ARLON ELECTRONIC MATERIALS

TECHNICAL ARTICLE

FAQ's About Moisture Control

Arlon 2013

Why is moisture an issue in prepreg.

With most high temperature, multifunctional epoxies, the effect of moisture is to reduce Tg by reacting with the resin (hydrolysis). The same rules that apply for polyimide may be applied to high temperature epoxies. While moisture technically doesn't reduce the Tg of polyimide (or interfere with crosslink density), it can later result in delamination or blistering when the finished board is post processed at high temps in soldering or leveling.

Cyanate esters are especially sensitive to moisture, and if there is water present during high temperature processing, carbamates may be formed by the reaction of water with the basic cyanate chemistry. Carbamates are "blowing agents", used commercially to make plastic and rubber foams, and are NOT desirable in MLB lamination as they may cause delamination or blistering during high temperature cures. For this and other reasons (especially an inherent propensity to resin cracking), Arlon discontinued the use of "pure" Cyanate ester systems.

MultiClad HF is a proprietary resin system that does contain some cyanate chemistry. Moisture in this product may affect the chemistry and process.

What about polyimide prepregs? Is moisture really a bigger issue with polyimides than with epoxy systems?

Polyimides are more moisture sensitive than epoxies in the sense that they will absorb water from the air more quickly. Moisture which is in the prepreg does not do the prepreg itself any harm, but may result in voids, delamination and other defects in laminated boards. Foamy flow is an indicator of moisture in prepreg. It is not exclusively a phenomenon associated with polyimide, but because polyimide picks up moisture faster than FR-4, it is seen as a problem more often. Drying any prepreg by storing at low humidity with desiccant, or vacuum drying before use will minimize this effect.

Moisture can cause additional problems in finished PWBs – the integrity of a multilayer board under high temperature stressed conditions (such as are simulated by the T288 or T300 test for materials designated "Lead-Free") can be compromised by excessive moisture. Thus it is important to control moisture even in finished bare boards prior to solder reflow or assembly processing, and even in finished boards if they are going to be subsequently exposed to high temperatures, such as when they are to be repaired, or when devices require replacement or re-soldering. Obviously this is exaggerated with lead-free solder systems due to their higher temperatures.

We recommend vacuum drying prepregs for several hours at 29” of Hg, and this will be discussed at length in the Processing section. Vacuum desiccation at room temperature is preferred over any kind of oven drying for standard epoxy or polyimide prepreg, even when you believe the oven drying is under controlled conditions and the temperature low enough not to hurt the prepreg. Oven drying may tend to advance the prepreg, and may result in marginal or insufficient flow or bond that can result in delamination or other failures on subsequent thermal processing. The only exception to that might be for non-woven aramid polyimide where a vacuum oven temperature of no more than 90˚F (32˚C) for 4-5 hours at 29” of vacuum may be helpful in removing excess moisture.

What about MultiClad HF prepregs?

The MCHF chemistry provides for apparent low water absorption, however, even low quantities of moisture can cause the same kind of issues present in other materials. As discussed above, moisture can react with the chemistry producing detrimental byproducts in addition to the effect on flow and thermal stability of the finished product.

MCHF has a very high content of organic fillers. These fillers along with the glass fabric do not absorb water so the water that may be absorbed is into the resin that is trying to bind all of the ingredients together. This can lead to additional sensitivity of the product to even low levels of moisture absorption.

Why does Arlon recommend vacuum desiccation of all prepregs prior to use?

Prepreg stored under refrigeration at < 5ºC (41ºF) must be allowed to stabilize at ambient temperatures prior to use. This time can be included as part of a vacuum desiccation process. Vacuum desiccation of Arlon’s prepreg is recommended to remove moisture. Moisture trapped in a multiplayer PCB can cause excess resin flow, measles, delamination and depressed Tg values. Store the dried prepreg in either a nitrogen dry box or chemical desiccation box. Control the temperature and humidity in these chambers per the conditions stated above.

Vacuum Times

Epoxy Prepregs:	44N, 45N, 45NK, 47N, 49N, 51N	= 6-8 Hours
Polyimide Prepregs:	33N, 35N, 37N, 38N, 84N, 85N	= 8-12 Hours
Thermount® Prepregs:	55NT, 55RT, 55ST, 65GT, 85NT, 85RT	= 24 Hours
25N & 25FR Prepregs:		= 4-6 Hours
MultiClad HF	MCHF	= 8-12 Hours

What special precautions must be taken with regard to moisture in high performance polyimide and some multifunctional epoxy prepregs?

Polyimide prepregs are more sensitive to moisture pickup from the air than conventional epoxy prepregs. The presence of moisture in prepreg, as is also the case in epoxies, can cause any of a number of process and product quality problems including foamy flow, voids, delamination and uneven thickness. In the case of polyimide, it also has an established link with poor bond to inner layer treated copper foil.

Grannells and Lamm (United Technologies) determined that as little as 0.2% moisture in a polyimide prepreg may result in some blistering and delamination, and as little as 0.4% can result in frothy flow and significantly deteriorated inner layer bonding. It is possible for polyimide prepreg to absorb this much or more water while sitting around in an uncontrolled humidity environment.

TROUBLESHOOTING TIP: Other volatile components of polyimide prepreg include low molecular weight resin fractions and some traces of retained high boiling solvents which may serve as process lubricant and actually react during prepreg cure, so measurement of total volatiles is not necessarily a good measure of retained water. Proactive removal of moisture by desiccation is important.

Although desiccant boxes have been used with some degree of success to remove water, the process is slow and unless you use dry desiccant and change it or “recharge” it regularly, it is often unreliable for critical jobs. Polyimide will eventually start to compete with desiccant for water if the desiccant is not dry and changed regularly. If you use a dry box, it is extremely helpful to use a slow bleed of dry nitrogen (use a bubble type flow meter to ensure that the flow is continuous) over the material. This will remove ambient moisture and by reducing the partial pressure of water over the material, aid in the diffusion of residual moisture out of the product.

The relationship between dry prepreg and quality of inner layer bond is a most important aspect of this phenomenon. The one method which has reliably removed water from prepreg down to the 0.2% level is vacuum desiccation. Roughly 12 to 16 hours (overnight) at 29 inches of mercury will remove essentially all the water that is not chemically bonded. “Hydrogen bonded” water at the surface of the fiberglass fibers, for example is not volatile under normal conditions although it can over time affect the degree of cure and the flow. Keeping prepreg in a dry environment prior to use will minimize the risk, but proactive drying is always recommended.

There are several ways to remove water effectively using vacuum:

1. Use a plasma unit during its “time off”. The plasma unit is capable of pulling a hard vacuum and unless it is in use 24 hours a day, it may be free overnight. Vacuum pumps are made for continuous duty and this will not hurt them.

2. If you are using vacuum assisted hydraulic presses or autoclave presses, prepreg can be dried in these units (without heat and pressure, of course!) during their off time at night.

3. Prepreg can be “turkey bagged” or placed into an improvised tool (made for example from silicone rubber sheeting designed for structural mold tooling) and held overnight in a vacuum.

4. A dedicated vacuum oven or vacuum chamber is always a good approach. Shelving will permit reasonable loading without stacking too high (which hinders water removal).

TROUBLESHOOTING TIP: The purpose of vacuum drying and vacuum lamination is the removal of volatile elements in the system to prevent their interfering with the process or product. While vacuum drying at room temperature or slightly above will largely remove only water, vacuum lamination may release low molecular weight resin or other trace materials at cure temperatures that over time can build up in the vacuum system and contaminate vacuum pump oil. Coalescing type filters collect this type of volatiles, but may become plugged up quickly, negating the effect of the vacuum. Cold trap filters, while they are less common and require continuing attention, do not plug up as readily. Valves and fittings in the system need to be cleaned periodically so they perform optimally. Be sure the vacuum pump has an oil return line and change vacuum pump oil on a regular preventive maintenance schedule to prevent damage to the pump.

Why is vacuum lamination superior to conventional press lamination for high performance multilayers? How does it differ?

Vacuum lamination is now the norm in most high end MLB facilities, although in some high volume FR-4 facilities, non-vacuum presses are still in use. For many reasons, vacuum lamination is highly desirable for MLB manufacture as it has become for the laminators themselves. Both enclosed vacuum presses and autoclave systems are in use. The biggest difference is that autoclave systems are slower in heat up rate and so consideration needs to be given to using prepregs with sufficient resin content to flow and fill at lower heat rate, hence higher melt viscosity/lower flow.

In a vacuum lamination system reduced lamination pressures are possible since the vacuum itself helps with resin flow around traces and into via holes. It may be possible to reduce pressures to about 60-70% of the pressure used in a non-vacuum press.

The reduced pressure also seems to help with registration, especially on complex PWB's.

Are there other sources of moisture that should be considered?

Laminate layers used in the multilayer board can be a significant source of moisture that can damage the final product. If the layers have not been sufficiently dried and kept dry up to the time of lamination, the moisture in the layers will try to escape during lamination. This occurs at the same time that the prepreg is melting and flowing. The moisture escape path is directly into the prepreg at its most critical phase. This will make problems appear to be a prepreg issue but the cause could be moisture in the layers and not prepreg conditioning.

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