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MANAGING MICRO-VOIDING OF ADHESIVE BONDS

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1. Introduction

This article has been written in response to several recent failures of adhesive bonded aircraft structures where there was evidence of micro-voiding which could be seen on the failure surfaces. While the article focuses on adhesive bonds, similar micro-voids also occur in laminated composite materials. The methods suggested for management of micro-voiding are equally applicable to both composite and adhesive bond production, and are also relevant for bonded and composite repair installation.

2. What Is Micro-Voiding?

Micro-voiding in adhesive bonds is characterised by the formation of a significant number of small voids in adhesive bonds cured at elevated temperature. These micro-voids are typically less than 0.5 mm diameter. Whilst each individual void is not of a sufficient size to be significant to structural integrity of the bond, the total sum of the area of micro-voids in the bond may be sufficient to cause a substantial loss of bond strength, see Figure 1.

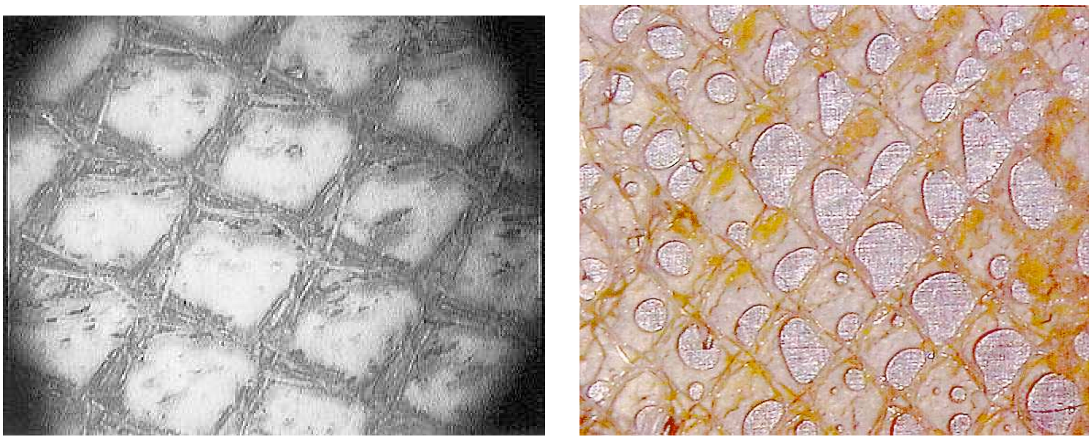

Figure 1: Adhesive bond fracture surfaces for an overlap joint. The figure on the left exhibits minimal voiding and the figure on the right shows the formation of micro-voids. The size may be gauged by comparing the void size against the woven carrier cloth embedded in the adhesive, which is 0.5mm pitch.

In sandwich structure, micro-voids may occur in the fillet bond (the region where capillary action causes the adhesive to wet the sides of the core, forming fillets), see Figure 2. This has the potential to significantly reduce the shear, peel and flat-wise tensile strength of the bond.

3. Causes of Micro-Voiding

Micro-voids in adhesive bonds are a direct result of the evolution of gasses during heat curing of adhesives. These gasses may result from vaporisation of chemicals such as solvents used in adhesive film production. However, the main source of micro-voids is the release of water as steam. Many adhesives (such as epoxies) are polar molecules which absorb atmospheric moisture whilst being exposed during the bonding process and that moisture is evolved as steam once the temperature exceeds the boiling point. In some cases (such as phenolics) the chemical reactions create water as a by-product in the process known as condensation polymerisation. Again, the moisture evolved turns to steam and creates micro-voids.

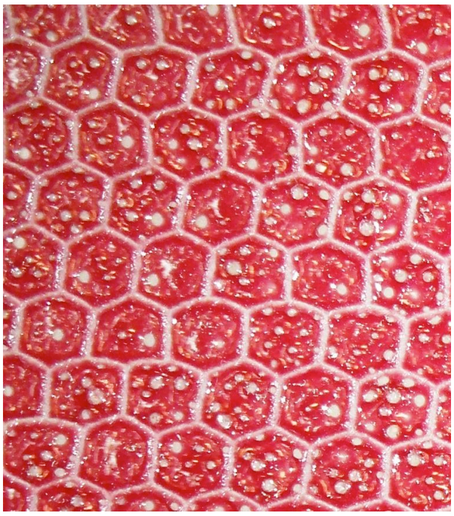

Figure 2: Micro-voids in sandwich structure. Note the occurrence of bubbles in the fillet areas of the bond, as well as in the centre of the cells.

4. Significance of Micro-Voiding

The effect of micro-voiding is to substantially reduce the area of adhesive available to transfer loads. Tests [1] of a typical structural adhesive showed that exposure to 30°C and 70% RH produced a 53% reduction in T-peel strength and 28% reduction in aluminium honeycomb peel strength compared to samples of the same adhesive batch exposed to 20°C and 50% RH when the samples were cured under vacuum. Such reductions in strength may have implications with regard to structural integrity.

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[1] Arnott, D.R., Wilson, A.R., Pearce, P.J., Mathys, G., Kindermann, M.R., Camilleri, A., DAVIS, M.J., Swan, G., *Void Development in Aerospace Film Adhesives During Vacuum Bag Cure*, Int. Aerospace Congress, Sydney, 25-28 Feb. 1997.

5. Detection of Micro-Voiding

Because there is still a significant level of contact between the adherends and the adhesive, NDI such as ultrasonic or tap-hammer testing will not detect the presence of micro-voids. Hence, structures with degraded strength will pass most common Quality Assurance tests.

The only reliable method for detecting micro-voiding is to visually examine the adhesive flash (the excess adhesive squeezed out of the joint as the bond forms). The presence of a significant number of micro-voids trapped in the adhesive flash is a strong indication that similar micro-voids also exist in the bond itself, see Figure 3.

6. Managing Micro-Voiding

6.1. Production pressurisation measures

There is a common perception that the application of vacuum during adhesive cure draws trapped volatiles out of the bond. This does occur within a small distance of the edge of a bond, but in general any volatiles trapped within the bond form micro-voids and the application of excessive vacuum actually *increases* the size of the voids by lowering the local vapour pressure. Hence, a strategy of trying to remove the volatiles during processing can not rely on vacuum alone. Reducing the applied vacuum to 250 mmHg at the time the adhesive flows does help to reduce the void content of the bond. Application of positive pressure (autoclave processing) will act to reduce the size of micro-voids by reducing their volume. For condensation polymerising materials such as phenolics, autoclave processing is essential.

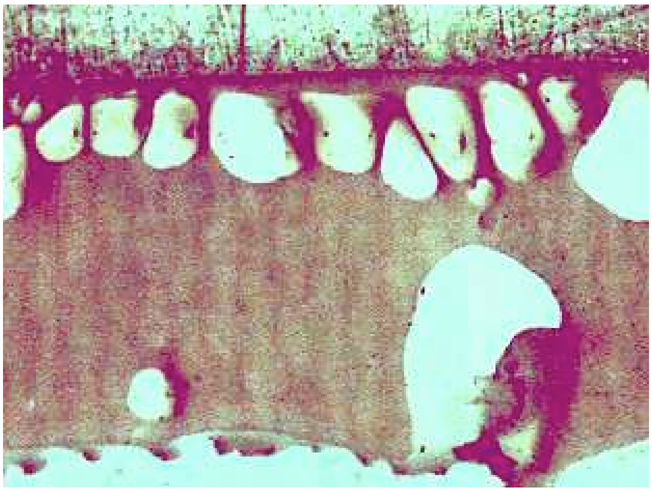

Figure 3: Excessive micro-voiding occurring adjacent to the end of one adherend, indicating the presence of micro-voids within the bond itself.

6.2. Production heating

Another factor which influences micro-voiding is the rate at which the temperature of the bond is increased during the cure cycle. Adhesive bonds which are cured at temperature must be heated in a controlled manner as specified by the adhesive manufacturer, typically at a rate of 3°C to 6°C per minute. If the heat up rate is too slow, the adhesive may gel before it has had a chance to flow. This will result in poor wetting of the surface and low bond strength. If the adhesive is heated too fast, then the volatiles are released rapidly and the adhesive gels before any volatiles can escape to vacuum. This situation may result from the use of radiant heating methods, or in cases where convection heating creates hotter regions within the oven or autoclave.

6.3. Minimising exposure to humidity

A far better strategy for minimising micro-voiding is to take measures to reduce the volatile content of the adhesive film prior to cure. Most adhesive manufacturers control the volatile content due to adhesive production solvents, so that source of voiding is usually not significant. By far the largest proportion of volatile products is water from atmospheric humidity which is absorbed by the adhesive during exposure for processing. Measures are therefore required to minimise that exposure.

6.3.1. Receipt of product

Film adhesives are usually transported in sealed plastic bags which are packed in dry ice to minimise progression of the adhesive cure during transport. Moisture contamination of the adhesive often results from penetration damage to the plastic bag. Atmospheric humidity condenses inside the bag and is absorbed by the adhesive as the product thaws prior to use. All products should be inspected for damage to the packaging and any material supplied with penetration of the plastic packaging should be rejected.

In some cases the material is supplied in plastic bags which have only been sealed using adhesive tapes. This is bad practice because the adhesive tapes often loose tack at low temperature and the seal is compromised. Only accept materials which have been packaged in heat-sealed plastic materials.

Another source of moisture contamination is the use of material which has an unknown history. Supplies of adhesives are often exchanged between production plants and even between different user organisations. The risks of contamination must be assessed based on the standards of management of humidity at the source facility.

To maximise the life of the product, the material should be placed in freezer storage as soon as practical after receipt. Measures are required during storage to prevent damage occurring to the plastic packaging.

6.3.2. Use of product

Prior to use, the packaging should be examined for signs of penetration and any damage packaging should be cause for rejection of the contents. Adhesives are usually stored in freezers at a temperature of -15°C or lower. The package must be allowed to stand unopened until the entire contents reach room temperature to prevent condensation forming on the adhesive. Only when condensation stops forming on the surface of the packaging may the package be opened. *Do not apply heat to accelerate the thawing process as damage to the adhesive may occur.*

Once the package is opened the adhesive will be exposed to atmospheric moisture due to humidity. To successfully restrict the moisture uptake, adhesives should only be exposed in an environment where both temperature and humidity are controlled. Temperature is controlled between 18°C and 23°C to manage tack of the adhesive; too cold and the adhesive will not tack to the bonding surface and too hot and the adhesive will tack to everything it contacts including protective gloves. To minimise moisture content, the upper limits on humidity are 65% RH at 18°C to 45% RH at 23°C. Lower humidity than these levels is highly desirable.

Another source of moisture is the surface of the adherends being bonded. All details must be thoroughly dried and stored in the same controlled environment as detailed in the last paragraph. This is especially true for honeycomb core materials. Nomex core requires a special mention. Nomex core also absorbs significant amounts of moisture. If that moisture is not removed by thoroughly drying the core before fabrication, then that moisture is liberated during the bonding process and can lead to micro-voiding of the bond to a level which is far more significant than for aluminium core. One effective measure to manage moisture on core and part details is to store these in a dedicated room which is maintained at 50°C and <10% RH.

7. Use of this data

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