/heatRateC := e2/heatRateB
                     e2/heatRateB := e2/heatRateA
                     e2/heatRateA :=  -666e0 \ -666 is dummy value
          ENDRULE,

          RULE: FindMaxSlope
              IF: { ( Magnitude( Part-1-E2wf5 0 ) >= 5.5e0 ) AND
                    ( e2/heatRateA < ( e2/heatRateB * 1.2e0 ) ) AND
                    ( e2/heatRateA <> -666e0 ) }
              THEN:  SlopeMx := { SlopeMx MAX e2/heatRateA }
          ENDRULE,

          RULE: FindingDecreasingSigma
              IF: {   ( Magnitude( Part-1-E2wf5 0 ) >= 5.5e0 ) AND
                      ( e2/heatRateA < ( 4.0e0 * SlopeMx ) ) AND      \ changes made\
                                                              to 4.0e0 3/19/92 by smh.
\ 3/19/92--Because the resin is highly fluid at this point and the change in slope as\
 the signal begins to fold over is much greater than that found during the crosslinking\
 min viscosity ramp.                                                              \

                      ( e2/heatRateB < ( 4.0e0 * SlopeMx ) ) AND
                      ( e2/heatRateC < ( 4.0e0 * SlopeMx ) ) AND
                 \    ( e2/heatRateD < ( 0.9e0 * SlopeMx ) ) AND
                      ( e2/heatRateA <> -666e0 )
                                                            }

              THEN:  DecreasingSigma? := 1e0
          ENDRULE,
```

```
                              ( Magnitude( Part-1-E2wf4 2 ) >= 6.4e0 ) AND
                              ( Magnitude( Part-1-E2wf6 2 ) >= 6.4e0 ) AND
                              ( Magnitude( Part-1-E2wf5 3 ) >= 6.4e0 ) AND    \\
 ++(12/07/92,W&M)--that is, >= 6.4 on log scale for 4 consecutive readings to eliminate\
                                                                          random noise
                              ( Magnitude( Part-1-E2wf4 3 ) >= 6.4e0 ) AND
                              ( Magnitude( Part-1-E2wf6 3 ) >= 6.4e0 ) OR

                              ( Magnitude( Part-1-E2wf5 0 ) >= 7.4e0 ) AND
                              ( Magnitude( Part-1-E2wf4 0 ) >= 7.4e0 ) AND
                              ( Magnitude( Part-1-E2wf6 0 ) >= 7.4e0 ) AND
                              ( Magnitude( Part-1-E2wf5 1 ) >= 7.4e0 ) AND    \\
                                              ++(12/07/92,W&M)--for 2 consec. readings
                              ( Magnitude( Part-1-E2wf4 1 ) >= 7.4e0 ) AND
                              ( Magnitude( Part-1-E2wf6 1 ) >= 7.4e0 )
                                                                      }

                THEN:   WetOut? := 1e0
            ENDRULE,


\ **7-22-92**Added this to abort run if sensor does not wet out prior to reaching 150C.
            RULE: AbortRun
                IF: { ( Magnitude( Part-1-Mid-Temp 0 ) > ( AbortRunTemp ) ) }

                THEN:   SetPnt := 17e0                  \ changed to 8 and 10 7/6/92
                    \   SetPntINT := INTEGER( SetPnt )
                        SET( Heat-Ctrl INTEGER( SetPnt ) )
                        SET( Heat-Ctrl 17 )     \ ***emergency abort of run
            ENDRULE,

            RULE: SetAbortRunIndicator
                IF: {  SetPnt = ( 17e0 )  }

                THEN:   AbortRun? := 1e0
                ENDRULE,



        }}
    EndState


( ************************************************************************** )
( *                          STATE   Imidization Onset                     * )
( ************************************************************************** )

\ **** This state really is a hold near 80C.  Here solvent is eluted and there is part
\ **** consolidation.  For simplicity we have the state end when de*/dt is near zero
\ **** (2.5e-4) or when 60min has passed
\ **2/3/92,S.Hart,W&M**Removed minimum temperature requirement since decreasing sigma\
                                                                  from previous state
\                       determines the hold temperature.
\                       Also added the criterion that if the value of Log(e**w) for\
                                                                    desired frequency
\                       drops below a value of 5 for 3 cycles prior to other criteria\
                                                                      being met, that
\                       this state will terminate.  This is because the ICI Fiberite\
                                                                  PMR-15 prepreg rec'd
\                       in 11/91 shows precipitous drops in signal after reaching max\
                                                                        flow while
\                       previous samples did not.  I think that this is simply due to\
                                                                    solvent variation
\                       in the batches resulting from preparation or aging depending\
```

```
    VARIABLE: SetTempInitial := 85e0
\   STATECONDITIONS
\       {{
\           CONDITION: 80CholdCondition
\               TRUEIF: {

                        \ ++now using e''w
                        \   ( ABS( Rate( Part-1-E2wf5 0 ) ) < ( 2.5e-4 * TimeFactor ) )\
                                                              AND   \ ++for criterion
                        \   ( ABS( Rate( Part-1-E2wf5 1 ) ) < ( 2.5e-4 * TimeFactor ) )\
                                                                    AND   \ ++6/19/91
                        \   ( ABS( Rate( Part-1-E2wf5 2 ) ) < ( 2.5e-4 * TimeFactor ) )\
                                                                          AND
                        \   ( ABS( Rate( Part-1-E2wf5 3 ) ) < ( 2.5e-4 * TimeFactor ) )\
                                                                          AND
\ 3/9/92, SMH AT W&M--The above four lines were commented out b/c we no longer want to\
                                                                    check for a flattening
\ out as is sometimes exhibited by the aged prepreg.  Rather, we want to look for a\
                                                                      sharp drop in signal
\ during the hold as is exhibited by fresh prepreg losing lots of solvent.  Therefore\
                                                                        we will require
\ a decreasing rate, that the log values for freqs 4,5,6 be < 8e0, and that the value\
                                                                             of E2wf5
\ decrease by 10% from its maximum value as set at the beginning of the hold for 2\
                                                                           consecutive
\ cycles.  Otherwise we will simply hold for a maximum of 30 minutes.

    STATECONDITIONS
        {{
            CONDITION: 80CholdCondition
                TRUEIF: {                                                          \

                            ( Rate( Part-1-E2wf5 0 ) < 0e0 )    AND     \ **make this a\
                                                                decreasing rate 10/2/91
                            ( Rate( Part-1-E2wf5 3 ) < 0e0 )    AND
\ **2/3/92,S.Hart,W&M**added below criteria.
\ 3/9/92--changed to require that it only be below 8e0 on the Log scale
                            ( Magnitude( Part-1-E2wf5 0 ) < 8e0 ) AND
                            ( Magnitude( Part-1-E2wf4 0 ) < 8e0 ) AND
                            ( Magnitude( Part-1-E2wf6 0 ) < 8e0 ) AND
                            ( Magnitude( Part-1-E2wf5 1 ) < 8e0 ) AND
                            ( Magnitude( Part-1-E2wf4 1 ) < 8e0 ) AND
                            ( Magnitude( Part-1-E2wf6 1 ) < 8e0 ) AND
                            ( Magnitude( Part-1-E2wf5 2 ) < 8e0 ) AND   \\
 ++(1/20/92,W&M)--that is, >= 6.4 on log scale for 3 consecutive readings to eliminate\
                                                                          random noise
                            ( Magnitude( Part-1-E2wf4 2 ) < 8e0 ) AND
                            ( Magnitude( Part-1-E2wf6 2 ) < 8e0 ) AND

                            ( Magnitude( Part-1-E2wf5 0 ) < ( 0.9 * E2wf5Mx ) ) AND
                            ( Magnitude( Part-1-E2wf5 1 ) < ( 0.9 * E2wf5Mx ) ) OR

                            ( HoldTime >= ( 1.8e3 / TimeFactor ) ) \ 1.8e3 sec.is 30\
                                          min max hold time allowed as of 3/9/92 by SMH
                        }
                         ENDCONDITION,

        }}

    EXCITORS
        {{

\ 4/1/92, by SMH---I have moved the HoldTime increment excitor rule back to the\
                                                                          beginning.
```

```
                    Magnitude( Part-1-Mid-Temp 0 ) > 80e0 }
               THEN:  SetTempInitial := { Magnitude( Part-1-Mid-Temp 0 ) + 6e0 }
                    SetPnt := SetTempInitial    \ sets it to the part midtemp plus\
 8C for the hold,7/3/92;++6C allowed 7/5/92++ by smh (so not too hot but will maintain\
                                                          approx. current part temp.)
                  \   SetPntINT := INTEGER( SetPnt )  \ changes so have integer set\
                        point here made 3/10/92 by SMH so do not get system crashes
                    SET( Heat-Ctrl INTEGER( SetPnt ) )

          ENDRULE,

\ ***NoTempGrad.Set80CHold rule makes the small Press temp set point = 85C at the start\
                                                                        of the hold
\     if the part temp is less than 85C when State 1 is complete.--changed 3/12/92 by\
                                                                      SMH,W&M

          RULE: NoTempGrad.Set80CHold
              IF: { HoldTime <= SampleTime( Part-1-Mid-Temp )  AND       \ i.e.,\
                              will only do this when 1st enters hold,3/18/92 by smh
                    Magnitude( Part-1-Mid-Temp 0 ) < 80e0 }
              THEN:  SetPnt := { Magnitude( Part-1-Mid-Temp 0 ) + 6e0 }   \\
             ++changed to this so would be based on actual part temp--7/5/92HoldTemp1
                  \   SetPntINT := INTEGER( SetPnt )  \ same changes made as in\
                                                              NoTempGrad 3/10/92
                    SET( Heat-Ctrl INTEGER( SetPnt ) )

          ENDRULE,

          RULE: DefineE2wf5Mx
              IF: { HoldTime <= SampleTime( Part-1-Mid-Temp ) }

              THEN:  E2wf5Mx := { Magnitude( Part-1-E2wf5 0 ) }

          ENDRULE,


      }}
   EndState



( ************************************************************************** )
( *                          STATE Imidization                             * )
( ************************************************************************** )

\ **** This state is basically a ramp to 200C and a hold.  The end of the hold occurrs\
                                                                        when
\ **** de*/dt is nearly zero.

STATE:  Imidization

   VARIABLE:  HoldTemp1 := 193e0
   VARIABLE:  OvenHoldTemp1 := 205e0
   VARIABLE:  TimeInterval := 0e0
   VARIABLE:  RampRate := 6e0  \ Deg/min---as of 3/18/92 have boosted ramp rate from\
 5C to 6C b/c of gain problems at higher temp heating rates with small press at W&M and\
         b/c run data shows that this is only giving approx. 1.2C/min ramp rateby SMH
   VARIABLE:  HoldTime := 0e0
   VARIABLE: Now.End200Chold? := 0e0
   VARIABLE: Frequency4flat? := 0e0
   VARIABLE: Frequency5flat? := 0e0


   STATECONDITIONS
```

```
                        \ ( ABS( Rate( Part-1-E2wf5 3 ) ) < ( 3e-4 * TimeFactor ) )\
                                                                    OR \ AND
    \ **remove min time requirement 10/2/91                    ( HoldTime >= ( 1.8e2 /\
                              TimeFactor ) ) OR  \ 1800e0 sec is 30min minimum time

                        ( Now.End200Chold? = 1e0 ) OR   \ +++7/3/92++This is\
                                                              determined in a set
                                                         \ +++ of 3 rules that\
                                                               determines whether
                                                         \ +++freq4=5kHz or\
                                                             freq5=25kHz signal has
                                                         \ +++dropped below 3.0e-4\
                                                          for 4 consecutive cycles.
                        ( HoldTime >= ( 1.08e4 / TimeFactor ) )  }  \ \
                              5-22-92****10800 sec is 180 min max backup hold time

\ +++6-26-91++This state now requires that the part reach a certain minimum temp, that\
                                                                    the e*w signal
\ be decreasing for 4 consecutive readings, that the rate of change in e*w approach\
                                                                    zero, and that
\ the hold be at least 30 minutes long before it is considered complete.  If these\
                                                                 requirements are
\ not met in less than 120 minutes, the episode reaches completion after a max of 120\
                                                                       minutes.
\ 30 minutes seems to be a minimum time for this to occur and we want to ensure that we\
                                                                     hold here
\ long enough to complete the imidization; hence the requirement that it hold at least\
                                                                       30 minutes.

\ ****7-9-91 I commented off the requirement for there to be decreasing e*w values\
                                                                  because this is
\ difficult to have happen in the simulation and keep the rate under .006.  Also\
                                                                 looking at PMR-15
\ data the e* values are always reducing after being placed on the temp hold.  The\
                                                                    30min minimum
\ hold at the temp will take care of the initial flattening of the peak.

            ENDCONDITION,
        }}

    EXCITORS
        {{
            RULE: Excite200Cramp
                IF: {   ( Magnitude( Part-1-Mid-Temp 0 ) < HoldTemp1 )   AND          \
                                                  \ 1st temp check was 5 deg diff max
                        ( ABS( Magnitude( SmallPress-Temp 0 ) - Magnitude(\
   Part-1-Mid-Temp 0 ) )  < 8e0 ) AND \ **use absolute value of diff. btwn these temps\
    10/2/91;++changed allowed diff. to 4C from 8C 7/5/92;++changed allowed diff. to 8C\
                                                                    from 4C 7/6/92
                        ( ABS( SetPnt - Magnitude( Part-1-Mid-Temp 0 ) )  < 10e0  ) } \\
             **keep outtemp-midtemp diff. less than 12 degrees  10/2/91;++less than 8C\
                                                    7/5/92;++less than 10C 7/6/92
                THEN:   TimeInterval := { SampleTime( Part-1-Mid-Temp ) }
                        SetPnt := { SetPnt + ( ( RampRate / 60e0 ) * TimeInterval *\
                                                                    TimeFactor ) }
                    \   SetPntINT := INTEGER( SetPnt )
                        SET( Heat-Ctrl INTEGER( SetPnt ) )
                ENDRULE,

\ *** The above IF statement has a condition to not allow more than a 5C temp gradient\
                                                                       (7-9-91)
\ **actually 8C as of end of Jan.,1992.

            RULE: Excite200Chold
```

```
                IF:     {   Magnitude( Part-1-Mid-Temp 0 ) >= HoldTemp1 AND
                            ( ABS( Rate( Part-1-E2wf4 0 ) ) < ( 3e-4 * TimeFactor ) )\
                                                                          AND
                            ( ABS( Rate( Part-1-E2wf4 1 ) ) < ( 3e-4 * TimeFactor ) )\
                                                                          AND
                            ( ABS( Rate( Part-1-E2wf4 2 ) ) < ( 3e-4 * TimeFactor ) )\
                                                                          AND
                            ( ABS( Rate( Part-1-E2wf4 3 ) ) < ( 3e-4 * TimeFactor ) ) }\

                THEN:   Frequency4flat? := 1e0
                ENDRULE,

            RULE: End200Chold.now
                IF:     {   Frequency4flat? = 1e0 OR
                            Frequency5flat? = 1e0 }
                THEN:   Now.End200Chold? := 1e0
                ENDRULE,

        }}
    EndState


( ************************************************************************** )
( *                        STATE   CrossLinking-Min-Viscosity              * )
( ************************************************************************** )

\ We think that a viscosity min. exists before cross-linking at approx. 280C.  Thus we\
                                                                          have
\ attempted to normalize the change in consecutive E2 values with the corresponding\
                                                                        change
\ in temp. The achievement of >10% difference in e*/temp rate for three consecutive\
                                                                      cycles will
\ define the temp. of the hold to follow.

\ DecreasingSigma? is a quasi boolean variable used to signal when the condition for\
                                                                       decreasing
\ e*/tempRate is met.  It is given a value of 1e0 as the signal.

STATE: CrossLinking-Min-Viscosity
    VARIABLE: HoldTempMax := 280e0
    VARIABLE: HoldTempMmin := 265e0
    VARIABLE: DecreasingSigma? := 0e0
    VARIABLE: RampRate := 3e0   \ Deg/min---as of 3/13/92 have decreased ramp rate from\
 5C to 3C b/c of gain problems at higher temp heating rates with small press at W&M and\
        that a 5C/min rate is too fast giving 3.6C/min rather than desired 2C/minby SMH
    VARIABLE: TimeInterval := 0e0
    VARIABLE: SlopeMx := 0e0
    VARIABLE: e2/heatRateA := 0e0
    VARIABLE: e2/heatRateB := 0e0
    VARIABLE: e2/heatRateC := 0e0
    VARIABLE: e2/heatRateD := 0e0

    STATECONDITIONS
        {{
            CONDITION: CrosslinkMinViscosity
                TRUEIF: {   ( Magnitude( Part-1-Mid-Temp 0 ) >= HoldTempMax )  OR
                            ( DecreasingSigma? = 1e0 )  }
            ENDCONDITION,
        }}

    EXCITORS
        {{
            RULE: ExciteCrosslinkRamp
```

```
\ ***(7-10-91) The idea of the following three rules is the following.  The rule
\ FindingDecreasingSigma is based on e" for frequency 3 flattening out.  The e" is
\ increasing due to teperature rising thus we must normalize the increase with the
\ increase in temperature by dividing the e" rate by the temp rate.  This is the\
                                                                        variable
\ e2/heatRate.  If the temp rate happens to be zero at the beginning of the state there\
                                                                          will
\ be a division by zero error.  To avoid this the rules NonZeroRate and ZeroRate were\
                                                                         written
\ to assign values to e2/heatRate.  If temp rate is > .003 deg/min then the value will\
                                                                            be
\ calaculated but if it is less than .003 then the dummy value -666 is assigned.  The\
                                                                       HeatRate
\ values are also kept historic with e2/heatRateA being most current and e2/heatRateD\
                                                                     the oldest.

            RULE: NonZeroRate2          \ **7-9-91 changed to frequency 5 instead of 3\
                                                                         for sim.
                IF: { Rate( Part-1-Mid-Temp 0 ) > 1e-3 }
                THEN:   e2/heatRateD := e2/heatRateC
                        e2/heatRateC := e2/heatRateB
                        e2/heatRateB := e2/heatRateA
                        e2/heatRateA := { Rate( Part-1-E2f5 0 ) / Rate( Part-1-Mid-Temp\
                                                                          0 ) }
            ENDRULE,

            RULE: ZeroRate2
                IF: { Rate( Part-1-Mid-Temp 0 ) < 1e-3 }
                THEN:   e2/heatRateD := e2/heatRateC
                        e2/heatRateC := e2/heatRateB
                        e2/heatRateB := e2/heatRateA
                        e2/heatRateA :=  -666e0 \ -666 is dummy value
            ENDRULE,

            RULE: FindMaxSlope2
                IF: { ( e2/heatRateA < ( e2/heatRateB * 1.2e0 ) ) AND
                      ( e2/heatRateA <> -666e0 ) }
                THEN:   SlopeMx := { SlopeMx MAX e2/heatRateA }
            ENDRULE,

            RULE: FindingDecreasingSigma2
                IF: {
                        ( e2/heatRateA < ( 0.6e0 * SlopeMx ) ) AND
                        ( e2/heatRateB < ( 0.6e0 * SlopeMx ) ) AND
                        ( e2/heatRateC < ( 0.6e0 * SlopeMx ) ) AND
                        ( e2/heatRateD < ( 0.6e0 * SlopeMx ) ) AND
                        ( e2/heatRateA <> -666e0 )
                                                              }

                THEN:   DecreasingSigma? := 1e0
            ENDRULE,

        }}
    EndState


( ************************************************************************** )
( *                        STATE   CrossLinking Hold                       * )
( ************************************************************************** )

\ ****  This is just a hold for 30 min. at that min visc temp. The addition of pressure\
                                                                          will
```

```
    VARIABLE:  HoldTime := 0e0
    VARIABLE:  SetTempInitial := 270e0
    VARIABLE: Now.EndHighhold? := 0e0
    VARIABLE: Frequency4flat(#2)? := 0e0
    VARIABLE: Frequency5flat(#2)? := 0e0

    STATECONDITIONS
        {{
            CONDITION: CrossLinkingHold-Conditions
                TRUEIF: {   \ ( ABS( Rate( Part-1-E2wf5 0 ) ) < ( 3e-4 * TimeFactor ) )\
                                                                              AND
                            \ ( ABS( Rate( Part-1-E2wf5 1 ) ) < ( 3e-4 * TimeFactor ) )\
                                                                              AND
                            \ ( ABS( Rate( Part-1-E2wf5 2 ) ) < ( 3e-4 * TimeFactor ) )\
                                                                              AND
                            \ ( ABS( Rate( Part-1-E2wf5 3 ) ) < ( 3e-4 * TimeFactor ) )\
                                                                               OR
                            \ 5-22-92***REMOVED SET 30 MIN HOLD AND REPLACED WITH\
     REQUIREMENT THAT IT FLATTEN OUT AS WITH 200C HOLD UNLESS A MAX OF 2HRS IS REACHED.


                            ( Now.EndHighhold? = 1e0 ) OR   \ +++7/3/92++This is\
                                                                    determined in a set
                                                             \ +++ of 3 rules that\
                                                                     determines whether
                                                             \ +++freq4=5kHz or\
                                                                 freq5=25kHz signal has
                                                             \ +++dropped below 3.0e-4\
                                                              for 4 consecutive cycles.



                            ( HoldTime >= ( 7.2e3 / TimeFactor ) )  \  is a 120 min.\
                                                                  hold is desired at the temp.
                                                           \ where crosslink min. viscosity\
                                                                                 occurred.
                        }
            ENDCONDITION,
        }}

    EXCITORS
        {{
            RULE: CrossLinkHold
                IF:     TRUE
                THEN:   TimeInterval :=  { SampleTime( Part-1-Mid-Temp ) }
                        HoldTime := { HoldTime + TimeInterval }
                ENDRULE,


            RULE: No-TempGrad
                IF: { HoldTime <= SampleTime( Part-1-Mid-Temp ) }       \ i.e., will\
                                          only do this when 1st enters hold,3/18/92 by smh
                THEN:   SetTempInitial := { Magnitude( Part-1-Mid-Temp 0 ) + 6e0 }
                        SetPnt := SetTempInitial    \ sets it to the part midtemp plus\
       10C for the hold,7/3/92 +++6C 7/5/92+++by smh (so not too hot but will maintain\
                                                                 approx. current part temp.)
                    \   SetPntINT := INTEGER( SetPnt )  \ changes so have integer set\
                            point here made 3/10/92 by SMH so do not get system crashes
                        SET( Heat-Ctrl INTEGER( SetPnt ) )

            ENDRULE,

    \ +++7/3/92++The next 3 rules determine whether the High temperature consolidation and\
                                                                                 the final
```

```
                ENDRULE,

            RULE: EndHighhold.freq.4
                IF:     {   Magnitude( Part-1-Mid-Temp 0 ) >= HoldTemp1 AND
                            ( ABS( Rate( Part-1-E2wf4 0 ) ) < ( 3e-4 * TimeFactor ) )\
                                                                                AND
                            ( ABS( Rate( Part-1-E2wf4 1 ) ) < ( 3e-4 * TimeFactor ) )\
                                                                                AND
                            ( ABS( Rate( Part-1-E2wf4 2 ) ) < ( 3e-4 * TimeFactor ) )\
                                                                                AND
                            ( ABS( Rate( Part-1-E2wf4 3 ) ) < ( 3e-4 * TimeFactor ) ) }\

                THEN:   Frequency4flat(#2)? := 1e0
                ENDRULE,

            RULE: EndHighhold.now
                IF:     {   Frequency4flat(#2)? = 1e0 OR
                        .   Frequency5flat(#2)? = 1e0 }
                THEN:   Now.EndHighhold? := 1e0
                ENDRULE,

        }}
    EndState


( ************************************************************************** )
( *                          STATE   Final Cure Ramp and Hold              * )
( ************************************************************************** )

\ **** This is just the final ramp to 320C and hold until de*/dt is near zero.

STATE: Final-Cure-Ramp-and-Hold

    VARIABLE: HoldTime := 0e0
    VARIABLE: TimeInterval
    VARIABLE: HoldTemp1 := 315e0
    VARIABLE: RampRate := 5e0   \ Deg/min---as of 3/12/92 have boosted ramp rate from\
 2C to 5C b/c of gain problems at higher temp heating rates with small press at W&M,by\
                                                                                SMH
    VARIABLE: Now.End320Chold? := 0e0
    VARIABLE: Frequency4flat(#3)? := 0e0
    VARIABLE: Frequency5flat(#3)? := 0e0



    STATECONDITIONS
        {{
            CONDITION: FinalCureRampAndHoldConditions
                TRUEIF: {   ( Magnitude( Part-1-Mid-Temp 0 ) >= HoldTemp1 ) AND
                            \ ( ABS( Rate( Part-1-E2f5 0 ) ) < ( 3e-5 * TimeFactor ) )\
                                                                                AND
                            \ ( ABS( Rate( Part-1-E2f5 1 ) ) < ( 3e-5 * TimeFactor ) )\
                                                                                AND
                            \ ( ABS( Rate( Part-1-E2f5 2 ) ) < ( 3e-5 * TimeFactor ) )\
                                                                                AND
                            \ ( ABS( Rate( Part-1-E2f5 3 ) ) < ( 3e-5 * TimeFactor ) )\
                                                                        OR   \ AND
    \ **remove min hold time and let sensor do the work 10/2/91                     \
                    ( HoldTime > ( 1.8e3 / TimeFactor ) ) OR    \ would have 30min hold



                            ( Now.End320Chold? = 1e0 ) OR   \ +++7/3/92++This is\
                                                                determined in a set
```

```
        }}

    EXCITORS
        {{
            RULE: ExciteLastHold
                IF: {   Magnitude( Part-1-Mid-Temp 0 ) >= HoldTemp1 }
                THEN:   TimeInterval :=  { SampleTime( Part-1-Mid-Temp ) }
                        HoldTime := { HoldTime + TimeInterval }
            ENDRULE,

            RULE: ExciteLastRamp
                IF: {   ( Magnitude( Part-1-Mid-Temp 0 ) < HoldTemp1 ) AND
                        ( ABS( Magnitude( SmallPress-Temp 0 ) - Magnitude(\
    Part-1-Mid-Temp 0 ) )  < 8e0 ) AND \ **use absolute value of the difference 10/2/91
                                                                                \
                                                                \ 8C allowed 3/2/92
\ ***prevents 4C temp gradient                                                  \
                                                    \ 4C allowed 7/5/92;back to 8C 7/6/92
                        ( ABS( SetPnt - Magnitude( Part-1-Mid-Temp 0 ) ) < 10e0 ) } \\
                              **keep outtemp-midtemp diff. less than 12 degrees  3/9/92
                                                                                \
                \ **keep outtemp-midtemp diff. less than 8 degrees  7/5/92; <10C 7/6/92
                THEN:   TimeInterval := { SampleTime( Part-1-Mid-Temp ) }
                        SetPnt := { SetPnt + ( ( RampRate / 60e0 ) * TimeInterval *\
                                                                    TimeFactor ) }
                    \   SetPntINT := INTEGER( SetPnt )
                        SET( Heat-Ctrl INTEGER( SetPnt ) )

                ENDRULE,

\ +++7/3/92++The next 3 rules determine whether the final crosslinking process is\
                                                                    complete for
\ +++frequency4=5kHz or for frequency5=25kHz by requiring that 4 consecutive values be\
                                                                        <=3.0e-4.
\ +++If either of these is true and we are above 315C (ie, into the desired 320C hold),\
                                                                    then the KB
\ +++determines that the hold should end and makes the corresponding quasiboolean\
                                                                        variable
\ +++Now.End320Chold = 1 (ie, true) and this is checked in the state requirements.
            RULE: End320Chold.freq.5
                IF:     {   Magnitude( Part-1-Mid-Temp 0 ) >= HoldTemp1 AND
                            ( ABS( Rate( Part-1-E2wf5 0 ) ) < ( 3e-5 * TimeFactor ) )\
                                                                            AND
                            ( ABS( Rate( Part-1-E2wf5 1 ) ) < ( 3e-5 * TimeFactor ) )\
                                                                            AND
                            ( ABS( Rate( Part-1-E2wf5 2 ) ) < ( 3e-5 * TimeFactor ) )\
                                                                            AND
                            ( ABS( Rate( Part-1-E2wf5 3 ) ) < ( 3e-5 * TimeFactor ) ) }\

                THEN:   Frequency5flat(#3)? := 1e0
                ENDRULE,

            RULE: End320Chold.freq.4
                IF:     {   Magnitude( Part-1-Mid-Temp 0 ) >= HoldTemp1 AND
                            ( ABS( Rate( Part-1-E2wf4 0 ) ) < ( 3e-5 * TimeFactor ) )\
                                                                            AND
                            ( ABS( Rate( Part-1-E2wf4 1 ) ) < ( 3e-5 * TimeFactor ) )\
                                                                            AND
                            ( ABS( Rate( Part-1-E2wf4 2 ) ) < ( 3e-5 * TimeFactor ) )\
                                                                            AND
                            ( ABS( Rate( Part-1-E2wf4 3 ) ) < ( 3e-5 * TimeFactor ) ) }\

                THEN:   Frequency4flat(#3)? := 1e0
                ENDRULE,
```

```
\ *** this state is the cool down to 40C hence ending the cure. It's Miller time\
                                                                        (7-10-91)

STATE: Cool-Down

\   VARIABLE: RampRate := 5e0       \ **290 for sim but would be more like 5 in run
    VARIABLE: RampRate := 15e0      \ **290 for sim but would be more like 15 in run
    VARIABLE: ShutdownTemp := 295e0
\   VARIABLE: ShutdownTemp := 40e0

    STATECONDITIONS
        {{
            CONDITION: CooledDown
                TRUEIF: {    ( Magnitude( SmallPress-Temp 0 ) <= ShutdownTemp ) }
            ENDCONDITION,
        }}

    EXCITORS
        {{

            RULE: ExciteCoolDown
                IF: { ( ABS( Magnitude( Part-1-Mid-Temp 0 ) - Magnitude(\
       SmallPress-Temp 0 ) )  < 15e0 ) }  \ **use absolute value of the diff. in temps\
                                                                        10/2/91
                THEN:   TimeInterval := { SampleTime( Part-1-Mid-Temp ) }
                        SetPnt := { SetPnt - ( ( RampRate / 60e0 ) * TimeInterval *\
                                                                TimeFactor ) }
                    \   SetPntINT := INTEGER( SetPnt )
                        SET( Heat-Ctrl INTEGER( SetPnt ) )
                        SET( Pressure-Ctrl 0 )
                ENDRULE,
        }}
Endstate

( ************************************************************************** )
( *                          STATE   Shut Down                             * )
( ************************************************************************** )

STATE: Shut-Down

    STATECONDITIONS
        {{
            CONDITION: Shutdown?
                TRUEIF: { SetPoint?( Heat-Ctrl ) = 27e0 }
            ENDCONDITION,
        }}

    EXCITORS
        {{

            RULE: Shutingdown
                IF: True
                THEN:   SET( Heat-Ctrl 27 )     \ ***use 20 for autoclave FDEMS run\
                                                                shutdown trigger

                ENDRULE,
        }}
Endstate

( ************************************************************************** )
( ************************************************************************** )
( ************************************************************************** )
( *                                Episodes                                * )
```

```
( ************************************************************************** )
( ************************************************************************** )


( ************************************************************************** )
( *                          EPISODE  Achieve PreImidization Max Flow      * )
( ************************************************************************** )
EPISODE:    Achieve-PreImidizationMaxFlow
    GOAL is ACHIEVE  PreImidizationMaxFlow
    EndEpisode


( ************************************************************************** )
( *                          EPISODE Achieve Imidization Onset             * )
( ************************************************************************** )
EPISODE:    Achieve-ImidizationOnset
    GOAL is ACHIEVE ImidizationOnset
    StartConditions
        {{
        CONDITION:  StartImidizationOnset
            TRUEIF: Complete?( Achieve-PreImidizationMaxFlow )
            ENDCONDITION,
        }}
    EndEpisode


( ************************************************************************** )
( *                          EPISODE Achieve Imidization                   * )
( ************************************************************************** )
EPISODE:    Achieve-Imidization
    GOAL is ACHIEVE Imidization
    StartConditions
        {{
        CONDITION:  StartAchieveImidization
            TRUEIF: Complete?( Achieve-ImidizationOnset )
            ENDCONDITION,
        }}
    EndEpisode


( ************************************************************************** )
( *                          EPISODE Achieve CrossLinking-Min-Viscosity    * )
( ************************************************************************** )
EPISODE:    Achieve-XLinking-Min-Viscosity
    GOAL is ACHIEVE CrossLinking-Min-Viscosity
    StartConditions
        {{
        CONDITION:  StartAchieveCrossLinking-Min-Viscosity
            TRUEIF: Complete?( Achieve-Imidization )
            ENDCONDITION,
        }}
    EndConditions
```

```
    EndEpisode

( ************************************************************************** )
( *                     EPISODE Achieve CrossLinking Hold                  * )
( ************************************************************************** )
EPISODE:    Achieve-CrossLinkingHold
    GOAL is ACHIEVE CrossLinkingHold
    StartConditions
        {{
        CONDITION:  StartAchieveCrossLinkingHold
            TRUEIF: Complete?( Achieve-XLinking-Min-Viscosity )
            ENDCONDITION,
        }}
    EndEpisode

( ************************************************************************** )
( *                          EPISODE Achieve Final Cure Ramp and Hold      * )
( ************************************************************************** )
EPISODE:    Achieve-Final-Ramp-and-Hold
    GOAL is ACHIEVE Final-Cure-Ramp-and-Hold
    StartConditions
        {{
        CONDITION:  StartAchieveFinal-Cure-Ramp-and-Hold
            TRUEIF: Complete?( Achieve-CrossLinkingHold )
            ENDCONDITION,
        }}
    EndEpisode

( ************************************************************************** )
( *                          EPISODE Achieve Cool Down                     * )
( ************************************************************************** )
EPISODE:    Achieve-Cool-Down
    GOAL is ACHIEVE Cool-Down
    StartConditions
        {{
        CONDITION:  StartAchieveCool-Down
            TRUEIF: Complete?( Achieve-Final-Ramp-and-Hold )
            ENDCONDITION,
        }}
    EndEpisode

( ************************************************************************** )
( *                          EPISODE Shut Down                             * )
( ************************************************************************** )
EPISODE:    Achieve-Shut-Down
    GOAL is ACHIEVE Shut-Down
    StartConditions
```

```
        {{
        CONDITION:  StartAchieveShut-Down
            TRUEIF: { Complete?( Achieve-Cool-Down ) OR
                          AbortRun? = 1e0 } \ **7-22-92**This added for runs aborted\
                                                                    during 1st episode.
            ENDCONDITION,
        }}
    EndEpisode



( ************************************************************************** )
( ************************************************************************** )
( ************************************************************************** )
( *                                 Plans                                  * )
( ************************************************************************** )
( ************************************************************************** )
( ************************************************************************** )


( ************************************************************************** )
( *                                 PLAN Cure                              * )
( ************************************************************************** )
PLAN:   Cure
    Episodes {{

               Achieve-PreImidizationMaxFlow
               Achieve-ImidizationOnset
               Achieve-Imidization
               Achieve-XLinking-Min-Viscosity
               Achieve-CrossLinkingHold
               Achieve-Final-Ramp-and-Hold
               Achieve-Cool-Down
               Achieve-Shut-Down
            }}
    EndConditions
            {{
            CONDITION:  EndCure
                TRUEIF: {   Complete?( Achieve-Shut-Down ) }
                ENDCONDITION,
            }}
    EndPlan

( ************************************************************************** )
( ************************************************************************** )
( ************************************************************************** )
( *                                 Schedule                               * )
( ************************************************************************** )
( ************************************************************************** )
( ************************************************************************** )


( ************************************************************************** )
( *                           SCHEDULE Cure-1-Part                         * )
( ************************************************************************** )
SCHEDULE:   Cure-1-Part
    Plans
        {{  Cure

        }}
```

```
    Messages
        {{
        }}

    ENDSCHEDULE

End KnowledgeBase
```

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