

Pultruding Epoxy Resin

ABSTRACT

The RP pultrusion industry as a whole is most familiar with the polyester family of thermoset resin systems. Because of this fact, epoxy pultrusion can present numerous problems to many pultruders. For the most part, the basic inherent differences between epoxy and polyester resins, and the role these differences play in the pultrusion process is the cause for the problems encountered. Some of the problems are encountered before the resin reaches the machine. All too often trial and error overcomes these difficulties at a significant expense. Even then, there is always some doubt as to what was the real cause or cure for the problems. Without a clear understanding of the pultrusion process internal dynamics and how the inherent resin differences relate to the internal dynamics, it is difficult to perceive the correct measures to eliminate the processing related problems. Adopting certain techniques for handling epoxies from other epoxy end use industries can eliminate the non-process related problems pultruders may experience with epoxies. The intent of this paper is to define these inherent differences, and to relate them to the internal dynamics and to the pultrusion process in such a manner that it will become clear how and why certain provisions must be made for epoxy pultrusion. Knowing this in advance, the first time epoxy pultruder will be able to produce a state of the art advanced composite profile with a minimum amount developmental costs, time, and frustration.

THE INTERNAL DYNAMICS OF THE PULTRUSION PROCESS

Figure 1 is a depiction of the die cross section which will be referenced from time to time within this paper. It shows three zones of the pultrusion process. "Zone 1" is where the material enters the die at room temperature and is heated by the die which causes hydraulic pressure to rise. As the material progresses into "Zone 2," or the "Gel Zone" it changes from a viscous liquid into a non-flowing sticky gelatinous mass, then into a rubber like texture. As the material cures to a hard solid, volumetric shrinkage causes the pressure forces to decay and the product releases from the surface of the die. "Zone 3" is where minor sliding frictional forces are generated between the cured material and the die. Depending on the thickness of the part and the process rate, the inherent bullet nose shape of the "Gel Zone" will become shorter or longer. This is a well understood qualitative model of the pultrusion process. Sumerak (1985) quantitatively described the internal dynamics of the pultrusion process.

Based on Sumerak's work, Figure 2 shows a typical temperature/pressure/viscosity profile of the pultrusion process with a polyester resin (Reichold 31-020). However, for illustrative purposes, the

pressure profiles in this illustration are computer regenerations of the typical measurements reported by Sumerak using the cubic spline mathematical curve fitting formula. Two assumptions were made in computing these pressure curves. It was assumed that the pressure at the very entrance of the die is zero, and at some point inside the die as the resin cures, volumetric shrinkage causes the pressure to diminish to zero. These points were added to Sumerak's data in order to derive a pressure profile that is representative of the total picture. Until a pressure sensing device is invented that can be fed through the die to record the pressure profile much like a thermocouple, computer manipulated static data points is the best we can do.

As one can see by this illustration, the pressure/temperature/viscosity profiles of the pultrusion process are a direct function of the processing speed. Combining test results from Sumerak's work, Figure 3 shows the relationship of pull loads vs. processing speed of the catalyzed and uncatalyzed resin systems. The rising pull load associated with increased processing rate for the uncatalyzed resin is the result of increasing viscous shear forces over the entire length of the die. However, in the catalyzed resin, and as noted in Figure 1, viscous shear forces are generated only in the front portion of the die, "Zone 1." Within "Zone 2" or the "gel zone" cohesive forces come into play for a relatively small area until sufficient cure has taken place to change the physical state of the resin to a rubbery solid which contributes substantial frictional forces. As the resin continues to cure, it becomes harder and shrinks away from the surface of the die reducing the frictional forces in "Zone 3" quite dramatically. As one can see, the pull load is significantly higher for the catalyzed resin, particularly at the faster processing speed. This is evidence that the major portion of the pull loads associated with the pultrusion process is generated in the "gel zone" where cohesive forces and frictional forces are greatest. By referring back to Figure 2, one can see that as the speed increases, the pressure profile moves downstream and further into the "gel zone" which has also grown to cover a larger surface area on the die. The resulting increased pressure exerted over a larger area of cohesive and frictional forces results in greater pull loads.

One of the many things we learn from the diligent work of Sumerak is that a significant amount of pressure does develop inside the die during the pultrusion process, and it is proportional to the processing rate. If the hydraulic pressure is a result of thermal expansion, then, conversely, pressure loss or decay is the result of contraction, or shrinkage. It is clearly evident in Figure 2, that the pressure loss occurs well before the material begins to cool. Therefore, it is not thermal contraction we are witnessing, but volumetric shrinkage due to cure of the resin matrix. Although relatively small in effect, the dimensional change of the die cavity proportional to the coefficient of thermal expansion for steel also contrib-

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utes to the pressure loss. For example: with a temperature differential of only 240F, the hottest cross-sectional area of the die for 1/2" diam. rod will be 0.2-0.3 percent larger than the entrance. Pressure, and volumetric shrinkage together play a very important role in the pultrusion dynamics. Insufficient pressure causes sloughing problems. Insufficient shrinkage causes excessive pull loads. The rate of shrinkage affects the rate of pressure decay and is proportionally controlled by the cure rate of the resin. A delicate balance between pressure, cure rate, and shrinkage must be obtained in order for the pultrusion process to take place.

THE INHERENT DIFFERENCES BETWEEN EPOXIES AND POLYESTERS

Given this understanding of the internal dynamics of the pultrusion process, it will be easier to understand how the inherent differences in shrinkage and cure rate between polyesters and epoxies relate to those dynamics.

Shrinkage

Volumetric shrinkage tests were conducted on the apparatus shown in Figure 4. A known volume of resin was placed in a dropping bottle and low density mineral oil was placed on top. The dropping bottle was immersed in a room temperature constant temperature bath and allowed to stabilize; an initial volume reading was taken from the pipette. The dropping bottle was then immersed in a 70C constant temperature bath. The pipette volume was recorded at timed intervals. Since the resin sample volumes were nearly identical, the effect of the thermal expansion and contraction of the mineral oil between the samples is cancelled out. Therefore the data is relative. As the materials were heated, volumetric expansion was measured from the pipette. As the resin temperature approached the bath temperature and began to cure, the rate of expansion decayed. Eventually, resin volumetric shrinkage occurred, and finally, when the resin was fully cured, the volume change became zero. Then the dropping bottle was placed back into the room temperature bath. As the cured resin and oil cools, thermal contraction was measured. The volume change measurements were plotted against time. The net volume loss was compared to the initial resin volume, and a net volumetric shrinkage was computed. For comparison purposes, the dropping bottle with cured resin was recycled through the same temperature cycle, and volumetric expansion and contraction measurements were recorded and plotted on the same graph.

Figure 5 depicts the volume change profile of a polyester resin (Aropol 7030) and Figure 6 depicts the same for the epoxy resin (EPON® Resin 9310/EPON™ CURING AGENT 9360 & Accelerator 537). As expected, the polyester shrinks nearly twice that of the epoxy. However, the net shrinkage is not nearly as important as the shrinkage profile. Note how the polyester resin gels and continues to exhibit expansion, and suddenly shrinks at a high initial rate that gradually tapers off. On the other hand, the epoxy resin begins to shrink before it gels, and continues to shrink at a steadily declining rate until it is fully cured.

The results of these shrinkage experiments have revealed significant information that sheds new light on the understanding of the pultrusion characteristics of epoxies versus polyesters. In any thermoset molding process in the RP industry, it is always desirable to cure the product under pressure. Similarly, cure or gelation under pressure is desirable in the pultrusion process in that it provides for a glossy part surface by holding the product tightly to the surface of the die during gelation, which subsequently prevents sloughing.

From these test results it is readily apparent that the polyester resin shrinkage characteristics provide the best conditions for gela-

tion to occur under pressure. The sudden high initial rate of shrinkage after gelation which the polyester resin exhibits is also desirable, in that it provides for fast pressure reduction which reduces the frictional forces proportionally. Subsequently, the typical pull loads for the polyester resins are minimal. As mentioned earlier, if gelation occurs with insufficient pressure, the product will exhibit a dull surface and will be prone to sloughing and plugging.

The epoxy resin, however, begins to shrink well in advance of gelation, and gels under a condition of declining hydraulic pressure. Therefore by the time gelation does occur, a significant amount of the desirable hydraulic pressure resulting from thermal expansion is lost due to the effect of volumetric shrinkage. After gelation, the remaining small amount of shrinkage that can occur does so at such a slow rate that it retards the pressure decay needed to reduce friction. Because of this inherent shrinkage characteristic, there is insufficient pressure in the gel zone to prevent sloughing. This explains why sloughing is encountered when epoxy resin is substituted directly for the polyester resin in the pultrusion process. However, there is a simple solution that has been found to compensate for these unfavorable inherent shrinkage characteristics of the epoxy resin.

It is a well known fact that the total volumetric shrinkage of a resin is inversely proportional to the filler or reinforcement volume. In addition, the pressure due to thermal expansion is directly proportional to the filler or reinforcement volume. Therefore by increasing the reinforcement to resin volume ratio, the total volumetric shrinkage that can occur will be reduced, and simultaneously the hydraulic pressure will be increased. In another sense, there will be less shrinkage working on more pressure. Thus, even though the epoxy shrinks prior to gelation, sufficient pressure will remain to prevent sloughing. This explains why epoxy resins have been found to require higher fiber volume to resin ratios than polyesters in order to prevent sloughing.

*need high filler or
fiber content
to ↓ volume shrinkage*

Cure Rate

As we mentioned earlier, shrinkage rate is controlled by the inherent cure rate of the resin. Since it is desirable to have a high shrinkage rate to provide a quick pressure drop to reduce the frictional pull loads, it is important for the epoxy to have a fast cure rate. As a secondary benefit, a fast cure rate will provide a shorter gel zone, allowing faster processing rates.

The cure rate of polyesters can be varied chemically by varying the amounts and types of peroxides used to catalyze them. In that respect, epoxy resins are not as flexible.

For epoxy resins, the curing agent selection is based primarily on the desired cured resin properties, with pot life considerations. The cure rate can be varied somewhat by the use of thermally latent accelerators. However, the use of too much accelerator to enhance cure rate compromises pot life. Figure 7 shows the gel times of the epoxy resin with two accelerator levels and the polyester resin with two types of peroxide catalysts. Note that the epoxy resin requires significantly more heat in order to generate the same gel times even with doubling the accelerator level. Figure 8 shows viscosity vs. time of the epoxy resin at two accelerator levels and two temperatures. One can easily see from this graph, that a significant amount of pot life is sacrificed by doubling the accelerator level.

Temperature has an even more significant effect on pot life. As illustrated in Figure 8, a minor increase in temperature (7.2C) will reduce the time to double the initial viscosity by nearly half. Because of this inherent characteristic of epoxy resin, it is most important to minimize mixing times when using high shear mixers that can generate heat. From these two charts, it is easy to see that the least costly method to increase cure rate in the epoxy resin is by increasing the die temperatures.

CONTROLLING THE SHRINKAGE AND CURE RATE OF THE EPOXY

Up to this point, the inherent differences of shrinkage and cure rate between epoxies and polyesters and how they relate to internal dynamics have been discussed. In addition, the best methods to compensate for the epoxy's characteristics were identified. Those methods are: 1) controlling the reinforcement volume. 2) controlling the die temperature. Since both of these controls directly affect the forces contributing to the pull loads, this measurement can be used to help us establish the correct values.

Reinforcement Volume

Figure 9 shows the relationship of glass content (or non-combustibles) versus pull loads for the epoxy resin. This graph was generated from an experiment where the reinforcement volume was reduced in a stepped fashion and the pull loads recorded until the onset of sloughing. Then the fiber volume was increased slowly until sloughing was eliminated and further yet until the pull loads became excessive. Actual glass content values were determined by combustion, and include residual clay that did not burn off. As you can see by the data, there is a "plateau" in the pull load curve spanning approximately two percent of the glass content range. This plateau is the optimum level for pultruding a 1/2" diameter epoxy rod. Below that level, sloughing occurs because of insufficient hydraulic pressure at the gel point. Above the optimum level, the pull loads rise dramatically because of too much pressure during and after the gel zone. This illustrates how to determine the proper fiber volume level for epoxy resins.

Epoxies respond to varying types of reinforcement packages similar to that of polyesters. Therefore just as in polyester pultrusion, the minimum reinforcement level to prevent sloughing for a continuous mat and roving reinforced epoxy profile is somewhat less than that for an all roving part. Table 1 lists the target reinforcement volumes for the epoxy resin system for various types of reinforcement packages that are typical to the industry. These values were obtained by conducting experiments similar to that illustrated in Figure 9 above. These are target values that can be used to calculate a starting point. Adding and subtracting reinforcement may be necessary to fine tune the level for optimum surface quality and minimum pull loads. To the experienced polyester pultruder's trained eye, it may appear impossible for all the reinforcement to fit in the die. However, it will fit. In many cases, it is necessary to heat the die during thread up in order to reduce the frictional loads of the dry reinforcement.

Die Temperature Control

In polyester pultrusion, it is most common to use strip heaters strapped to the surface of the die with the controller thermocouple located some arbitrary short distance from the entrance of the die. Based on experience, a temperature set point is established that will produce the desired surface and internal quality of the part. Often the actual exotherm temperature may vary from batch to batch or day to day unbeknown to the operator.

In epoxy pultrusion it is important to control the peak exotherm and keep it stable. In general, a maximum exotherm in the center of the part should not exceed 225C. Above that temperature the epoxy can react with itself, leaving the curing agent behind. This is called homopolymerization, and will seriously degrade the physical properties. For most thin profiles (up to 1/2" thick), a single heating zone is sufficient. To obtain the most precise control of peak temperature, the location of the controller thermocouple becomes very important. Locating the thermocouple in the center of the strip heater not only minimizes the overshoot and lag time for

an on/off type temperature controller, but also provides a good reference to what the actual peak temperature is. For a 1/2" thick cross-section a temperature set point of 200C on the surface will yield an internal peak exotherm of 225C. Figure 10 is a graphic example of a single zone heating profile.

Figure 11 shows the relationship of die temperature set point and pull loads for the epoxy resin. Note that if the temperature set point is too low, the pull loads increase. This is the result of reducing the epoxy resin cure rate, which increases the size of the gel zone. Simultaneously, the rate of shrinkage or the rate of hydraulic pressure decay is reduced. The result is more pressure within a larger gel zone, which increases the pull loads. As the die temperature is increased, all these conditions begin to favor reducing the pull loads.

With profiles up 1/2" thick, a two-zone heating profile can be beneficial and may eliminate the need for Radio Frequency (R.F.) preheating to prevent internal cracking. Figure 12 is a graphic example of a typical two-zone heating profile for epoxy resin. Note that the pull loads are considerably lower and the temperature decay rate after exotherm is less than the single zone heating profile. Therefore, a two-zone heating profile will produce a greater degree of cure than the single zone. This type of heating must be tuned to the processing rate and the thickness of the part.

For pultrusions 1/2" thick and above, R.F. preheating will allow faster processing rates without internal cracking. Figure 13 shows a graphic example of the die temperature conditions of a 1/2" diameter rod pultruded with R.F. preheating. Because R.F. preheating reduces the temperature differential from the entrance of the die to the gel zone there is less volumetric thermal expansion, or pressure. In addition, it reduces the temperature lag between the surface and the center of the part causing the gel zone to become smaller. The net result is reduced pull loads. By increasing the processing rate, the pressure, gel zone size, and pull loads will return to normal. This is how R.F. allows room for faster processing rates without increasing the pull loads beyond that of pultruding without R.F. preheating at the reduced rate.

OTHER PROBLEM AREAS

There are other inherent differences between epoxies and polyesters that do not relate to the internal dynamics, but can cause problems for many pultruders. Unavoidably, epoxies do have a limited pot life. Start-up, trouble shooting and shut down practices that vary from shop to shop, may not accommodate the epoxy's nature. A review of suggested techniques for handling the epoxy in these areas will complete the mission of this paper.

How to Live with a Pot Life

Batching of epoxy resins for pultrusion is just as important as die temperature control and reinforcement level. Careful attention to the mixing procedures will reduce the resin scrap and the potential for a non-scheduled shut down. For short production runs or developmental prototyping runs of up to 4 hours long, a simple single part batching operation will suffice. Table 2 shows the typical formulation and mixing times for a single batch. Note that this is a step-wise mixing process. First, all the liquid ingredients except the curing agent are mixed for a very short time (30 to 60 seconds). Leaving out the curing agent reduces the resin viscosity which aids in dispersion of the clay in the second step. Minimize the mixing time during the second step to reduce the amount of heat that may be generated, particularly if a high shear disperser is used. Shear induced heat will shorten the pot life of the resin system. Finally, just prior to start up, the curing agent is added, and blended at a reduced mixer speed, as high shear mixing is not needed once the clay is dispersed. Adding the curing agent last will dilute any tem-

perature rise that may have resulted from dispersion of the clay. The curing agent should be added at the last possible moment before start-up. The pot life clock starts ticking when it is added to the batch.

For longer production runs, a two part batching operation can be extremely beneficial. Table 3 shows the typical two part batching operation. Part "A" (the master batch), is the basic resin, accelerator, and processing additives including the clay. This portion of the resin mix can be prepared well in advance of the scheduled run. Part "A" will not gel, and is stable for up to 3 days. At that point some minor loss of reactivity may occur on behalf of the accelerator. Some settling of the clay filler will occur over time and mild mixing will re-disperse it readily.

A simple calculation is necessary for deriving the correct curing agent ratio for the particular Part "A" formulation used. As illustrated in Table 3, the sum of all the parts (phr) in Part "A" is divided into the normal curing agent to resin ratio (33 phr), and multiplied by 100. The new figure is parts of Part "B" to 100 parts of Part "A."

When a certain amount of resin is needed to start the run, simply weigh it out of the masterbatch, Part "A," and blend in the correct amount of Part "B" (the curing agent). Subsequently, when additional amounts of resin are needed to replenish the bath, repeat this step again. Scale the size of the replenishment batches to the resin depletion rate for the run. If the process consumes 3 gallons per hour, add 3 gallons per hour. This will enhance the pot life of the epoxy resin by diluting it with fresh resin on a frequent basis. Using the smallest size bath possible will increase the dilution effect of the replenishment batches, and will reduce the overall mass of the resin aging in the bath. Depending on the size of the bath, this technique can enhance the useable pot life of the resin in the bath by 800 percent or more. Eventually, the resin viscosity becomes too thick for good fiber wetting. Providing a high volume drain hole in the resin bath will allow for a quick drain and refill operation "on the fly." This will essentially reset the time on the pot life to zero. With careful attention to the details of this method, it is easy to see that epoxies can be pultruded continuously without pot life restrictions.

The ideal, but more costly approach to enhance pot life, is to incorporate a metered mixing device that can dispense just the right amount of resin to the bath when it is needed.

As with any epoxy resin system, it is always possible to have a runaway exotherm. Therefore it is very wise to incorporate certain safety measures in the event of an exotherm. Always provide a means to monitor the temperature of the bath. The best place to monitor the temperature is in a corner of the resin bath where circulation is poor. A sudden rapid rise in temperature is a clue that immediate action is necessary. If action is needed, having a quick acting, high volume drain hole in the resin bath will allow for a rapid change out of the resin "on the fly." The ability to drain and refill the bath quickly can prevent an unscheduled shut down.

Start Up Procedures

Certain provision must be made for epoxy resins during start up. It is important to start up with the minimum required amount of reinforcement. Leaving out a few percent of the reinforcement level will no doubt lead to sloughing because of insufficient total pressure in the gel zone. Once the sloughing begins there is a potential for a build up on the surface of the die to form, that may not purge out cleanly. The most troublesome areas are low pressure areas like a corner or small radius.

Many pultruders like to "pre-lube" the die with pure mold release just prior to the resin entering. Experience has shown us that this is not necessary for any resin related reasons and could lead to other problems. The types and levels of internal mold releases recommended for the epoxy were determined by laboratory and

pultrusion experiments and provide the most effectiveness with the least detriment to the physical properties of the resin. Any levels in excess of those recommended will not provide any significant amount of added benefit. In fact, substantial resin performance penalties will result. The technique of pre-lubing the die introduces the chance for high concentrations of mold release to squeeze off at the die entrance and wash back into the resin bath and "poison the batch." If pre-lubing must be used to reduce friction on the dry reinforcement, do not allow the squeeze off to wash back into the bath.

The die temperature must be completely up to the set point and lined out. If the die temperature is not stabilized, the potential for problems is increased. Using the recommended die temperature profiles will provide the proper amount of exotherm from the very beginning of the run. There is no need to start out "low and slow" to allow the resin exotherm to heat the middle portion of the die.

A good rule of thumb for most parts, is to start off at about 10-12 inches per minute until cured stock is in the grippers to minimize the loads imposed on the dry reinforcement. Depending on the conditions, increases in speed can be made gradually. Because of the fixed cure rate of the epoxy resin, processing speeds normally do not exceed 18 inches per minute. Typically, 12-14 inches per minute will provide the best combination of pull loads vs. surface gloss.

When using R.F. pre-heating, start-up *without* the R.F. at a reduced speed (6-8 inches per minute) to minimize pull loads. When cured stock is in the grippers, gradually introduce the R.F. pre-heater. Monitor the temperature of the resin entering the die, and as it increases to about 160F, gradually increase the processing speed. Do not recycle the resin squeezed off at the die entrance to the resin bath, as this will greatly affect the resin bath temperature and severely shorten the pot life.

Trouble Shooting

The most common problem encountered with epoxy pultrusion is poor surface finish or sloughing. When this occurs, it is often because of one of the two variables we discussed earlier. Either the reinforcement level is too low, or the die temperature (cure rate) is too low. The cure for low reinforcement level is obvious. On the other hand if the reinforcement level is already within the target values, and additional reinforcement increases the loads beyond reason, most likely the die temperature profile is too low. The GELSTAR¹ Thermal Analyzer can be used to obtain a graphic picture of the die temperature and internal temperature profile. With a graphic profile of the die temperature, one is able to assess the temperatures accurately, and to determine the size of the "gel zone." The lag time between the die and internal temperature profiles is proportional to the size of the "gel zone." If the temperatures appear to be within limits, but the lag time is too large, then the processing rate is too fast for the cross-section thickness. Either reducing the processing rate, or using R.F. preheating will alleviate the problem by reducing the size of the gel zone.

When sloughing is encountered, often by correcting the aforementioned variables, the part will automatically clean itself up. Contrary to popular belief, the typical purge techniques can be used with epoxy resins. However, at times there may be a particularly stubborn portion that will refuse to clean up. Inserting a copper "Chore Boy" in the area of the part will often push the offending plug out, or catch onto it and pull it out. The remaining portion of the trail of copper gauze will provide a mild scrubbing action that will clean the surface of the die.

¹GELSTAR is a product trademark of Pultrusion Technology, Inc., Twinsburg, OH.

Shut Down

Normal polyester shut down procedures work very well for the epoxy resin. Simply empty the resin bath or remove the reinforcement from passing through it, and pull all the dry reinforcement completely through the die. Do not cut out any of the reinforcement. If the reinforcement volumes were correct, and no sloughing tendencies were encountered during the run, the die will be free from build up and ready for restart.

The resin drained from the bath should be placed in an area with good ventilation and spill protection. Fill the containers only half way. When it "kicks-off," it will become hot, expand and may overflow the container if it is too full.

CONCLUSION

With the clear understanding of how the epoxy resins differ inherently from the polyester resins, it is easy to understand why certain provisions must be made for the epoxy in the pultrusion process. We have revealed the importance of having the proper reinforcement level and die temperatures, and how those parameters affect the internal dynamics through volumetric shrinkage. Guidelines for target fiber volumes, die temperature profiles, and procedures for optimizing resin pot life have been established. Start up procedures, trouble shooting tips, and shut down procedures have been outlined. With this information, the first time epoxy pultruder will be able to successfully produce an advanced composite for the

TABLE 1. Target Fiber Volume Ranges for Epoxy Pultrusion.

ALL GLASS ROVING REINFORCED COMPOSITES	
- MULTI-END TYPE ROVINGS	75-78 % By Weight
- SINGLE END TYPE ROVINGS	79-81 % By Weight
GLASS ROVING AND CONTINUOUS MAT REINFORCED COMPOSITES	
- 1/8" THICK CROSS-SECTIONS	64-67 % By Weight
- 1/4" THICK CROSS-SECTIONS	71-74 % By Weight
CARBON FIBER REINFORCED COMPOSITES	
- ALL UNI-DIRECTIONAL TOWS	67-74 % By Weight (57-65 % By Volume)

RP industry with a minimum amount of developmental costs.

ACKNOWLEDGEMENTS

The author would like to acknowledge Joseph E. Sumerak of Pultrusion Technology, Inc. for permitting the use of his valuable work to help further the understanding of the pultrusion process throughout the pultrusion RP industry.

REFERENCES

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BIOGRAPHY

G. A. Hunter

G. A. Hunter is a Technical Associate for Shell Development Co. His background includes 13 yrs. of extensive experience in the technical service field of various RP industries. Those industries include hand lay up and filament winding with vinyl ester and epoxy resins, polymer concrete R&D, and epoxy pultrusion R&D and technical service. In addition, he has constructed and pilots an all composite experimental aircraft.

TABLE 3. Two Part Batching for Long Production Runs.

PART "A" *		PART "B"
(IN ORDER OF ADDITION)	(PHR)	EPON CURING AGENT® 9360
1) EPON® Resin 9310	100.00	33 PHR TO THE RESIN
2) EPON CURING AGENT® Accelerator 537	0.67	OR
3) HMD HIZ INT-1846	0.70	RECOMPUTE FOR PART "A"
4) ZYLAC 907	0.40	$\left(\frac{33}{121.77}\right) 100 = 27.1$
BLEND THE ABOVE FOR 30 SEC., THEN ADD CLAY		AND USE
5) ASP-400P	20.0	27.1 PHR TO PART "A"
BLEND CLAY FOR NO MORE THAN 5-10 MINUTES		
TOTAL 121.77		

* PART "A" IS STABLE FOR UP TO 3 DAYS - AFTERMIDDN SOME LOSS OF REACTIVITY MAY OCCUR

TABLE 2. Single Batch Blending Order for Short Runs.

EXAMPLE 1 (PHR)	COMPONENTS (IN ORDER OF ADDITION)	EXAMPLE 2 (PHR)
100	EPON® Resin 9310 ¹	100
0.67	EPON CURING AGENT® ¹ Accelerator 537	0.65
0.7	BYK -A 501 ²	--
0.7	HMD HIZ INT 18-46 ³	0.7
--	ZYLAC 907 ⁴	0.4
BLEND THE ABOVE COMPONENTS FOR 30 SECONDS, THEN ADD FILLERS		
20	ASP-400P ⁵	30
BLEND CLAY FOR NO MORE THAN 5-10 MINUTES TO MINIMIZE HEAT BUILD UP		
33	EPON CURING AGENT® 9360 ¹	33
BLEND IN CURING AGENT ONLY WHEN EQUIPMENT IS READY FOR START-UP		

¹ SHELL CHEMICAL CO. ² AXEL PLASTIC RESEARCH LABS, INC. ³ ENGELHARD, MINERALS AND CHEMICALS DIV.

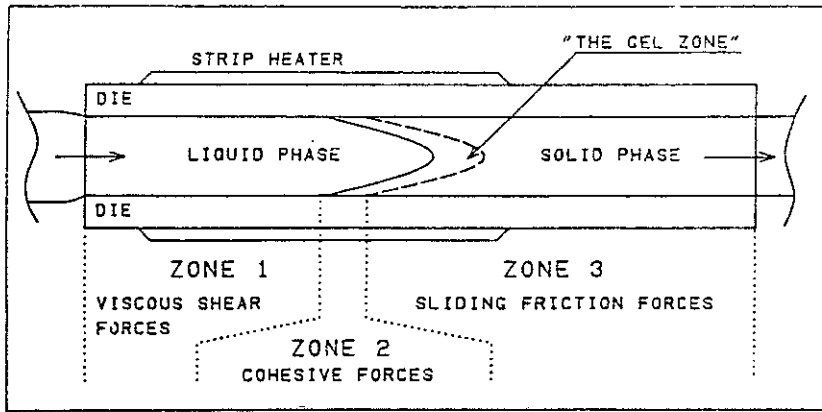


FIGURE 1. Three zone model of the pultrusion process.

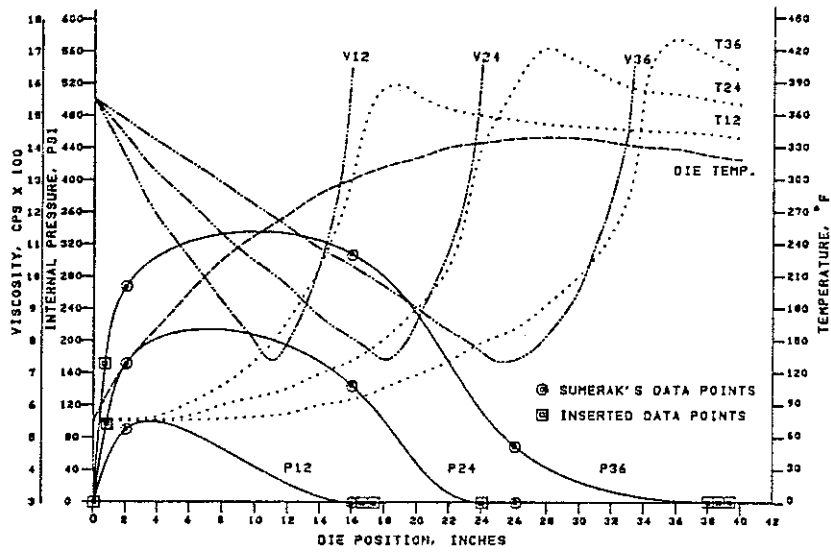


FIGURE 2. Sumerak's temperature/pressure/viscosity curves for Reichold 31-020 with 20 phr clay processed at 12, 24, and 36 inches/min.

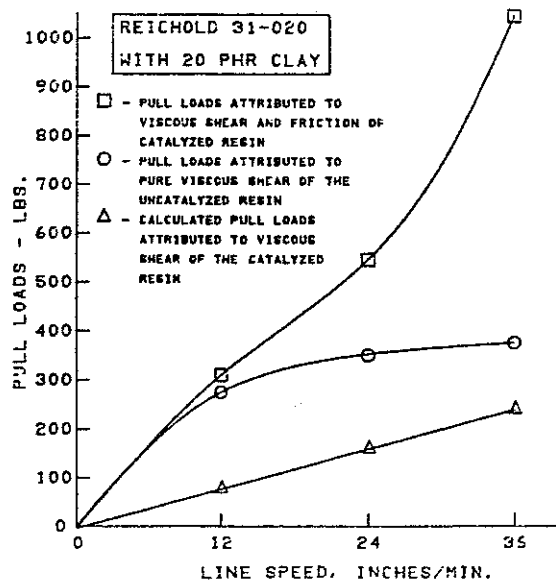


FIGURE 3. Pull loads vs. line speed.

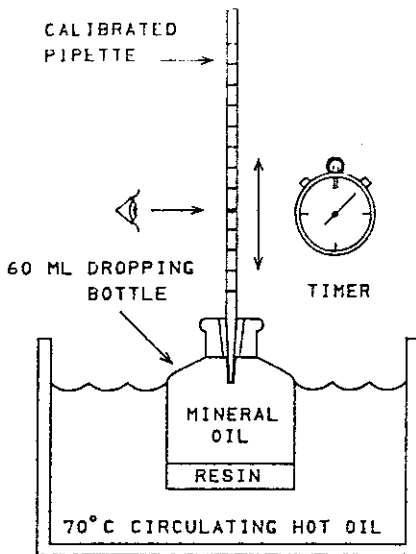


FIGURE 4. Volume change test apparatus.

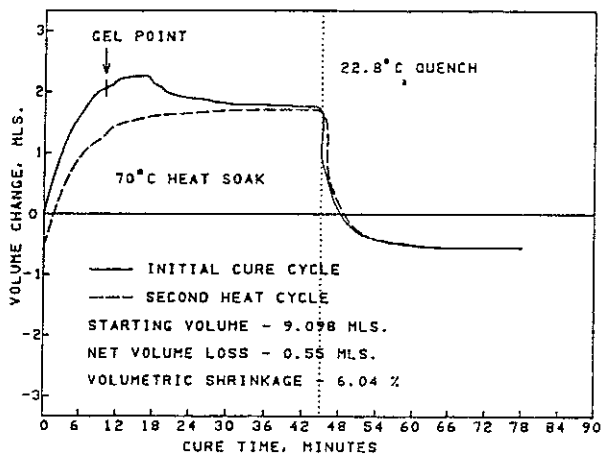


FIGURE 5. Volume change of polyester resin during cure.

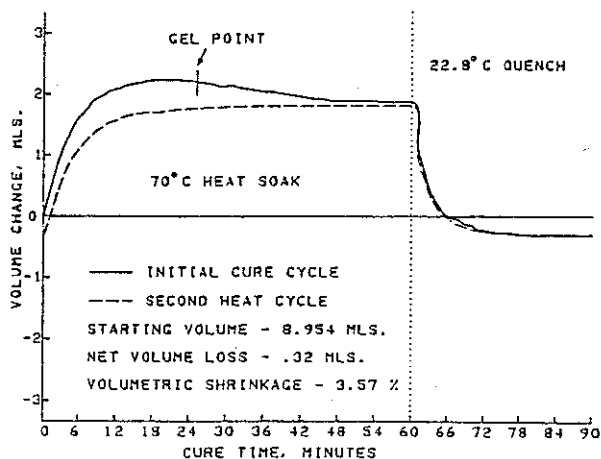


FIGURE 6. Volume change of epoxy resin during cure.

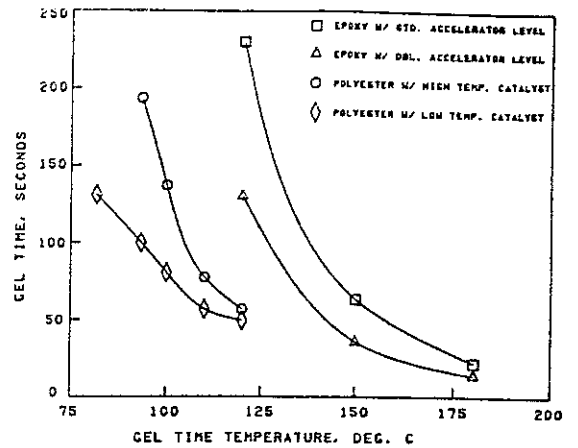


FIGURE 7. Gel time vs. temperature of epoxy and polyester resin.

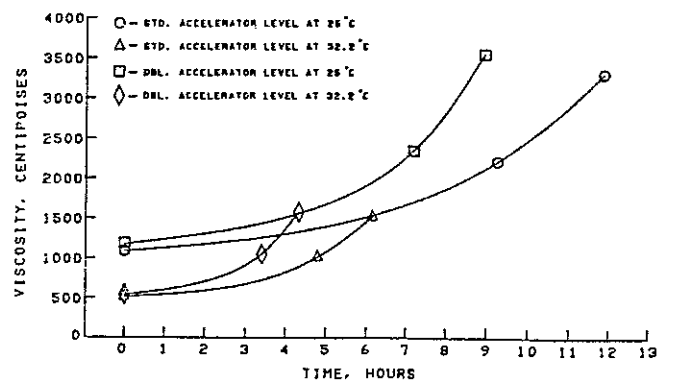


FIGURE 8. Epoxy resin viscosity vs. time and temperature.

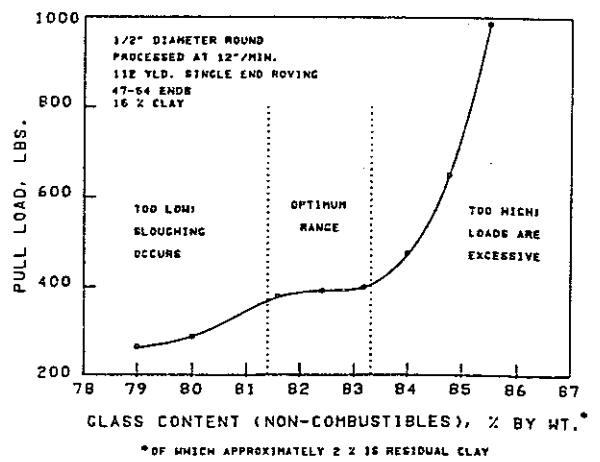


FIGURE 9. Epoxy resin pull loads vs. glass content.

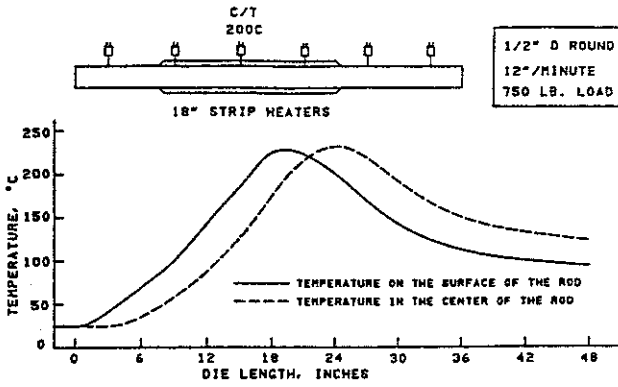


FIGURE 10. Die temperature profile of single zone heating.

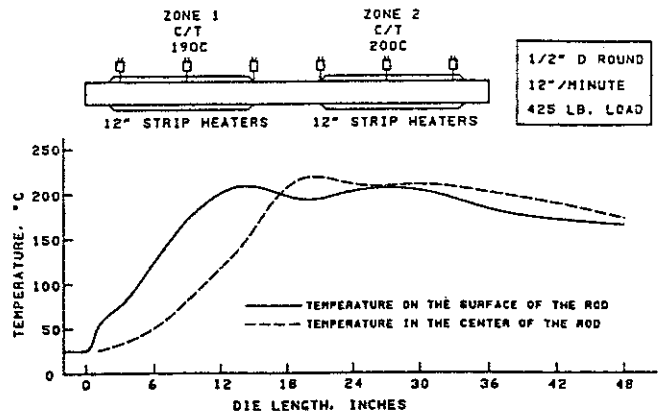


FIGURE 12. Die temperature profile of two zone heating.

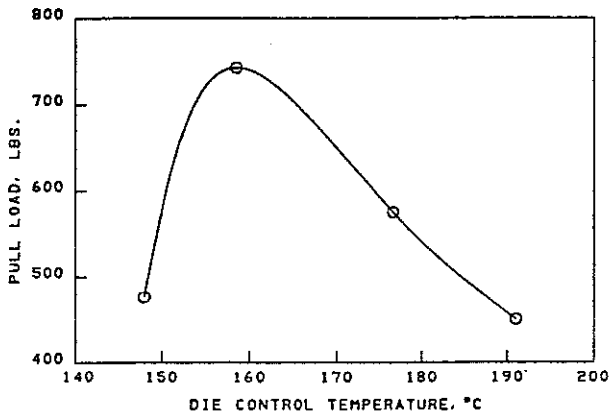


FIGURE 11. Pull load vs. die control temperature.

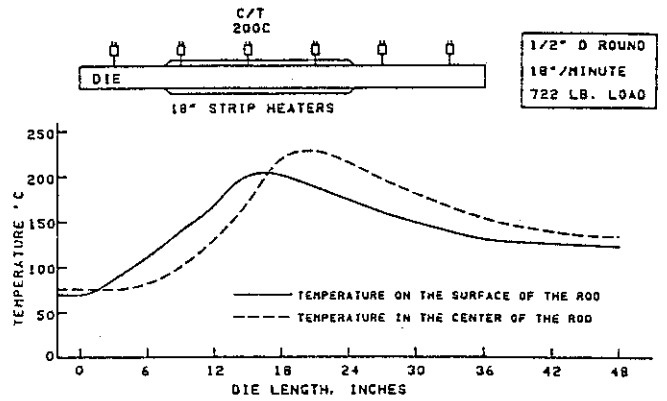


FIGURE 13. Die temperature profile with R.F. preheat.

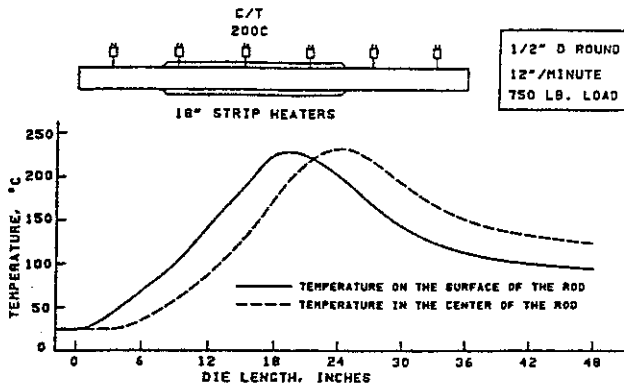


FIGURE 10. Die temperature profile of single zone heating.

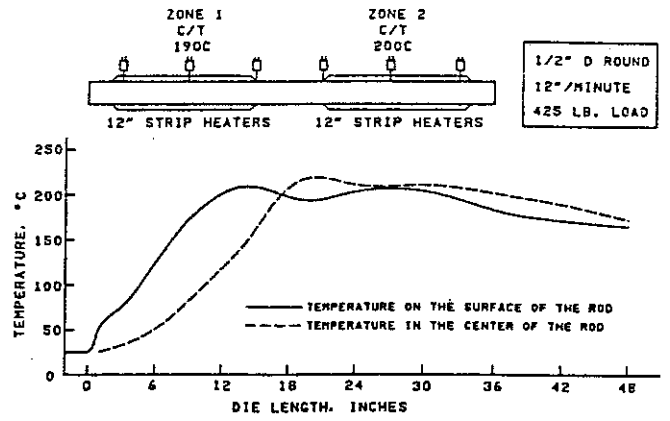


FIGURE 12. Die temperature profile of two zone heating.

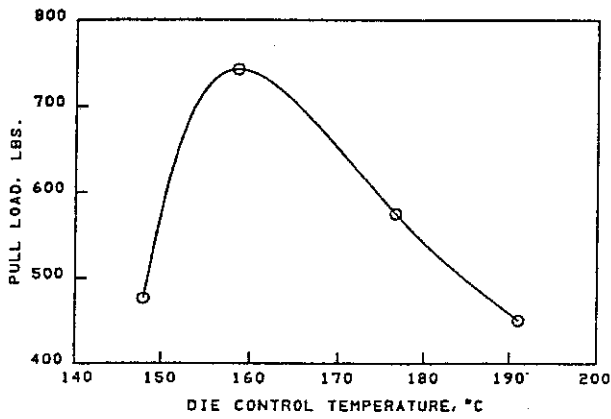


FIGURE 11. Pull load vs. die control temperature.

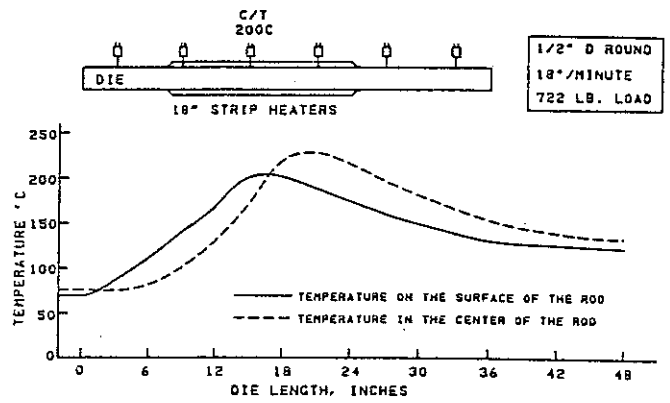


FIGURE 13. Die temperature profile with R.F. preheat.