

Outgassing: An Atypical Approach

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ABSTRACT

Outgassing of adhesives and coatings can cause problems in optics used in space applications. The actual test NASA runs on candidate materials is very expensive and time consuming. End users will typically test every batch of material they receive and reject any that do not pass the requirements.

This paper discusses a test which acts as an indicator to measure the relative performance and determine if the manufactured material will pass the outgassing requirement without going through the actual ASTM test. This is not a substitute for the final test. Its purpose is for screening materials being used in manufacturing or research.

ATYPICAL MEASUREMENTS OF OUTGASSING

This article describes a generic means of identifying the outgassing of an optical adhesive. To identify the residual outgassing of a material, in the past, one had to either test the material in-house or send it out. This paper will not reduce the necessity to ASTM test the initial material however it will save or eliminate the need to test each lot of material. It allows for quality checks using standard laboratory items, after the initial ASTM test has been performed.

Outgassing occurs in all adhesives to a varying degree. From water based adhesives to solvent based adhesives, outgassing or flashing off of materials occurs. More than many others, the optical industry requires adhesives that outgass less than standard adhesives. In optical applications materials can volatilize and deposit on optical surfaces causing changes in optical properties. This is particularly important in space applications due to the tremendous vacuum encountered. Numerous industries including aerospace, lens and coating and computer industries, are concerned with the outgassing of materials.

The adhesives tested, were selected from several categories. Some polymers are more dense than others, some polymers use photo-initiators, and others are activated by different methods (amines, amides, peroxides, etcetera). It should be noted that a variety of components flashed off the polymers.

ASTM Test Method E 595-90 for Outgassing

The ASTM test method is a technique to standardize and uniformly categorize all adhesives to an individual test. The test method describes the test apparatus to be used, the pressure to be used and the temperature and time necessary for the test.

The pressure used in the ASTM test is 1×10^{-5} torr. Samples of 2-5 grams are run for 24 hours at a temperature of 125°C.

This test measures the Total Mass Loss (TML), the Collectible Volatile Condensable Materials (CVCM), and the Water Vapor Regain (WVR).

The Atypical approach

To approximate the results of the ASTM test we used the following approach. The adhesive is totally polymerized, in triplicate, and then allowed to sit at ambient temperature in a desiccator overnight. The samples are then weighed using an analytical balance (0.0001 grams), and placed in an air-circulating oven at 125°C for 24 hours. Next the specimens are removed from the oven and placed in the desiccator overnight. The samples are then reweighed.

This test is based on the percent TML of the adhesive. The ASTM test represents the standard, and our test represents the sample. The difference in TML between the sample and the reference is then calculated. This should be consistent for all other lots of that adhesive purchased.

This test in all cases will produce data that is consistent and exceedingly reliable. It can be used to screen incoming materials as well as giving the quality control staff confidence in the outgassing characteristic of the incoming materials.

Acrylate/Methacrylate Optical Adhesive

Total Mass Loss Percent

Test Value 1.1378%

ASTM Test 3.6800%¹

% of ASTM--30.9197

As can be seen from the above data the actual percentage of cast off material with the acrylate/methacrylate system is 30% of the ASTM tested amount. This could be due to the molecular weight of the cast material. These compounds consist of primary, secondary or tertiary amines. The materials that flash off the polymer are either carboxylic acids, of varying molecular weights and molecular configurations, or other materials that do not become part of the matrix. This variety leads to the conclusion that it would be difficult to categorize a single standard for all

adhesives, but individual adhesives can be categorized using this method.

Epoxides Optical Adhesives

Total Mass Loss
Test Value 2.9548%
ASTM Test 3.4000%¹

% of ASTM--86.9067

If one examines the resulting numerics (percent TML) of optical epoxy adhesives after they have totally cured, a noticeable difference occurs. After this epoxy material has been exposed to the 125°C temperature for 24 hours, nearly all the material that flashes off when subjected to the ASTM test procedure flashes off using the atypical method.

In the testing the epoxy adhesives registered a TML of 87% of the ASTM value verses 31% for the previous type of material. For most epoxides this evaluative method can be used because most epoxy monomers used are di-functional and the polymer mesh isn't as tightly bound as a more functional polymeric structures.

It is recommended that the test be performed in triplicate on adhesives of this type prior to accepting the results as fact. It should also be noted that the ASTM test should be run to set the standard prior to accepting the above results.

Polyester Adhesives

Total Mass Loss
Test Value 0.5540%
ASTM Test 2.3500%¹

% of ASTM 23.5746

Total Mass Loss
Test Value 0.6463%
ASTM Test 3.6500%¹

% of ASTM 17.7069

When working with polyester materials it is common to have lower numbers for the laboratory outgassing (TML). Due to the functionality of the monomers that make up the adhesive and the resulting high cross-linking density. The polymer in many cases does not allow the release of the materials. The vacuum is necessary to get an accurate determination of the total outgassing.

Even though the resultant value is low it is an accurate gauge of the TML for that particular material.

Polyurethane

Total Mass Loss

Test Value 0.7113%

ASTM Test 0.8950%¹

% of ASTM 79.4710

Total Mass Loss

Test Value 1.1374%

ASTM Test 2.2400%¹

% of ASTM 50.7754

Total Mass Loss

Test Value 1.2434%

ASTM Test 1.8100%¹

% of ASTM 68.6976

The formulation for Polyurethane adhesives lends itself to a more specific and consistent analysis. Since these adhesives are very efficient kinetically the outgassing test performed are very reproducible.

In the test there were two types of material tested. There were samples with fillers and neat samples. The neat samples using the standard air circulating oven at 125°C yielded a 70-80% of overall TML.

The purpose for presenting the information here was to furnish a screening technique for outgassing of optical adhesives for laboratory personnel. The method used is not ASTM but if one knows enough about the product-Material Safety Data Sheet (MSDS), Technical Data Sheet (TDS)-the examiner will have a running start on categorizing the potential outgassing of optical adhesives. It must also be noted that the formal ASTM test should be performed prior to categorizing the adhesive products.

Now that you comprehend outgassing and its effects you can utilize the data provided to obtain the best technique and pattern for your product. Generally it must be realized that performance and marketability are the driving factors in the product chosen for an application. The determining factor is product performance and reliability.

¹-Values from NASA Reference Publication 1124 Revision @ authors William A Campbell, Jr. and John Scialdone.