Progress in the Reliability of Bonded Composite Structures

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Abstract

This paper reviews recent research progress in the detection of contamination on composites surfaces before bonding. Results to date indicate that it is possible to use a simple handheld instrument to determine if a composite surface is in such a state that a durable bond can be achieved. This study examined both airborne and contact contamination and found that contact contaminants can originate from unexpected sources. Monitoring of airborne contaminants in various manufacturing locations indicated that discrete contamination events can occur that are potentially detrimental to adhesion.

Introduction

To take full advantage of the many advantages that composite materials offer, subcomponents need to be assembled by adhesive bonding [1]. There is an inspection requirement to detect the absence of adhesion in bonded joints in order to meet the reliability and durability requirements of the aerospace industry. Detection of the lack of adhesion, even with intimate contact between the adhesive and composite, has been the "holy grail" of the nondestructive inspection (NDI) [2] research community for over 5 decades. In their search for a solution, NDI researchers attempted to use ultrasonic methods to estimate adhesive bond properties; an approach that was not successful [2]. Another approach was initiated in the 2000 time period showed that it is possible to measure the strength of a consolidated bond with a high power pulsed laser. When laser beams are focused on two opposing surfaces of the joint, they generate large compression waves that travel through the specimen and are reflected from the opposing free surfaces as tensile waves. These then travel back though the specimen overlapping at or near the center producing a large Mode I force [3-5]. A weak or "kissing bond[3]" results in a small internal delamination, which is detectable with traditional ultrasound inspection. This method, based on technology developed for laser shock peening (LSP), provides the designer with a quantitative evaluation of the mechanical performance of a bond, but only after it is cured [6]. This means that if a low strength bond is detected, the structure will require an expensive repair or will be scrapped at great expense. Since contamination of an adherend surface is recognized as the most a common cause of the weak or "kissing bond" a method to detect contaminated and non-compliant surfaces early in the fabrication is an important research topic. The detection of contamination in a manner compatible with bonded composite manufacturing processes is the subject of this paper.

Contamination of Bond Surfaces

It has been known for some time that a principal cause of low strength bonds is improper surface preparation and/or contamination surfaces prior adhesive curing. During the 1970's the U.S. Air Force conducted the "Primary Adhesively Bonded Structural Test" or PABST program to determine if adhesive bonding could be reliably used to join primary structural components in an aluminum airframe[4] [7]. During this effort, a great deal of attention was paid to all aspects of the

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[1] BTG Labs, Cincinnati, OH, USA
[2] Nondestructive inspection (NDI) and nondestructive evaluation (NDE) are used interchangeably in the literature and refer to inspection of a structures and materials to evaluate its properties or suitability for service.
[3] A kissing bond is a one in which there an intimate contact between adhesive and adherend but with little or no adhesion between them.
[4] The test article chosen for this program was the C-17 fuselage.

manufacturing process to ensure the long-term durability of the bonded joints. Additionally, research efforts examined previous bond failures to determine the cause of the failure. While some failures could be traced to physical flaws such as delaminations, a majority were associated with contamination or damage to the anodized aluminum surface. It was discovered that the surface can be compromised by contact with a hand, which crushes the fragile anodized oxide. As a result, careful attention was paid to handling of all anodized aluminum materials. Prevention of contamination was also a major concern and rigid process controls were established to prevent any possible contamination of a freshly anodized component. Several research programs were conducted to investigate methods of detecting contamination on anodized aluminum [8]. The conclusions reached at the end of these efforts are summarized as follows. Firstly, all surfaces are contaminated with some amount of non-native material. Extraneous substances/compounds are present in the environment and some may condense on the surface or are transferred to it by contact. Secondly, most contaminants are benign since they are absorbed into the adhesive during curing. Only a few contaminants were found to be detrimental to bond strength, such as the silicone greases used to lubricate the pumps that circulate the hot anodizing fluids. Finally, while several physical instruments could detect non-native films on the surface of the aluminum components, none could distinguish between benign and pernicious contaminants [9-17]. Additionally, these instruments took a great deal of time to detect contamination and were never adopted by the aircraft industry [8, 18-20]. In place of adopting instrumentation capable of detecting contamination, additional process controls were implemented to eliminate any possible contamination. These rigid controls were somewhat effective, but contaminated surfaces in subassemblies were still found later in the analysis of failed components. This situation has recently changed with the recognition that surface contamination can now be reliably and easily detected in the laboratory and on the production line with a simple liquid contact angle measuring instrument [21].

Surface Energy and Bond Strength

To prepare a surface for bonding there are a number of recommended steps, including cleaning with a suitable solvent, roughening with an abrasive to remove oxides and tenacious contaminants while increasing its surface area, and increasing surface energy with a treatment[5]. One of the simplest methods used to measure surface energy is to measure the contact angle of a standard liquid on the surface in question [22]. While measurement of this angle can be easily performed in a laboratory, until now there has not been a simple, reliable instrument available that could be used in a production environment [8, 23-25]. The analytical basis for the method of measuring the energy of a surface is based on the relationships between the surface energy vectors shown schematically in figure 1.
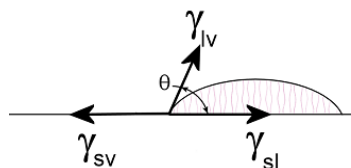

Figure 1   The Young diagram of surface energy relationships for a liquid drop on a solid substrate.

Accounting for all of the surface energy terms, the Young equation may be derived. This is the fundamental equation relating surface free energies to liquid wetting:

$$\gamma_{sv} = \gamma_s - \pi_s = \gamma_{lv} \cos\theta + \gamma_{sl} \tag{1}$$

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[5] It is very difficult to bond to a low energy surface.

where γ_s is the true surface energy of the substrate, π_s is the spreading pressure of the liquid reduction of solid surface energy due to interaction with the vapor of the wetting liquid spreading pressure of the liquid vapor on the surface (i.e. the spreading pressure), and θ is the contact angle formed between the liquid and the substrate. In the case of metal surfaces, π_s can be large. However, for low energy surfaces such as composite materials, π_s is generally considered small enough to be disregarded, which allows setting γ_{sv} equal to γ_s. Evaluation of this equation involves two quantities that are directly measurable (γ_{lv} and θ), and two quantities that are not directly measurable (γ_{sv} and γ_{sl}). Thus, to determine surface energy in general requires contact angle measurements with at least two (and preferably more) distinct liquids [26].

From equation 1, one can see that contact angle is directly related to surface energy. For practical quantification of surface energy in a manufacturing process, contact angles of a single liquid can be correlated to total surface energy in a very effective manner [27], greatly simplifying the use of these techniques for quantitative quality assurance of surface condition prior to bonding. A surface with a higher surface energy will have a stronger bond with adhesives [28, 29]. Previous work has shown excellent correlation between surface energy and peel strength of pressure sensitive adhesives (PSA's) [30-34]; another study has shown that the peel strength of a PSA to a composite surface is a sensitive indicator of surface energy of a composite surface [22]. In the same study, a common mold release contaminant, Frekote 44, was applied to the surface of the composite. This contamination lowered the surface energy of the composite and reduced the bond strength as indicated by the force needed to peel a PSA tape from the composite. Thus contamination may be detected and quantified with a simple contact angle test. In Figure 2 the diameter of a small drop of DMSO[6] is plotted versus the peel strength of a PSA adhesive on a carbon/epoxy composite. The diameter of the liquid drop is easily measured optically, which along with knowledge of the drop volume may be easily converted into the liquid contact angle on the composite surface. From this figure it is apparent the droplet diameters less than 4 mm are a good indicator of a low strength bond.
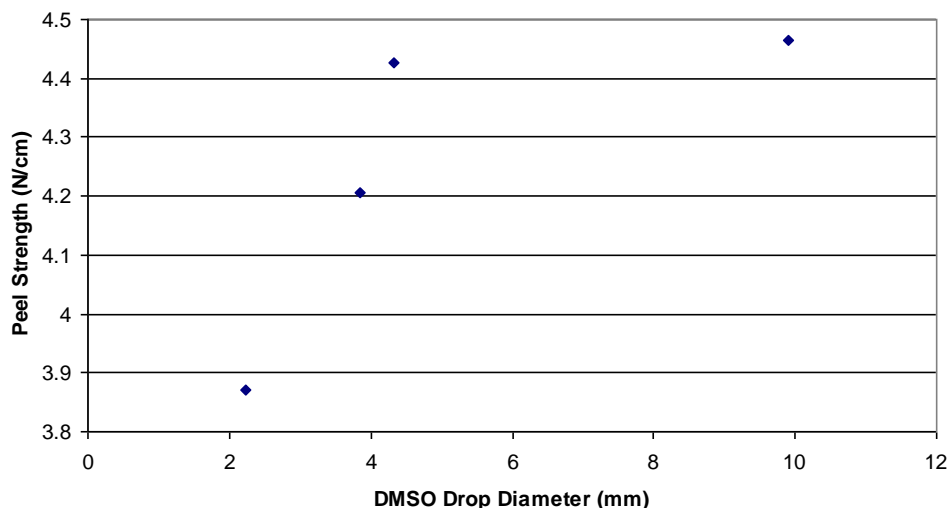

Figure 2 Comparison of peel strength as measured during tape peel test and the drop diameter of 3µl DMSO drops.

These techniques have been used to readily detect monomolecular layers of contaminants on composite surfaces with sensitivity essentially equivalent to X-ray Photoelectron Spectroscopy (XPS) [35].

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[6] DMSO is dimethyl sulfoxide, a liquid commonly used as surface energy standard.

With a simple method of detecting contamination available, the obvious question is "what constitutes a detrimental contaminant?" During the PABST program [36] it was discovered that many well-known contaminants did not reduce bond strength and are benign. For example, contact with a hand would transfer sebum or skin oil to the anodized surface. However, this did not degrade the bond strength because sebum is soluble in an epoxy adhesive during cure. The mechanism of bond strength reduction in this situation was the mechanical damage to the fragile anodized surface caused by contact with a human hand or other objects. Some potential contaminants were very effective at reducing bond strength. For example, even small amounts of silicone grease leaking from anodizing bath pump seals could ruin the adhesion properties of many parts for several days. The contaminated solution had to be discarded, the anodizing tank thoroughly cleaned, and the anodizing solution replace before primer would adhere to anodized aluminum panels. Numerous other potential contaminants were examined, but none were found to be as deleterious as silicones. These data were obtained for aluminum, and may not apply to composites. Therefore, a similar series of tests of potential contaminants have been conducted for composites in one manufacturing facility. These tests are a part of the current DARPA Open Manufacturing program (TRUST[7]) [37].

Classification of Contaminates

Potential contaminants can be divided into two groups: intrinsic and extrinsic compounds. Intrinsic contaminants are those compounds that originate within the material and migrate to the surface over time. An example would be internal mold released in injection molded thermoplastics. Such contaminants would be expected to play a role in adhesive bonding of aerospace composites. Extrinsic contaminants are those that originate external to the material and are transported to the surface via several paths. These include airborne substances and a range of materials that may be transferred by contact. Airborne contaminates are vaporizable substances such as low molecular weight compounds (e.g. light hydrocarbons) and water. Contact contaminants originate in materials used in and around the manufacturing process; contamination events can occur by contact with materials or tools.

The traditional approach to evaluating the effects of possible contaminants has been to evaluate all possible compounds found in a facility for their impact on bond strength. Contaminants are frequently complex formulated multicomponent substances; evaluating the effect of all possible contaminants would require a large testing program involving many potential contaminant compounds currently in use or anticipated for future use – a daunting task. Furthermore, such an investigation provides little information as to *why* a particular contaminant is benign or detrimental. A much simpler and more effective approach being pursued in the TRUST effort is to examine the effects of carefully chosen constituents of the commonly available materials found in many composite manufacturing facility and those likely associated with workers, and using these compounds as representative archetypes of more complex contaminating substances. An abbreviated set of such compounds is shown in table 1. These compounds represent constituents of substances commonly investigated as contaminants. This approach indicates that any complex contaminating substance can be represented as a mixture of compounds belonging to specific classes of chemical compounds. For the purposes of this investigation 8 distinct chemical classes were chosen. Making the assumption that the effects of a complex contaminant are a linear combination of the effects of the constituent compounds, knowledge of the effects of the individual constituents permits prediction of the effects of an arbitrary mixture of these constituents. To determine this, a single archetype compound was selected to represent each class of constituent (table 2).

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[7] Transition Reliable Unitized STructure (TRUST)

Table 1   Common contaminants broken down into their chemical classes [38]

chemical class / Archetype Compounds	triglyceride	fatty acid	alcohol			surfactant/ emulsifier				long chain ester	non-polar hydrocarbon			water soluble polymer	inorganics	
	glycerol trioleate	octanoic acid	lauryl alcohol	cholesterol	glycerol	lecithin	soap	polymeric/Cationic/anionic	triethanolamine	glycerol monostearate	squalene	mineral oil	petrolatum	poly(vinyl pyrrolidone)	Dimethyl Siloxanes	molybdenum disulfide
biological																
sebum	✓	✓								✓	✓		✓			
earwax		✓	✓	✓							✓					
food products																
mayonnaise	✓			✓		✓										
vegetable oils	✓										✓					
chocolate	✓															
personal care																
hair gel/cosmetics					✓				✓					✓	✓	
lotion (generic)		✓	✓		✓				✓	✓		✓	✓		✓	
R&R I.C. blue lotion		✓			✓					✓						
industrial chemicals																
lubricating grease							✓					✓				✓
lubricant oil												✓				
water based cutting fluids	✓							✓	✓			✓				
oil based cutting fluids												✓				
mold releases													✓		✓	
cleansers		✓						✓	✓							
standard soils																
SAE AMS 3167B	✓	✓	✓	✓		✓			✓			✓	✓			
Cessna							✓					✓				

Table 2.   Archetype compounds selected as standard contaminants [38]

chemical class:	triglyceride	fatty acid	long chain esters	alcohol	surfactant/ emulsifier	non-polar hydrocarbon	water soluble polymer	inorganics
archetype compound:	glyceryl trioleate	octanoic acid	glycerol monostearate	glycerol	sodium tallowate	mineral oil	polyvinyl pyrrolidone	dimethyl siloxane

Based on this approach, the current phase of the TRUST test program has evaluated the effects of the archetype contaminant compounds in table 2 on adhesive bond performance. Their effect on composite bond performance has been evaluated for various concentration levels on a standard composite surface, along with the quantitative detectability of these compounds using water contact angle measurements. A database of the wetting behavior of both pristine composite surfaces as well as those contaminated to carefully controlled levels is being compiled using a

hand held contact angle measuring instrument [21]. A future phase of this program will evaluate combinations of archetype contaminant compounds to confirm the validity of an approach based on linear combinations of effects. Not only does this approach greatly reduce the number of contaminants to be evaluated, but it provides a framework for evaluation of products that may be added to the manufacturing environment at a later date. The database will be a part of the software in a handheld instrument that will permit manufacturing personnel to examine a questionable article for degrading contamination prior to a bonding process.

Airborne Contamination

Once a contaminant compound is identified as detrimental to a bonding process, it becomes a controlled substance in the manufacturing environment; its presence must be detectable. A novel approach developed in the TRUST program to quantitatively detect and identify airborne contaminants involves exposing aluminum foil witness coupons to the various manufacturing environments. These are retrieved at two week intervals for examination using contact angle measurements to evaluate changes in surface energy that are indicative of exposure to a contaminant. Coupons that show changes are then evaluated using XPS and/or and reflection absorption infrared spectroscopy (RAIR).

As an example, Figure 3 shows surface atomic composition data obtained from foil samples exposed in one manufacturing location. The increase in relative % carbon indicates the presence of airborne aliphatic hydrocarbon contaminants in the environment that have adsorbed to the foil surface. In general these are benign contaminants that don't adversely affect adhesive bond performance. Of more significance was the appearance of ~5 atomic % silicon at week 40. This singular event was traced to the inadvertent introduction of volatile silicone compounds to the environment. It represents surface contamination by a potentially detrimental contaminant. Detection of these events allows bond process quality to be much more tightly controlled.
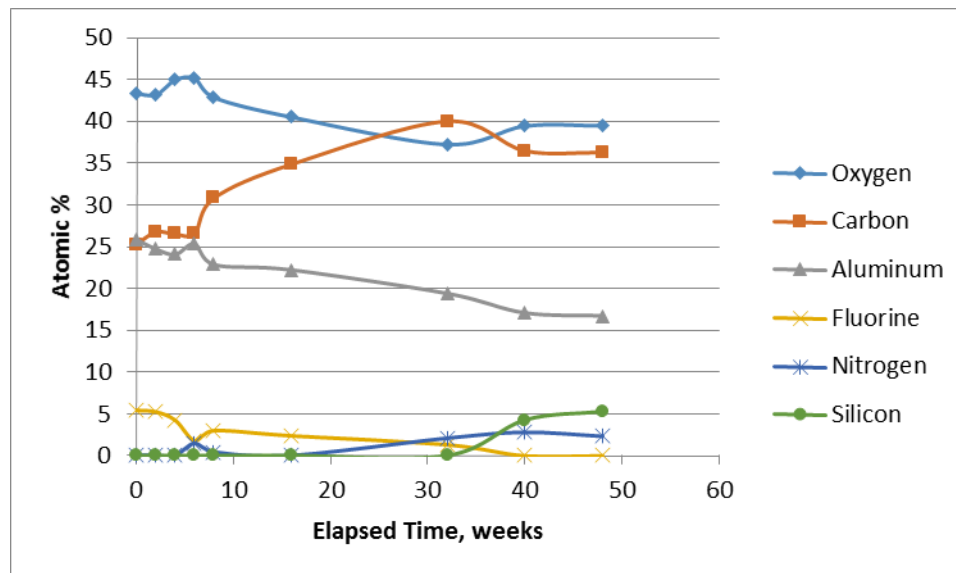

Figure 3   Atomic composition of aluminum foil witness surfaces after exposure to an aerospace manufacturing environment. The increase in relative % carbon indicates the presence of airborne aliphatic hydrocarbon contaminants. The appearance of silicon at week 40 was due to the inadvertent introduction of volatile silicone compounds to the environment.

Contact Contamination

A separate study examined possible contamination by direct contact with a standard surface by materials commonly found in the composite fabrication facility. In this effort, the possibility of

direct transfer of a contaminant by contact between the common shop materials and the diamond surface of an Attenuated Total Reflectance Infrared Spectroscopy (ATR-IR) instrument was investigated as indicative of the potential for transfer of contaminants to a bonding surface. Transfer was shown to occur quickly with minimal contact pressure. As an example, figure 4 shows representative ATR-IR spectra of a polyethylene bag used for storing sensitive surfaces along with the spectrum of the diamond ATR-IR reflection element obtained after the sample bag was removed. The transfer in this case consisted almost exclusively of non-polar hydrocarbons, in general a non-detrimental contaminant. Table 3 shows results for many such shop supplies and indicates that direct transfer of potentially detrimental extrinsic contaminant compounds found in a fabrication facility appears to be easy and fast.
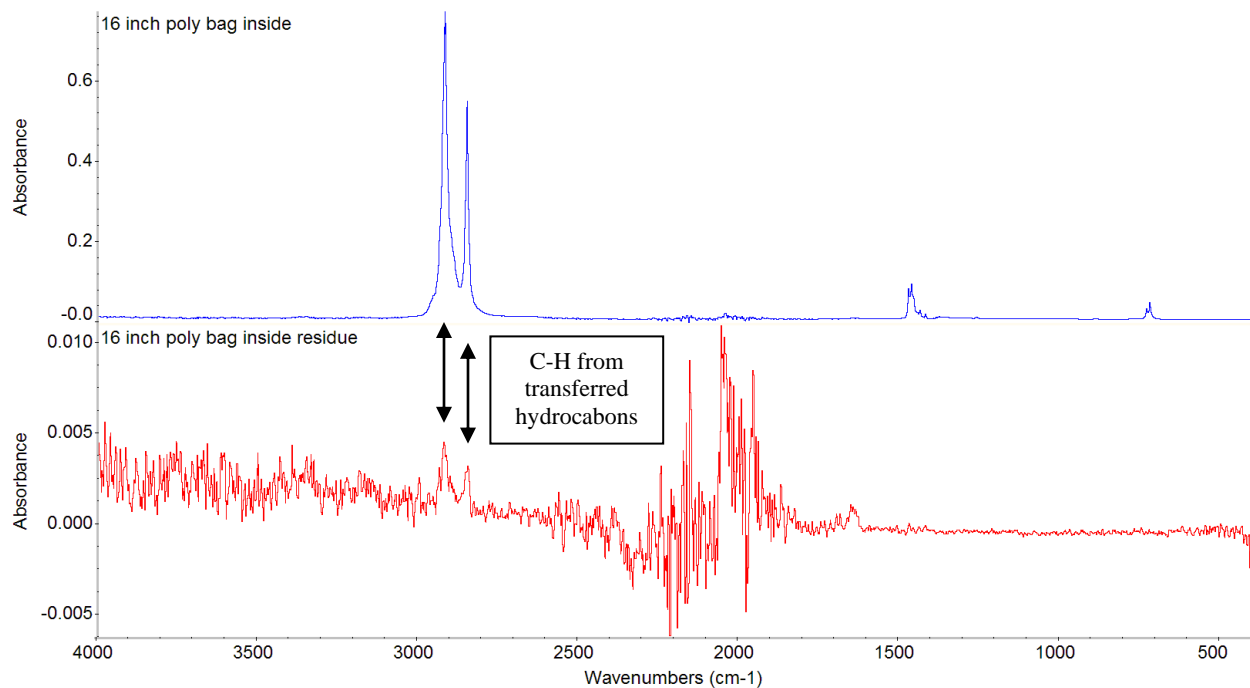

Figure 4 ATR-IR spectra obtained from a polyethylene storage bag (top), along with the spectrum obtained immediately after removing the bag from contact with the diamond ATR-IR reflection element, showing that simple contact with the poly bag can result in transfer of contaminant.

Table 3   Evaluation of materials a composite manufacturing environment

materials	standard contaminant chemical class								
	triglyceride	fatty acid	long chain ester	alcohol	surfactant/ emulsifier	non-polar hydrocarbon	water soluble polymer	silicone/ fluorocarbon	transfer?
shop supplies									
Flashbreaker tape						✓		✓	✓
razor blades						✓			✓
silicone edge dams						✓		✓	
n-10 breather						✓			
bagging material						✓			
violet FEP								✓	
red FEP								✓	
breather on edge dam									
tacky tape						✓		✓	
paper backing						✓		✓	✓
16" poly bag						✓			✓
24" poly bag						✓			✓
personal protective equipment									
blue nitrile gloves						✓			
white gloves, new						✓			
white gloves, used						✓		✓	✓
Tyvek® suit, new						✓			
hairnet, new						✓			
Tyvek® booties, new						✓			
Tyvek® booties, used						✓			

Cost of Detection

The question that almost all inspection personnel ask at this point is how many spatially separate locations must be tested to qualify as a 100% inspection of a part surface. While this is a question for the future examination, if the past is any guide the answer is a satisfying one. Data from failure analyses of bonded joints suggests that the smallest contaminated area on a failed component is on the order of 650 cm^2 [39]. This is a rather large area compared to the usual scan density that is required for a 100% inspection of a component for potential physical flaws such a cracks or delaminations. Thus, the sampling requirements for contamination detection and identification are not expected to be costly or extensive.

Conclusions

Reliable and durable adhesive bonding is necessary for composite materials to achieve their full potential in aerospace structures. A limiting factor to achieving reliable bonds has been the detection of pernicious contamination. This work has shown that a simple surface energy measurement can be used for this purpose. Data obtained in this study also show that composite surfaces can be contaminated by aerosols as well as by low pressure contact with materials commonly found in composite fabrication facilities. Because the number of potential contaminants is quite large, potential contaminates were examined and potential contaminants compounds identified as belonging in one or more eight chemical classes. Within each class an archetype compound was identified for detailed examination of its contamination potential. By assuming that the contamination potential of a suspect material can be modeled as a linear combination of the effects each compound, the testing complexity for suspect materials can be reduced to a manageable number. Experiments are underway to determine how each archetype compound, at various concentration levels, effects adhesion to a standard composite surface. Initial results confirm predictions based upon chemical incompatibility. Further detailed experiments will provide a more detailed prescription for the control of materials that may contact with a composite surface. These data are significant to both composite manufacturers and scientific community. Few researchers appreciate how easily pernicious contaminants can be transferred to the specimen during specimen preparation. One positive result of the testing to date indicates that most airborne and contact contamination does not seem to be important to bond performance. While the initial results are quite encouraging, the results of long term durability test will not be available for some time. Another positive result, based upon failure analysis of many specimens, is that sampling for contamination need only be performed at only a few widely spaced locations.

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