KRÜSS

Application Report

Wetting in composite fabrication

Application report: AR248e
Industry section: Textiles, fabrics, electronics
Author: Dr. Christopher Rulison
Date: 2005

Drop Shape Analysis System DSA10

Force Tensiometer – K100

Method:

Keywords: glass fabric, pore wetting, interfacial tension, micro-void, lead frame, circuit board, composite, non-woven, epoxy resin, viscosity

Interfacial Tension as a Predictor of the Completeness of Pore Wetting in Epoxy Resin Impregnated Non-Woven Glass

Abstract

The manufacture of circuit boards is a major application for woven glass fabric. The glass fabric is impregnated with epoxy resin and cured to produce a board onto which circuitry is placed. The glass fabric is commonly treated by the glass manufacturer (who is usually a supplier to the actual board producer) with a coating that makes it wet well with epoxy resin and also usually has some functionality which reacts chemically with the epoxy. Acrylic, amine, and ester functionalities are common, as well as others.

Background

Of all applications for glass fabric, this is probably the most critical in terms of the control one must have over the surface properties of the glass and how those properties compare to the surface properties of the resin with which it is to be impregnated. Incompatibility between the surface properties of the resin and those of the glass causes micro-voids in the impregnation – even in cases where the glass is much higher in surface energy versus the surface tension of the resin, so that wetting is quite good, as judged in an overall contact angle sense.

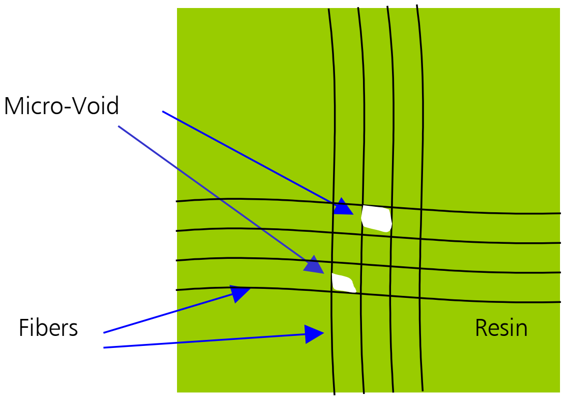

Fig. 1: schematic of micro-void

These micro-voids in wetting mainly occur at the nodes where the fibers of the glass meet, and are most often internal within the fiber bundles (or tows) wherein the pores of the glass fabric are the smallest and capillary wetting is most important. Such voids are unacceptable, and if widespread enough, cause a board to be rejected (either before or after circuitry is applied), because they affect the insulating properties of the boards. As circuitry gets printed closer and closer on these boards with new technologies, the acceptable size and of these imperfections becomes smaller and smaller. The demands of the industry are also exceeding high in terms of the lack of rejected parts, because testing all the parts that come off an assembly line is often not an option. For example, 1 rejected board in 1000 is often considered completely unacceptable and very poor quality.

One would initially think that the voids would be a function of resin viscosity at the time of impregnation (since that is going to affect the efficiency of penetrating the pores) and the contact angle between the resin and the glass surface. However, when we were challenged to develop a predictive analytical technique in terms of measurable surface properties to assist in solving this problem, we found that raw wettability and rates of penetration were both poor predictors of the occurrence of voids. However, resin/glass surface (liquid/solid) interfacial tension, as calculated based on the surface properties of the glass and the resin measured independently was very predictive.

Experimental section

Detailed below are the results for three different glass fiber treatments on the same type of woven glass (same weave and fiber diameters, differing only in fiber finish), along with three different epoxy resins. We have since tested literally hundreds of samples of both glass fabrics and tens of different resins from various suppliers of both, to allow them to use interfacial tension as a predictor of the minimization of such voids.

We will call the fabrics #1, #2, and #3, and the resins A, B, and C.

They had been determined to have the following failure rates per 1000 boards, identified as being due to the existence of the micro-voids.

Fabric / Resin	Resin A	Resin B	Resin C
Fabric 1 Board Failures per 1000 Boards	0.93	1.20	3.60
Fabric 2 Board Failures per 1000 Boards	0.15	0.30	1.80
Fabric 3 Board Failures per 1000 Boards	0.34	0.08	0.70

Table 1:Board failure rate for different fabric/resin compositions

We first tested the non-wovens directly for contact angle with the various resins. The time resolved wetting data from a KRÜSS Drop Shape Analysis System DSA10 were as shown below.

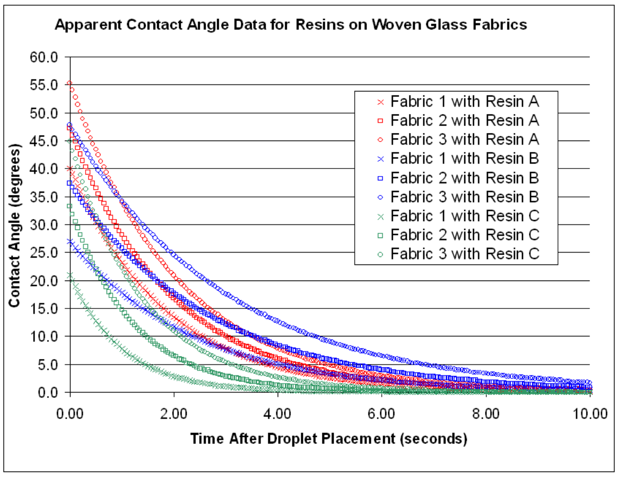

Fig. 2: Non-woven fabric contact angle

From these data we can conclude that in terms of rate of wetting resin C wets all three fabrics the most rapidly, resin B wets all three resins the least rapidly, and resin A is intermediate. However, the initial (zero time) contact angles on any given fabric follow the trend: Resin A > Resin B > Resin C.

Neither the initial data nor the rate data correlate with the board failure rates given above. For example, the following plot shows initial contact angle versus rate of board failure.

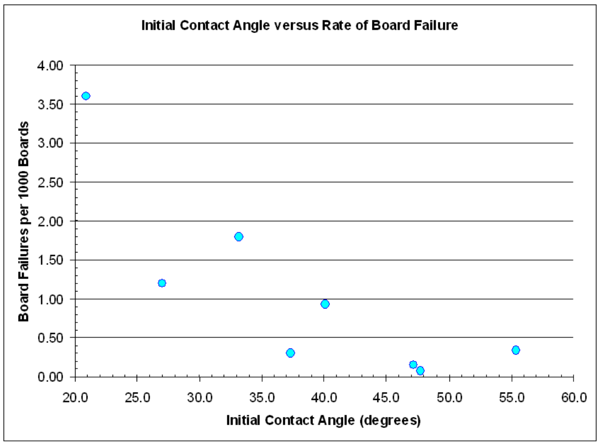

Fig. 3: Initial CA vs. board failure rate

We next measured the surface tensions of the resins and their viscosities with the following results:

Resin	A	B	C
Viscosity (cp)	19.40	36.90	12.30
Surface Tension (mN/m)	33.21	32.03	28.43

Table 2: Viscosity/surface tension of resins

These data explain the time resolved contact angle data shown above in terms of both sorption rate and initial contact angle. The resin C wets the fabrics the most rapidly, because it has the lowest viscosity, resin B wets the fabrics the least rapidly because it has the highest viscosity, and resin A is intermediate, since it has intermediate viscosity. By the same token, the initial contact angle is explained by the surface tension data – Resin A provides for the highest initial contact angles because it has the highest surface tension, Resin C has the lowest initial contact angles because its surface tension is the lowest, and Resin B is intermediate in both categories. Therefore it is apparent that initial spreading is governed by the surface tension on any given fabric, and the rate of wicking governed by the viscosity. However, none of this gives a reasonable correlation with the board failure data. If anything, the above graph gives the impression that board failures decrease with increasing initial contact angle due to surface spreading – which makes very little logical sense.

We next measured contact angles by the Washburn method for the resins against the fabrics using one inch by one inch squares of fabric and the Washburn method as described in KRÜSS Technical Note #302e (available at www.kruss.de or www.augustinescientific.com) on a KRÜSS Force Tensiometer – K100. But, for every resin/glass fabric combination the contact angle was found to be very close to zero degrees. So, we decided to measure the fabrics for surface energy by the Washburn method using diiodomethane and water as probe liquids and the Fowkes theory. The Fowkes theory is described in detail on pages 10-12 of KRÜSS Technical Note #306e (available at www.kruss.de or www.augustinescientific.com).

Again we employed the Washburn technique on one inch by one inch squares of fabric for the contact angle measurements with diiodomethane and water as shown schematically below.

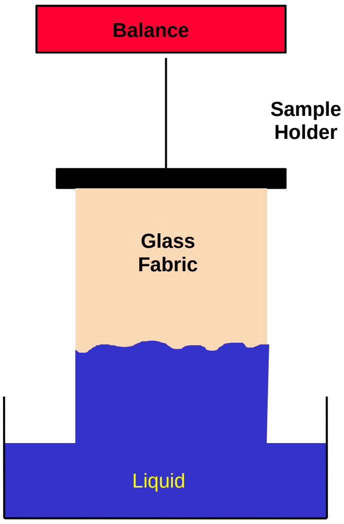

Fig. 4: Schematic of CA measurement

However, it should be noted that in further work with a variety of glass fabrics we have employed other liquids in many cases – particularly in cases where the fabrics were more hydrophobic at the surface than the ones being discussed currently. We have also employed fabric packing methods for sorption experiments on fabrics that were too low in capillarity to wick liquid far enough up to saturate the sample in standard Washburn techniques. A typical packing method for such a sample is to cut circles of the fabric (using a circular punch) which neatly fit into a standard KRÜSS Fiber Cell (SH0620) and to pack perhaps 50 to 100 such circles into the fiber cell for each wicking test. Some of our customers prefer this, in that it wets the fabric in the perpendicular direction, as the resin actually wets the fabric in application, rather than in the parallel direction up the fibers. However, the single square of sample is easier and more rapid, if the fabric will wet this way. The following surface energy data were determined for the fabrics discussed here using the Washburn method with water and diiodomethane.

Fabric	1	2	3
Overall Surface Energy (mJ/m^2)	43.59	38.91	35.70
Polar Component (mJ/m^2)	10.95	7.75	5.69
Dispersive Component (mJ/m^2)	32.64	31.16	30.01
Surface Polarity (%)	25.12	19.92	15.94

Table 3: Surface energy data of fabrics

The resin surface tensions were also separated into polar and dispersive components by contact angle work on poly(tetrafluoroethylene) (PTFE) and the application of Fowkes theory with the following results:

Resin	A	B	C
Overall Surface Energy (mJ/m^2)	33.21	32.03	28.43
Contact Angle on PTFE (°)	73.0	69.9	59.8
Polar Component (mJ/m^2)	7.63	6.30	3.07
Dispersive Component (mJ/m^2)	25.58	25.73	25.36
Surface Polarity (%)	22.96	19.68	10.80

Table 4: Resin surface tension

Combining these data with Good's equation for the interfacial tension between a liquid and a solid (page 6 of KRÜSS Technical note #306e) yields the following interfacial tension data between the fabrics and the resins:

Fabric / Resin	Resin A	Resin B	Resin C
Fabric 1 Interfacial Tension (mN/m)	0.73	1.05	2.88
Fabric 2 Interfacial Tension (mN/m)	0.28	0.33	1.36
Fabric 3 Interfacial Tension (mN/m)	0.32	0.18	0.60

Table 5: Interfacial tension Fabric/Resin

The graph below shows a good correlation of these data back to the board failure value cited at the beginning of this note.

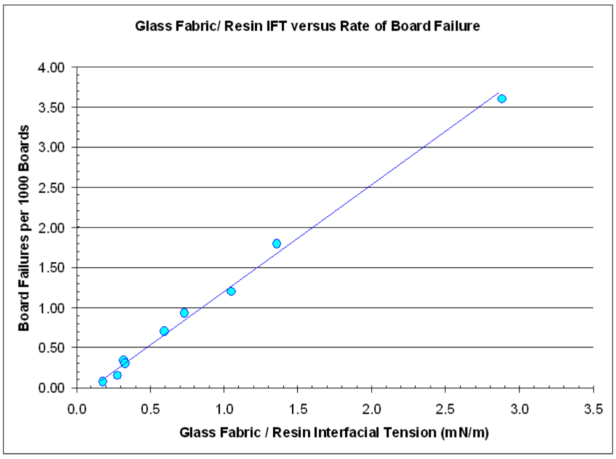

Fig. 5: Fabric/resin interfacial tension vs. board failure rate

Conclusions

There seems to be a strong and linear correlation between increasing interfacial tension and incomplete wetting issues in the case of woven glass fabrics being impregnated with epoxy resin. Because of this correlation, we have a fair number of customers who have a spectrum of either their treated glasses (in the case of a glass manufacturer) or resins (in the case of resin suppliers) pre-characterized. They then come to us for measurement of the opposite component and to calculate interfacial tension as a predictor of possible issues whenever they start working with a new glass or resin material. This gives them an idea of which of their materials to offer for that particular system which will minimize micro-voids. Most customers are looking for interfacial tensions certainly below 1.0 mN/m, and usually below 0.5 mN/m as well, to minimize board failures.

Literature

[1] Dr. Christopher Rulison, *Wettability Studies for Porous Solids Including Powders and Fibrous Materials* (TN302e), 1996

[2] Dr. Christopher Rulison, *So You Want to Measure Surface Energy?* (TN306e), 1999