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# Some Surface Chemical Aspects of Glass-Resin Composites

## Part 2 - The Origin and Removal of Microvoids in Filament-Wound Composites

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Chemistry Division*

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Washington, D.C.

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Previous work of this program established that a very considerable number of air microvoids are entrained in the glass roving during the winding of glass-resin composites. Air entrapment results from the difficult capillary penetration of resin into spaces between filaments during the impregnation process. The poor resin wettability of commercially finished filaments aggravates the situation. Microscopic examination has shown that the microvoids observed in glass-filament rovings are also present in cured, filament-wound composites, prepreg roving, and glass-resin laminates. The voids are present to the extent of  $10^7$  to  $10^9$ /cu in., are about 0.4 mil in diameter, and have lengths from 0.4 to 20 mils and even greater.

A microscopic examination was made of the process of resin impregnation into a strand of glass filaments under simulated winding conditions with particular attention given to the effect of the operating variables on air entrapment. When the contact angle of the resin against the individual filaments was nonzero the void content was too high to be measurable, even at low resin viscosities and slow strand speeds. On the other hand, reducing the contact angle to zero or causing the tension to oscillate as the strand passed through the resin markedly reduced the number of voids. A combination of good wetting and oscillating strand tension gave better results than either alone. These observations can be explained in terms of the forces that obstruct the release of the air bubbles from between filaments.

#### INTRODUCTION

In the first report of this series (1) it was shown that resin-impregnated glass-filament roving taken from a winding machine contains a large number of air microbubbles trapped between the individual filaments. It was suggested that these air bubbles were entrapped during the impregnation process because the resin is unable to completely displace the air from between the individual filaments. The viscous resistance to flow in the narrow spaces between filaments is too great to allow the resin to completely penetrate the roving as the latter enters the liquid, especially at the high winding rates normally employed. Impregnation is made even more difficult by the high contact angle of epoxy liquids against the commercially coated filaments presently in use. Measurements of the contact angles for various epoxy-monomer and epoxy-resin liquids against some commercial filaments were generally between 60 and 80°(1).

The purpose of the present work was threefold: (a) to determine if the microvoids observed in filament roving are also present in the wound and cured composite material, (b) to determine experimentally how these air bubbles originate and, (c) to suggest means of eliminating them from filament-wound composites.

#### THE DIRECT OBSERVATION OF MICROVOIDS IN COMPOSITES

Microscopic inspection of various filament-wound and cloth-laminate glass-resin composites revealed interfilament air bubbles in a density of  $10^7$  to  $10^9$ /cu in. and having a diameter of 0.4 mil and lengths ranging from 0.4 mil to over 20 mils. The photomicrographs in Figs. 1 and 2 are typical of the general appearance of almost all composites examined, which included both commercial and laboratory-prepared materials. These observations were made with a light microscope using transmitted illumination without a substage condenser. The optical contrast between bubbles and filaments could in some instances be improved by applying to the specimen surface a thin film

<sup>1</sup>NRL Problem C03-19; Project RR 001-02-01, ONR Contr. Nonr PO 4-0075, Task NR 356-459 (Technical Report No. 2). This is an interim report; work on this problem is continuing. Manuscript submitted March 5, 1965.

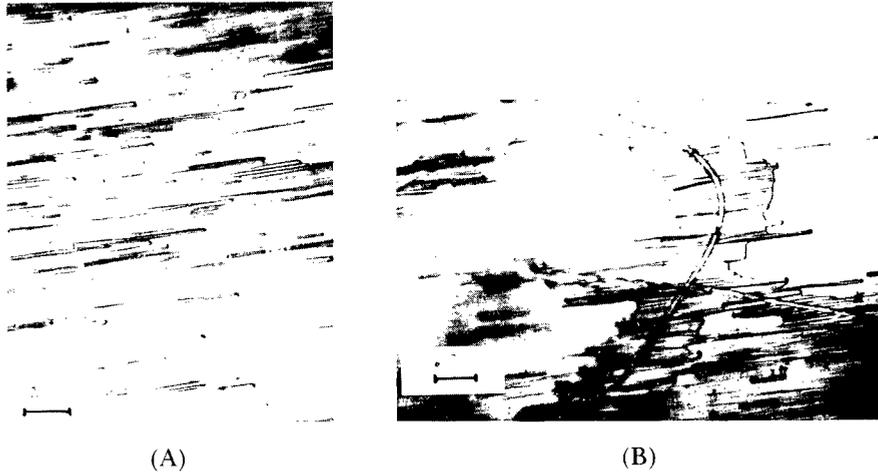


Fig. 1 — Photomicrographs of microvoids in glass-resin, filament-wound, NOI-ring segments: (A) ring segment wound without misaligned filaments, (B) ring segment wound carelessly

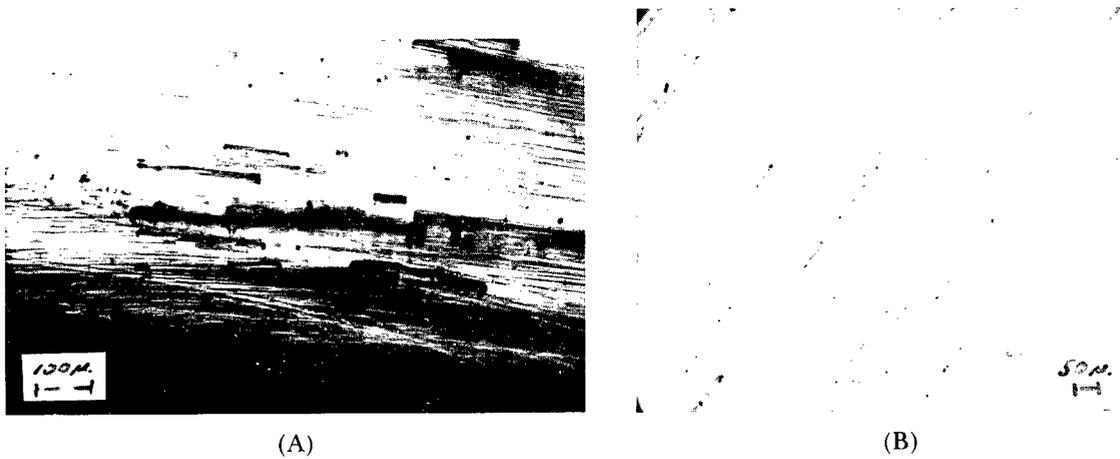


Fig. 2 — Photomicrographs of (A) a section of prepreg roving and (B) a fiber-glass cloth-resin laminate

of liquid having a refractive index close to that of the resin. The liquid film reduced light scattering by the rough surface of the sample.

The photomicrographs deserve some individual comment. The specimens of Figs. 1(A) and 1(B) were segments of NOL rings (2) wound at this Laboratory using HTS-finish E-glass (Owens-Corning Fiberglas Corporation) and m-phenylenediamine-bisphenol A epoxy resin. The ring segment of Fig. 1(A) was carefully wound to avoid misalignment of filaments, but no such precautions were taken in constructing the ring segment in Fig. 1(B). Clearly, careful winding did not prevent the entrapment of microvoids, but the presence of misaligned filaments made the situation worse by allowing the air bubbles to accumulate into much larger voids. The appearance and density of the microvoids was much the same throughout the segments. This fact was determined by filing into the material to various depths. The photomicrograph of Fig. 2(A) is typical of the appearance of a commercial prepreg roving, and Fig. 2(B) is typical of a commercial glass-cloth-laminate material. Both composites were constructed from epoxy resins and silane-finished glass filaments. In Fig. 2(B) the air bubbles in the underlying laminate layer can be seen as faint shadows perpendicular to the direction of the bubbles in the layer in focus. Also, there are a few voids in Fig. 2(B) which appear to be filled with small, dark specks. These voids were at the surface of the laminate in the resin covering the first layer of filaments. In the course of handling

the material, these bubbles broke open, leaving a hole into which resin particles and dust had accumulated.

An interesting aspect of these microvoids is that in many instances the filaments passed through the void as in Fig. 3 where three filaments are exposed.



Fig. 3 - Photomicrograph of air void extending across three adjacent glass filaments

### THE IMPREGNATION PROCESS

To determine how the microvoids are entrapped during the winding process, an apparatus was constructed which permitted microscopic observation of a single end or strand of filaments as it passed into and through a cell containing the impregnating liquid. The apparatus is illustrated schematically in Fig. 4. The strand was held in tension by a magnetic hysteresis brake (A), pulled through the liquid over pulleys (B), and fed onto a

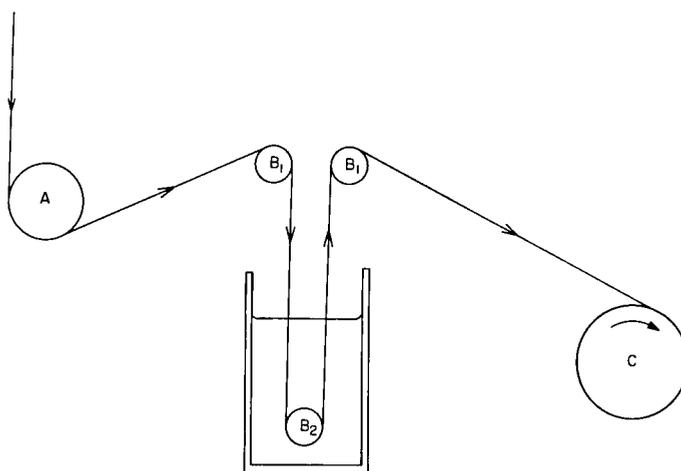


Fig. 4 - Schematic diagram of single-strand impregnating device:  
(A) hysteresis brake, (B<sub>1,2</sub>) pulleys, (C) windup reel

windup spool driven by a variable-speed motor. The path length through the resin bath was about 4 in. A microscope fitted with a long-working-distance objective was used to examine the submerged strand at various positions. Photomicrographs were taken of the strand in motion using an electronic flash technique. The strand used in all experiments described here was the HTS-finish E-glass having 204 filaments per strand and a filament diameter of 0.00036 in.

Use of the single-end impregnation device revealed the sequence of events that took place as the strand passed into and through the liquid. These processes are illustrated in Fig. 5(A) and 5(B) for the impregnation of HTS-finish E-glass with bisphenol A diglycidyl ether (Dow Corning DER 332) at room temperature. Photography was assisted by the fact that the movement of the bubbles was relatively slow in this viscous liquid (ca 5000 cp) and at the slow strand speed used (ca 3 in./min). However, the qualitative aspects of the impregnation in this sluggish system were no different from the sequence of events in less viscous liquids and at higher strand speeds. Photomicrographs 5(A) were taken through the liquid just below the point where the strand



Fig. 5 — Electronic flash photomicrographs of air entrapment in a single strand by an impregnating liquid: (A) strand entering the liquid, (B) strand appearance during passage through the liquid

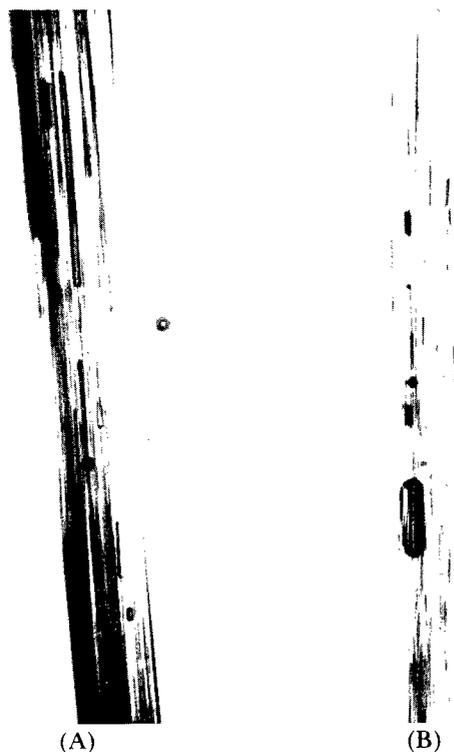


Fig. 6 — Photomicrographs of single strands just prior to emerging from the liquids having different contact angles against the filament: (A) contact angle  $60^\circ$ , (B) contact angle  $0^\circ$

entered the liquid. In photographs A-1 and A-2 air that the resin had not displaced appears as dark threads extending down from the funnel-shaped depression produced in the liquid surface by the entering strand. In photograph A-3 a relatively large mass of air can be seen clinging to the outside of the strand. Photomicrographs B-1, B-2, and B-3 (Fig. 5) were taken at various positions below the liquid surface and show the entrapped air coalescing into distinct bubbles. In Fig. 6 the strand has been stopped and the bubbles are seen to be identical in shape and size with the ones that were found in the final, cured composites (Figs. 1 and 2).

Once coalesced, the bubbles were more-or-less mobile in the direction of the filament axis, depending on the liquid viscosity and more importantly on strand tension and resin wettability of the filaments. For instance, when the epoxy liquid was replaced by a methyl silicone oil having about the same viscosity, the bubbles such as are seen in Fig. 6(B) were much more mobile and were seen to move freely up and down in the spaces

between filaments, especially if the tension on the strand was increased or decreased slightly. The most important difference between the epoxy liquid and the silicone oil is that the epoxy exhibits a contact angle of 60 to 80° against the HTS-finish single filaments (1), while the silicone oil was found to have a zero contact angle.

A further observation important to finding a means of eliminating voids was that lateral movement of the bubbles out of the yarn required that the contact angle be zero and that the tension on the filaments be first relaxed and then re-applied. It was observed that, if a bubble is mobile in the axial direction, relaxing the tension allows it to pull up into a more nearly spherical shape. When the tension is reapplied the filaments push the bubble out into the surrounding liquid.

Quantitative determinations were made of the effect of the resin properties and different winding conditions on air entrapment. The procedure was to pull the single end through the liquid for about 5 min to allow the processes of entrapment and release to reach a steady state. The strand was then stopped and a count made of the number of voids that could be seen through the microscope in one field of view at a point 1/4 to 1/2 in. from where the yarn leaves the liquid. The entire procedure was repeated a total of ten times and an average taken. The field of view included about a 1/16-in. length of strand, and the count was reported as voids/in. of strand. The reproducibility was about 25% for counts of less than 500 voids/in. of strand. This low precision, which was worse at larger void densities, makes the method useful only as an indication of which variables have a strong effect on void density.

It became evident almost immediately that the void count was unmeasurably high unless the contact angle for the liquid against the filament was zero or the tension on the strand was periodically reduced as it passed through the bath. These facts are illustrated in Table I for a single end of HTS-finish glass filaments passing through an alicyclic epoxy liquid UC-0400 (bis[2,3-epoxycyclopentyl]ether, Union Carbide Plastics Co.). At a constant tension of 60 g the count was greater than 1000 voids/in. of strand, and Fig. 6(A) illustrates the appearance of the strand. Addition to the liquid of an agent that reduced the surface tension to 25.6 dynes/cm and the contact angle to

near zero reduced the count to about 400 voids/in. of strand. Similarly, a methyl silicone polymer oil (DC-200, Dow Corning Corp.), having a viscosity slightly higher than the 0400 epoxy and a zero contact angle against the filaments, also gave a count of about 400 voids/in. of strand.

The effect of releasing voids by alternately relaxing and reapplying the strand tension was further studied by allowing the strand to slip over the brake wheel so that the tension oscillated between 20 and 100 g at 1 oscillation/sec. This condition lowered the void content considerably from when the tension was kept constant. In fact, the reduction in the void count by oscillating the tension was comparable to the reduction in the count when the strand was under constant tension in a zero-contact-angle liquid.

The greatest air removal was obtained when the contact angle was zero and the strand tension was also allowed to oscillate. This combined effect reduced the void count by more than one half from that obtained with either good wetting or oscillating tension alone. It is possible that there are an amplitude and frequency in oscillating tension that give an optimum in air release, but the present apparatus does not permit a study of these variables.

The effects of differences in liquid viscosity, strand speed, and strand tension were also studied. In Table I it can be seen that when the contact angle was zero, only changes in strand tension had a demonstrable effect on void count. In other studies with various epoxy liquids, all of which had large contact angles against the filaments, it could be observed that the greater the resin viscosity or the faster the strand speed, the greater the air entrapment. However, in these experiments the void density was too high to permit a void count, so differences could only be judged qualitatively. These same differences in air entrapment at different resin viscosities and strand speeds undoubtedly occur when the contact angle is zero. However, because of the low angle these voids are released to a level that is approximately the same regardless of the amount of air entrapped initially.

#### THE REMOVAL OF MICROVOIDS FROM FILAMENT-WOUND COMPOSITES

The foregoing observations suggest that an effective means of reducing the amount of

TABLE I  
The Influence of Resin Properties and Operating Conditions on Void Content

Liquid	Viscosity (cs)	Surface Tension (dynes/cm)	Contact Angle (deg)	Strand Speed (in./min)	Strand Tension (g)	Void Count (voids/in. of strand)
Effect of Contact Angle						
0400*	33	42.0	60	300	60	> 1000
0400 + 5% A†	33	25.6	0	300	60	400
silicone oil‡	50	20.8	0	300	60	380
Effect of Oscillating Tension and Contact Angle						
0400	33	42.0	60	300	20-100	330
0400 + 5% A	33	25.6	0	300	20-100	150
silicone oil	50	20.8	0	300	20-100	80
Effect of Viscosity						
silicone oil	50	20.8	0	300	60	384
silicone oil	350	21.1	0	300	60	352
silicone oil	1200	21.2	0	300	60	512
Effect of Strand Speed						
silicone oil	350	21.1	0	7	60	368
silicone oil	350	21.1	0	300	60	352
silicone oil	350	21.1	0	720	60	348
Effect of Strand Tension						
0400 + 5% A	33	25.6	0	300	5	512
0400 + 5% A	33	25.6	0	300	60	384
0400 + 5% A	33	25.6	0	300	100	272

\*bis(2,3-epoxycyclopentyl)ether (Union Carbide Plastics Co.).

†surface-active agent "A".

‡DC-200 polymethylsiloxane (Dow Corning).

entrapped air would be to provide that the resin have a zero contact angle against the glass finish and to oscillate the tension on the strands as they pass through the resin bath. It is doubtful that air entrapment can be prevented. In Part I (1) it was pointed out that if the strand is compared to a bundle of capillaries in which the resin must rise at a rate equal to the rate at which the strand is entering the liquid the maximum possible rate of capillary rise was three orders of magnitude too slow for the resin to penetrate the entering strand axially. It was anticipated in the earlier report and was demonstrated here that once the strand is in the bath lateral movement of the liquid into the strand traps the undisplaced air. The entrained air then pulls up into discrete bubbles in an effort to minimize surface area.

The release of air while the strand is submerged is opposed by the lateral forces on the filament holding the bubble within the strand and the fact that when the contact angle is nonzero many of the bubbles are attached to the filaments. It is the lateral component of tension on the filaments that constrains the bubble in their elongated shape. These lateral forces arise at least in part from the twist of the strand. When a strand passes over a pulley the lateral forces are redistributed and provide an opportunity for the air to be squeezed out. However, there appears to be a limit to the amount of air released by this squeezing process, and this limit is easily reached but not exceeded when the contact angle is zero. There was no measurable reduction in void count by increasing the number of pulleys in the bath from one to

three, by altering the curvature of the pulley surface, or by preventing the pulley from revolving and thus causing the strand to slide around the pulley. Postulating that there is a limit in air release by squeezing provides an explanation for the observation that in zero-contact-angle systems the strand speed and liquid viscosity had little effect on void count but that the count could be further reduced by increasing strand tension.

A major reduction in void count could be realized when the strand was relaxed to allow the bubbles to reduce their surface area further by pulling into a more nearly spherical shape. Once having done so, the bubbles resisted being reshaped when the tension was reapplied and so were expelled from the strand. This process of expelling air by relaxing and reapplying tension is hindered if the bubbles must be detached from the filaments, which is the case when the contact angle is nonzero. A measure of the detaching force is given by the work the liquid must do to displace air from the filament surface ( $W_D$ ) and is equal to the difference between the work of adhesion of the liquid to the finish coating on the glass fibers and the work of cohesion of the liquid itself (3):

$$W_D = W_C - W_A. \quad (1)$$

The surface energy relations for  $W_C$  and  $W_A$  are

$$W_C = 2\gamma_{LV} \quad (2)$$

$$W_A = \gamma_{LV} (1 + \cos \theta) + f_{SV} \quad (3)$$

where  $\theta$  is the contact angle,  $\gamma_{LV}$  is the surface tension of the liquid, and  $f_{SV}$  represents the reduction in the surface energy of the filament coating due to adsorption from the vapor. Since the surface energy of the silane-derived coating on this HTS-filament would be extremely low,  $f_{SV}$  is assumed negligibly small. Combining (2) and (3) we see that the work required for displacement is positive

$$W_D = \gamma_{LV}(1 - \cos \theta)$$

when the contact angle is nonzero. When the angle is zero,  $\cos \theta = 1$ , and the work for displacement is zero so that the bubble is spontaneously detached.

Once the strand has left the resin bath, the release of air bubbles becomes considerably more difficult. The surface tension of the resin on the strand compresses the filaments and voids into a tight compact. Even if a bubble does reach the outside of the bundle of filaments, its release requires the rupture of a thin film of liquid; and although the process is spontaneous, it is slowed by the viscous resistance of the liquid to flow and the need for a solid particle, such as a dust speck, to nucleate the film rupture.

The problem of applying a fluctuating tension to the glass strands during the winding operation is the subject of current research at this Laboratory. The task is made difficult by the fact that in the filament-winding operation the glass must be applied to the mandrel at constant tension. Thus, any variation in tension during impregnation must be removed from the strand after it leaves the resin bath. This is essentially an engineering problem and is beyond the scope of this project. However, NRL personnel charged with studying the process engineering of filament winding are presently building and testing equipment designed to apply a fluctuating tension during the winding of test composites.

Having demonstrated here that the number of voids in composites can be significantly reduced if the contact angle is zero, it is imperative to find ways of overcoming the inherent poor resin wettability of the commercially finished glass filaments presently available. Two possible approaches to improved wetting are to replace the present coating with finishes that are more readily wet by epoxy liquids or to add surface-active agents to the resin that will promote better wetting. Research is now in progress both on new finishes for glass filaments and in finding useful surfactants, and these topics will be the subject of future reports. It already has been mentioned (1) that many additives exhibit the anomalous behavior of reducing the surface tension of the epoxy liquids without reducing the contact angle against the finish surface. Only one resin additive has been found that effectively reduces the contact angle, the material designated "A" in Table I. This additive was found to be effective in the alicyclic resin liquid but not in the aromatic epoxy monomer, bisphenol A diglycidyl ether. The chemical constitution of this additive is not

fully known, and the reason for its unique behavior is as yet undetermined.

### SUMMARY

The microscopic examination of glass-resin composite materials has shown the presence of microvoids in densities of  $10^8$ /cu in. or greater in filament-wound structures, prepreg roving, and laminate materials. In carelessly wound composites these voids gather into large void spaces around misaligned filaments.

A bench-scale single-strand impregnation device was constructed to permit microscopic observation of a strand of filaments passing into and through a liquid bath. It was determined that the entrapment of air cannot be prevented even at relatively low liquid viscosities and slow strand speeds. The void content is considerably reduced if the liquid has a zero contact angle against the filament surface and if the strand tension is fluctuated between 20 and 100 g. at about 1 oscillation/sec. Under these conditions, liquid viscosity and strand speed have relatively little effect on void content. Evidently, when the contact angle is zero the bubbles of entrapped air are detached from the filaments and are sufficiently mobile to be easily released as the strand moves through the bath. Release is

hindered when the contact angle is not zero because of the work necessary to displace air from the filament surface. In the zero-contact-angle systems under steady tension there appears to be a lower limit to which the void content can be reduced. This limit is relatively unaffected by changes in viscosity, strand speed, or the number of pulleys around which the strand passes. The limit in air release seems to be set, in part at least, by the strand tension. The alternate release and reapplication of strand tension permits the bubbles to pull up into more spherical shapes and thus be squeezed out more easily. This effect, combined with good wetting, gave the lowest void count. Work is now in progress to use these findings in the actual winding of glass filament-epoxy resin composites.

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13. ABSTRACT <p>Previous work of this program established that a very considerable number of air microvoids are entrained in the glass roving during the winding of glass-resin composites. Air entrapment results from the difficult capillary penetration of resin into spaces between filaments during the impregnation process. The poor resin wettability of commercially finished filaments aggravates the situation. Microscopic examination has shown that the microvoids observed in glass-filament rovings are also present in cured, filament-wound composites, prepreg roving, and glass-resin laminates. The voids are present to the extent of <math>10^7</math> to <math>10^9</math>/cu in., are about 0.4 mil in diameter, and have lengths from 0.4 to 20 mils and even greater.</p> <p>A microscopic examination was made of the process of resin impregnation into a strand of glass filaments under simulated winding conditions with particular attention given to the effect of the operating variables on air entrapment. When the contact angle of the resin against the individual filaments was nonzero the void content was too high to be measurable, even at low resin viscosities and slow strand speeds. On the other hand, reducing the contact angle to zero or causing the tension to oscillate as the strand passed through the resin markedly reduced the number of voids. A combination of good wetting and oscillating strand tension gave better results than either alone. These observations can be explained in terms of the forces that obstruct the release of the air bubbles from between filaments.</p>		

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Microvoid Glass-resin composites Roving Filament Laminate Contact angle Strand tension Capillarity						

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