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subject : Cure Schedule for Stycast 2651/Catalyst 11

Executive Summary

The Henkel technical data sheet (TDS) for Stycast 2651/Catalyst 11 lists three alternate cure schedules for the material, each of which would result in a different state of reaction and different material properties. Here, a cure schedule that attains full reaction of the material is defined. The use of this cure schedule will eliminate variance in material properties due to changes in the cure state of the material, and the cure schedule will serve as the method to make material prior to characterizing properties. The following recommendation was motivated by (1) a desire to cure at a single temperature for ease of manufacture and (2) a desire to keep the cure temperature low (to minimize residual stress build-up associated with the cooldown from the cure temperature to room temperature) without excessively limiting the cure reaction due to vitrification (i.e., material glass transition temperature, T_g, exceeding cure temperature). The recommended cure schedule is as follows:

15 hours @ T=120C

The time and temperatures are what the Stycast 2651/Catalyst 11 material must experience to attain full cure. Thus if material temperature lags oven temperature, this should be accounted for. Ramp rates to temperature are not specified and are not critical assuming (1) there is not significant heating due to the reaction exotherm and (2) that cracking and debonding associated with the stress due to cure are not an issue. We acknowledge that shorter reaction times may be desired and that cure times as short as 5 hours attain a T_g within 10-15C of the fully-cured T_g (~125C). If such changes in the T_g are acceptable to the product, shorter cure times may be considered.

Introduction

The Henkel technical data sheet (TDS) for Stycast 2651/Catalyst 11[1] lists three alternate cure schedules for the material:

1. 8-16 hours @ T=80C
2. 2-4 hours @ T=100C
3. 0.5-1 hours @ T=120C

[1] June 2010 revision, see Appendix

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A “post cure” of the material for 2-4 hours at the highest expected use temperature is also suggested.

Since there is no explicit mention of differences in the resulting material after the listed cure schedules, one might be tempted to assume that all schedules result in the same product and use whichever one is most convenient for a particular process. On the other hand, the suggestion of a “post cure” implies that none of the recommended schedules result in full cure. It turns out that the three recommended cure schedules do not result in the same product. The differences in the product after each of the three cure schedules listed above will not be explicitly shown here. Rather, the focus of this work will be on the state of the material after the cure schedule in #3: isothermal cure at T=120C. It will be shown that the material does not reach a full extent of cure at the recommended 1 hour cure time. And at even lower cure temperatures (e.g., cure schedules #1 and #2), the material vitrifies during cure and will not reach a full extent of reaction in a reasonable amount of time, if ever. Methods of attaining a full extent of reaction in the material will then be demonstrated. Finally, a recommendation on the cure schedule to be used will be made.

Experimental

The polymerization reaction that occurs in the Stycast 2651/Catalyst 11 material is exothermic and enables an evaluation of the cure schedule using calorimetric techniques. In this case the focus will be on the heat produced during the polymerization reaction and on the temperature at which the glass transition of the material occurs after the reaction process. These two quantities enable an assessment of when the reaction is occurring/completed and the glass transition temperature (T_g) of the material, respectively. In all work below, the material was mixed according to the recommendations in the TDS,[2] placed in an aluminum pan, and tested on a Q2000 differential scanning calorimeter (TA Instruments) as soon as possible after mixing. For brevity, we present only how the T_g approaches the fully-cured T_g (as a measure of whether the reaction has reached completion) with cure time in Figure 1. If more details of the experimentation performed on this material (e.g., compressive stress-strain, etc.) are of interest, please contact the authors.

[2] 100:8.5 parts by weight mix ratio of Stycast 2651:Catalyst 11

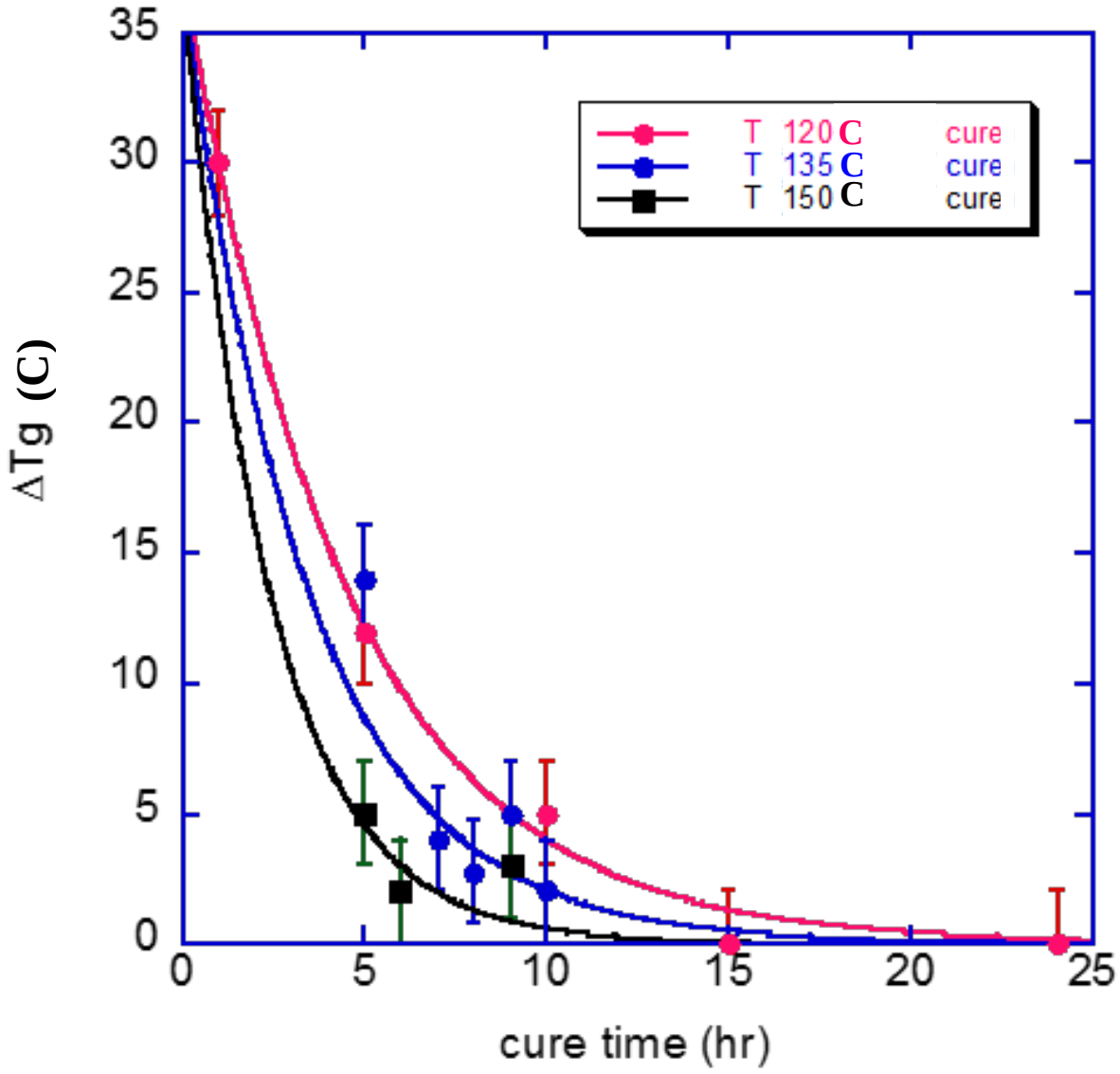

Figure 1. The difference between the fully-cured T_g and the T_g after a given amount of cure time for the Stycast 2651/Catalyst 11. Three independent cure temperatures are evaluated: 120, 135, and 150C. The curves are provided only as guides for the eye.

To obtain the data in Figure 1, the material was (1) cured at the indicated time and temperature, (2) quenched well below the T_g, (3) heated at 10C/min to well above T_g and held isothermal for 10 min, (4) cooled at 10C/min to well below T_g again, and (5) heated at 10C/min to well above T_g again (in some cases an additional cool and heat were completed to verify that the T_g was equivalent in subsequent heating cycles). When assessing the data, midpoint T_gs were evaluated for the initial heating after cure and for the fully-cured material (when T_g no longer increased with additional heating). The $\Delta T_g = T_g$(full cure) – T_g(initial heating). We acknowledge that "physical aging" occurred during the T=120C cure, as defined enthalpic peaks were observed that grew in magnitude with increasing cure time. This is typical of materials that have been held at a temperature below T_g (or "aged") for an extended amount of time and 120C is less than the fully-cured T_g (~125C). So once the T_g of the material is above 120C, the physical aging process begins. If necessary, the material can be "rejuventated" (or "unaged") by heating above T_g,

as is done in the subsequent heating cycle in this testing. From the above plot, we estimate the following cure conditions result in a fully-cured material:

1. 15 hours @ T=120C
2. 10 hours @135C
3. 6 hours @ 150C

Discussion and Recommendation

It is desired to use the Stycast 2651/Catalyst 11 material in such a way that material properties are known and do not vary amongst batches and/or products. Defining a cure schedule that attains full reaction of the material will help achieve this. The data in Figure 1 demonstrate that even the highest temperature cure recommended in the TDS does not attain full reaction of the material. This led to the evaluation of longer cure times at T=120C and cure at higher temperatures of 135C and 150C.

All of the cure temperatures tested give an equivalent ultimate T_g, suggesting final properties are independent of the cure history of the material as long as cure is taken to completion. During the extended T=120C cure times, "physical aging" of the material was apparent due to the T_g of the material exceeding the cure temperature. While the physical aging would certainly embed a different history in the material that would have implications on the physical response of the material under a mechanical load and on the stress-state within a confined material (e.g., the polymer bonded to rigid interfaces), this history could be erased by "annealing" the material above its T_g and cooling back down to room temperature to impose a known history in the material. When the material is cured at the higher temperatures, 135 and 150C, it is always above T_g and no aging occurs during the cure process. The history of the material is defined by the cooldown conditions from the cure temperature to room temperature. Of course, at the higher cure temperatures the difference between cure temperature and room temperature is larger and thermal expansion mismatches between the polymer and the surfaces it is bonded to will induce a larger residual stress in the system for the higher ΔT associated with the cooldown. This can be important if the residual stress levels are high enough to break components in the product design or crack/debond the polymer. However, if stress levels are low relative to these "failure points", the shorter cure schedules at the higher temperatures are an option.

Since application drivers (at the time anyway) were to (1) cure at a single temperature for ease of manufacture and (2) keep the cure temperature low (to minimize residual stress build-up associated with the cooldown from the cure temperature to room temperature) without excessively limiting the cure reaction due to vitrification (i.e., material glass transition temperature, T_g, exceeding cure temperature), a cure schedule at T=120C is recommended. To attain full cure at T=120C a 15 hour reaction time is necessary. We acknowledge that shorter reaction times may be desired and that cure times as short as 5 hours attain a T_g within 10-15C of the fully-cured T_g (~125C). If such changes in the T_g are acceptable to the product, shorter cure times may be considered.

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Appendix

Hysol®

STYCAST 2651/Catalyst 11

June 2010

PRODUCT DESCRIPTION

STYCAST 2651/Catalyst 11 provides the following product characteristics:

Technology	Epoxy
Appearance (Resin)	Black
Components	Two component - requires mixing
Mix Ratio, by weight - Resin : Hardener	100 : 8.5
Mix Ratio, by volume - Base : Hardener	100 : 12.5
Product Benefits	• General purpose • Excellent adhesion • Excellent electrical properties • Thermally conductive • Long pot life • Excellent chemical resistance • Excellent physical and chemical properties at elevated temperatures
Cure	Heat Cure
Application	Encapsulant
Operating Temperature	-55 to 155 °C

STYCAST 2651/Catalyst 11 is a dielectric grade epoxy encapsulant designed for general purpose and has excellent adhesion to a wide variety of substrates.

STYCAST 2651/Catalyst 11 passes NASA outgassing standards.

STYCAST 2651 can be used with a variety of catalysts. For more information on mixed properties when used with other available catalysts, please contact your local technical service representative for assistance and recommendations.

TYPICAL PROPERTIES OF UNCURED MATERIAL

Part A Properties *2651*

Viscosity, Brookfield , 25 °C, mPa·s (cP):	
Speed 5 rpm, # 7	225,000
Specific Gravity	1.65
Shelf Life @ 25°C, months	6
Flash Point - See MSDS	

Part B Properties *Catalyst 11*

Viscosity @ 65 °C, mPa·s (cP)	35 to 60
Flash Point - See MSDS	

Mixed Properties

Mixed Viscosity, mPa·s (cP)	25,000
Specific Gravity	1.56
Working Time, 100 g mass, @ 25ºC, hours	>4
Flash Point - See MSDS	

TYPICAL CURING PERFORMANCE

Cure Schedule

8 to 16 hours @ 80°C or
2 to 4 hours @ 100°C or
30 to 60 minutes @ 120°C

Post Cure

Post Cure: 2 to 4 hours at the highest expected use temperature

The above cure profiles are guideline recommendations. Cure conditions (time and temperature) may vary based on customers' experience and their application requirements, as well as customer curing equipment, oven loading and actual oven temperatures.

TYPICAL PROPERTIES OF CURED MATERIAL

Physical Properties:

Coefficient of Thermal Expansion TMA:		
Below Tg, ppm/°C		40
Thermal Conductivity, W/mk		0.66
Hardness, Shore D		88
Water Absorption 24 hours, %		0.1
Compressive Strength, psi		16,000
Flexural strength , ASTM D790	N/mm² (psi)	103 (15,000)
Tensile Strength, psi		9,000

Electrical Properties:

Dielectric Strength, volts/mil	450
Volume Resistivity @ 25ºC, ohm-cm	5×10^{16}
Dielectric Constant :	
60Hz	4.8
1 kHz	4.6
1 mHz	3.9
Dissipation Factor:	
60Hz	0.02
1 kHz	0.01
1 mHz	0.02

Outgassing Properties:

Total Mass Loss, %	0.63
Collected Volatile Condensable Material, %	0.01

GENERAL INFORMATION

For safe handling information on this product, consult the Material Safety Data Sheet, (MSDS).

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DIRECTIONS FOR USE

1. Complete cleaning of the substrates should be performed to remove contamination such as oxide layers, dust, moisture, salt and oils which can cause poor adhesion or corrosion in a bonded part.
2. Some separation of components is common during shipping and storage. For this reason, it is recommended that the contents of the shipping container be thoroughly mixed prior to use.
3. Accurately weigh resin and hardener into a clean container in the recommended ratio.
4. Blend components by hand, using a kneading motion, for 2 to 3 minutes and scrape the bottom and sides of the mixing container frequently to produce a uniform mixture.
5. If possible, power mix for an additional 2 to 3 minutes. Avoid high mixing speeds which could entrap excessive amounts of air or cause overheating of the mixture resulting in reduced working life.
6. To ensure a void-free embedment, vacuum deairing should be used to remove any entrapped air introduced during the mixing operation.
7. Pump-down or pull vacuum on the mixture to achieve an ultimate vacuum or absolute pressure of 1 to 5 torr or mm Hg. The foam will rise several times in the liquid height and then subside.
8. Continue vacuum deairing until most of the bubbling has ceased. This usually takes 3 to 10 minutes.
9. To facilitate deairing in difficult to deair materials, add a few drops of an air release agent, such as ANTIFOAM 88 into 100 grams of mixture.
10. Gentle warming will also help, but pot life will be shortened.
11. Pour mixture into cavity or mold.
12. Gentle warming of the mold or assembly reduces the viscosity. This improves the flow of the material into the unit having intricate shapes or tightly packed coils or components.
13. Further vacuum deairing in the mold may be required for critical applications.

Storage

Store product in the unopened container in a dry location. Storage information may be indicated on the product container labeling.

Optimal Storage: 25 °C

Material removed from containers may be contaminated during use. Do not return product to the original container. Henkel Corporation cannot assume responsibility for product which has been contaminated or stored under conditions other than those previously indicated. If additional information is required, please contact your local Technical Service Center or Customer Service Representative.

Certain resins and hardeners are prone to crystallization. If crystallization does occur, warm the contents of the shipping container to 50 to 60°C until all crystals have dissolved. Be sure the shipping container is loosely covered during the warming stage to prevent any pressure build-up. Allow contents to cool to room temperature before continuing.

Not for product specifications

The technical data contained herein are intended as reference only. Please contact your local quality department for assistance and recommendations on specifications for this product.

Conversions

(°C x 1.8) + 32 = °F
kV/mm x 25.4 = V/mil
mm / 25.4 = inches
N x 0.225 = lb
N/mm x 5.71 = lb/in
N/mm² x 145 = psi
MPa x 145 = psi
N·m x 8.851 = lb·in
N·m x 0.738 = lb·ft
N·mm x 0.142 = oz·in
mPa·s = cP

Note

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Reference 0.3