RHEOLOGICAL TESTING FOR DETERMINING THE REACTIVITY PROFILE OF HEM FLANGE ADHESIVES

Application

This method is used to evaluate the shear modulus of adhesives and sealers as a function of time and extent of cure. Such properties help to optimize the processing of these materials throughout the manufacturing process.

Apparatus Required

Rheometer

A controlled-stress or controlled-strain rheometer, equipped with 20 mm parallel plate fixtures, induction heating coil, and liquid nitrogen cooling. The rheometer should be capable of achieving the induction heating profiles shown in Figures 5 and 6, and it should also meet the following specifications:

Torque Range = 0.1 x 10^{-6} Nm to 100 mNm
Frequency Range = 0.1 mHz to 100 Hz
Angular Velocity Range = 10^{-2} to 100 rad/s
Angular Displacement Resolution = 0.62 x 10^{-6} rads

For example, TA Instruments AR1000 Rheometer equipped with 20 mm disposable parallel plate fixtures. (Figure 1)

Materials Required

Adhesive

Hem flange adhesives (Ford specifications WSS-M2G373-B/C and WSS-M2G300-A2) will be tested according to this test method.

Conditioning and Test Conditions

All test values indicated herein are based on material conditioned in a controlled atmosphere of 23 +/- 2 °C and 50 +/- 5% relative humidity for not less than 24 h prior to testing. Materials will be tested at 23 °C.

Procedure

1. Prior to testing, calibrate the instrument following the procedure provided by the manufacturer. Also calibrate the parallel plates for inertia offset and gap separation.

2. Load material into rheometer and set the gap at 0.75 mm. Trim excess material from parallel plates. [Note that for two-component adhesives, the material must be loaded into the rheometer as quickly as possible. These adhesives should be mixed by the most reproducible method, either manually or by using a mixing nozzle].

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3. Test the material under the following conditions:
   a. Oscillatory Mode
   b. Single Frequency = 1 Hz

4. Three different tests should be run under the conditions of Step 3. For one-component adhesives, only Tests Two and Three (parts b and c) should be run. For two-component adhesives, all three tests should be run. These three tests are shown schematically in Figures 2 through 4, respectively.

   a. Test One: Room Temperature Testing (5 Hours)
      1. Time Sweep = 5 Hours at 23 °C
         a. Sample Interval = 10 Minutes (30 Data Points)
         b. Controlled Variable = % Strain = 0.5%
      2. E-Coat Oven Simulation (See Step 5)

   b. Test Two: Induction Cure Testing (2 Hours) – Low Induction Cure Profile
      1. Low Induction Cure Simulation (See Step 6 and Figure 5)
      2. Time Sweep = 2 Hours at 23 °C
         a. Sample Interval = 5 Minutes (24 Data Points)
         b. Controlled Variable = % Strain = 0.1%
      3. E-Coat Oven Simulation (See Step 5)

   c. Test Three: Induction Cure Testing (2 Hours) – High Induction Cure Profile
      1. High Induction Cure Simulation (See Step 6 and Figure 6)
      2. Time Sweep = 2 Hours at 23 °C
         a. Sample Interval = 5 Minutes (24 Data Points)
         b. Controlled Variable = % Strain = 0.1%
      3. E-Coat Oven Simulation (See Step 5)

5. E-Coat Oven Simulation Procedure
   a. Temperature Ramp Up = 23 °C to 170 °C
      1. Rate = 20 °C per Minute
      2. 10 Data Points
      3. Controlled Variable = % Strain = 0.5% (Test 1)
         = 0.1% (Tests 2 and 3)

   b. Time Sweep = 20 Minutes at 170 °C (Hold)
      1. 10 Data Points
      2. Controlled Variable = % Strain = 0.5% (Test 1)
         = 0.1% (Tests 2 and 3)

   c. Temperature Ramp Down = 170 °C to 23 °C
      1. Rate = 20 °C per Minute
      2. 10 Data Points
      3. Controlled Variable = % Strain = 0.01%

6. Induction Cure Simulation Procedure: The induction cure profiles shown in Figures 5 and 6 should be programmed into the rheometer. The tolerance on these induction cure profiles is +/- 3%. The cure profile in Figure 5, labeled 'Low Induction Cure Profile', shows a maximum temperature of 115 °C (240 °F) achieved in approximately 70 seconds. The profile in Figure 6, labeled 'High Induction Cure Profile', shows a maximum temperature of 135 °C (275 °F) achieved in approximately 80 seconds.

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Each rheometer is different and thus will require a slightly different program. The induction cure profiles required are only possible with induction coil heating available on certain rheometers. Appendix I illustrates an example of a typical program that may be used to obtain the profile shown in Figure 6. The majority of data will be collected after Induction Cure takes place; it is not vital to collect data during this step. Note that these induction cure profiles are not intended to simulate actual production induction cure profiles, due to equipment limitations. The intent is to use the results for comparison purposes only.

Some rheometers are not equipped with induction heating coils. In these cases, the material (loaded between two parallel plates) may be induction cured in a remote location, according to the profiles shown in Figures 5 and 6, and then loaded into the rheometer. Data should be collected for at least the last 30 minutes of the 2-Hour Time Sweep. (It is not absolutely necessary to collect data during the first 1.5 hours).

7. Plot $\log_{10}$ complex shear modulus (G*) versus time (hr) for the three tests performed in step 4.
The complex shear modulus (G*) is a sum of real (G') and imaginary (iG") parts, as shown in the equation below (1, 2):

$$G^* = G' + iG''$$

Tests 1, 2 and 3 should each be plotted on a separate graph. Two examples are shown in Figures 7 and 8.

NOTE: In some instances, a 'Normal Force Overload' may occur on the rheometer during the E-Coat Oven Simulation Procedure. This is due to the thermal expansion and contraction of both the parallel plates and the adhesive during the heating and cooling cycles. If this occurs, the value of % Strain may be reduced to possibly alleviate the problem.

NOTE: It is recommended that disposable parallel plate fixtures be used since the material will be curing during the testing. These plates can be reused by burning off the material in a furnace at 400 °C for a duration of 24 hours.

Report

1. Adhesive identification including: Manufacturer, code number, type, date manufactured, lot number.
2. Conditioning procedure for material prior to testing.
3. Test conditions including: Plate size, gap size, frequency, tests performed, % strain for each test.
4. Induction cure profile for each test and each adhesive.
5. Graphs of $\log_{10}$ complex shear modulus, G*, vs time for each test performed and each adhesive.

References

1. Bird, R.B., Armstrong, R.C., and Hassager, O., Dynamics of Polymeric Liquids, Volume 1: Fluid Mechanics [2nd Edition, New York: John Wiley & Sons, 1987].
2. Macosko, C.W., Rheology: Principles, Measurements, and Applications [New York: Wiley-VCH, 1994].
3. ISO 6721, Part 10 Plastics – Determination of Dynamic Mechanical Properties, Part 10, Complex Shear Viscosity Using a Parallel-Plate Oscillatory Rheometer.

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