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Conformal Coatings for 225ºC

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National Nuclear Security Administration

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Disclaimer

- Much of this work is based around the use of epoxies and other materials manufactured by specific vendors. We do not endorse these specific materials, but we have tested them as a demonstration of a class of materials. Due to limited funding we can only test a small number of materials.

Outline

- Epoxy and Epoxy like compounds
  - 863, 868 … 4538S
  - DEG
- Parylene
- Ceramic Coatings for Higher Temperatures

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Why Conformal Coat

- Protection from vibration
- Protection from water vapor
- Protection from oxygen
- Protection from hydrogen

Two Varieties of Tools

- Drilling tools
  - Vibration (most important for drilling tools)
  - Lifetime
- Well monitoring tools
  - Lifetime (most important for monitoring tools)
  - Vibration

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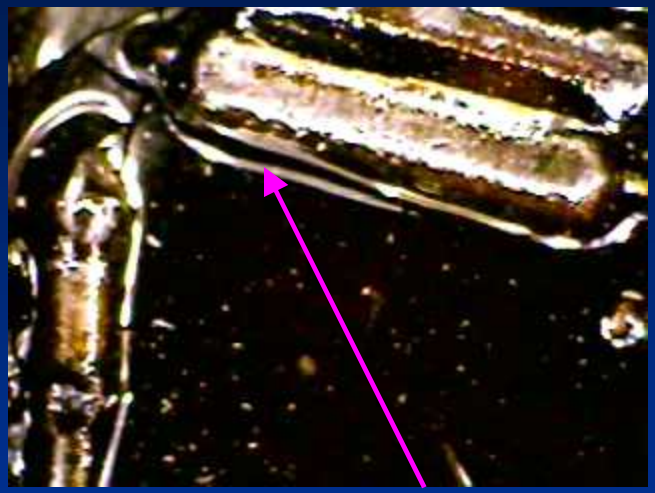
Older Cotronics Epoxies

- 868 260ºC Flexible
- 863 343ºC
- 4525 260ºC
- 4538 232ºC Super Flexible

868



- Sample circuit board shows cracks after heating.
- Material shows cracking after first heat cycle to 230ºC

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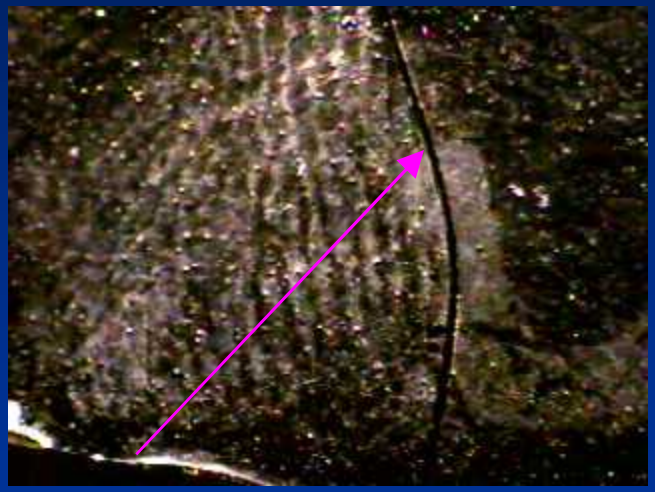
863



- Material shows cracks on sample circuit boards after one heat cycle to 230ºC

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4538S-100

- This sample shows a crack that developed as a result of 10 days in argon at 230ºC

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4525

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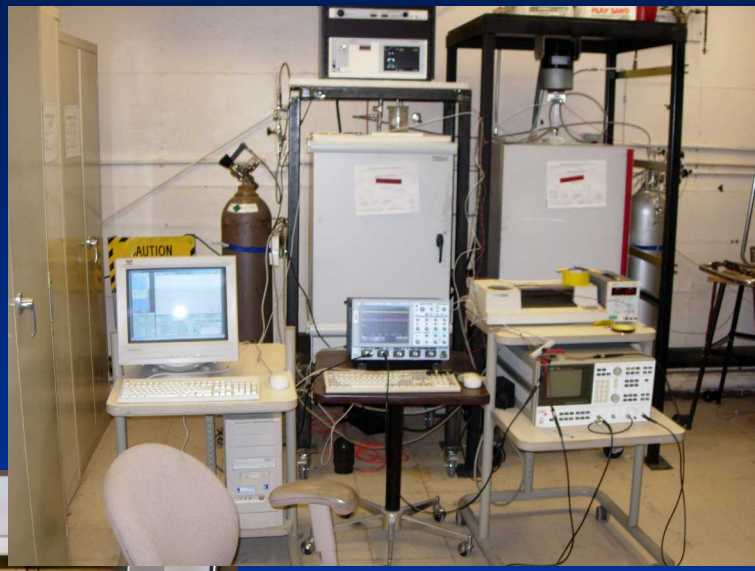
High Temperature Testing Methods

- Testing in argon (Ar) 500 hours
- Testing in 5% Hydrogen(H_2) and Ar 240 hours
- Testing in 100 PPM Water (H_2O) and Ar 280 hours

Oven System



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General Precautions

- Weigh your chemicals
- Check your oven temperature
- Vacuum de-gas epoxy products
- Good housekeeping is important

Some Days It Just Doesn't Pay To Get Up In The Morning !

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4538 Nomenclature

- 4538 or 4538S
  - 4538 is the standard product
  - 4538S is high temperature product
- A dash for readability
- The number of parts of hardener per 100 parts of resin
- Example 4538S-300

4538S

- Special high temperature resin - 100 parts
- Mix with 80 parts hardener yields a rigid epoxy
- Mix with 100 parts hardener yields a flexible epoxy
- Mix with 200 parts hardener yields a soft epoxy
- Mix with 300 parts hardener yields a very soft epoxy

4538S Preliminary Testing

Material	500 Hours Ar 230° C	240 Hours 5% H2/ 95% Ar 230°C	280 Hours 100ppm H2O /Ar 230 °C
4538S-80	Brittle; Dark in color; No cracks	Hard; Black; Some chipping on edge	Hard; Black; Brittle
4538S-100	Somewhat flexible; Dark in color; No cracks	Hard; Black; Some chipping on edge	Black; Dents slightly with pen; Brittle on edge
4538S-120	Flexible; Dark in color; No cracks	Hard Black; Inflexible	Black; Dents slightly with pen; Brittle on edge
4538S-200	Very flexible; Darker in color; Non-tacky; No cracks	Softer, Black ; No cracks	Black; Dents with pen
4538S-300	Very flexible; Darker in color; Non-tacky; No cracks	Soft; Black; No cracks	Black; Dents with pen

Mass Loss Testing

- Weigh samples
- Bake samples 99 hours in Ar at 230ºC
- Do this 10 times

Mass Loss Results 4538S

Hours Mix 100/	99	198	297	396	495	594	693	792	890	990
100	463	423	367	326	283	254	227	209	191	173
200	637	520	429	360	310	275	247	225	205	190
300	821	634	497	406	343	303	268	244	220	204

Hours vs. micrograms / gram loss per hour at 230ºC

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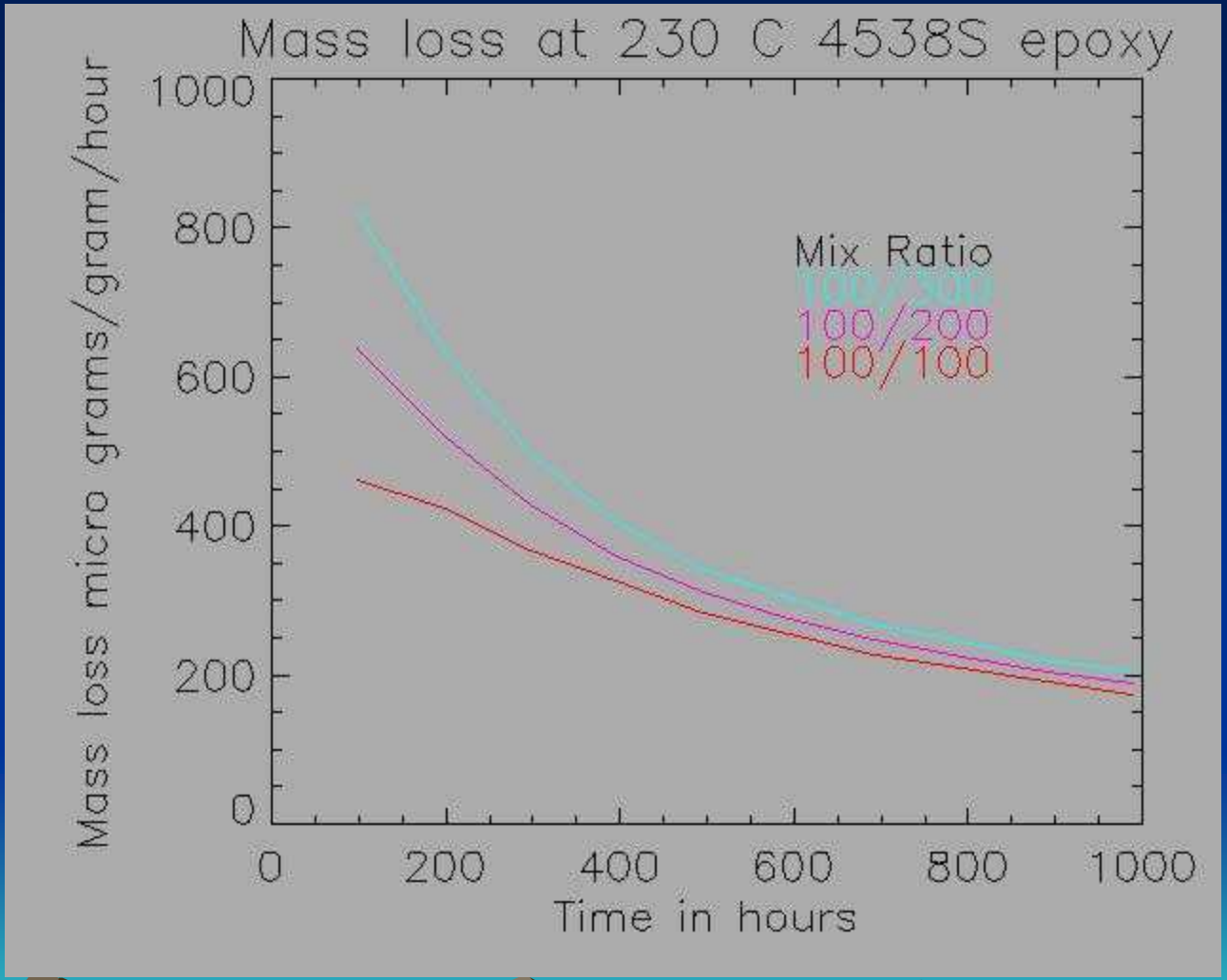
Mass Loss Graphic



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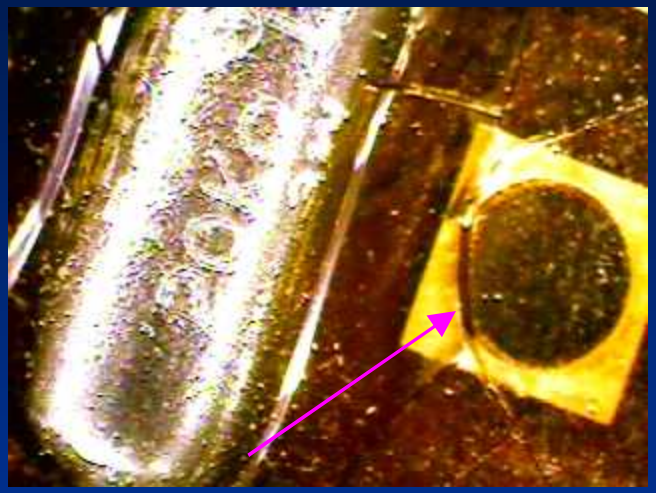
DEG



- Green Mountain Inc.
- DEG was originally intended as a lost circulation control material. We used a small amount of it for a trial conformal coating.

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DEG

- Green Mountain International's DEG material use as a coating for fiberglass wire and ceramic sleeving

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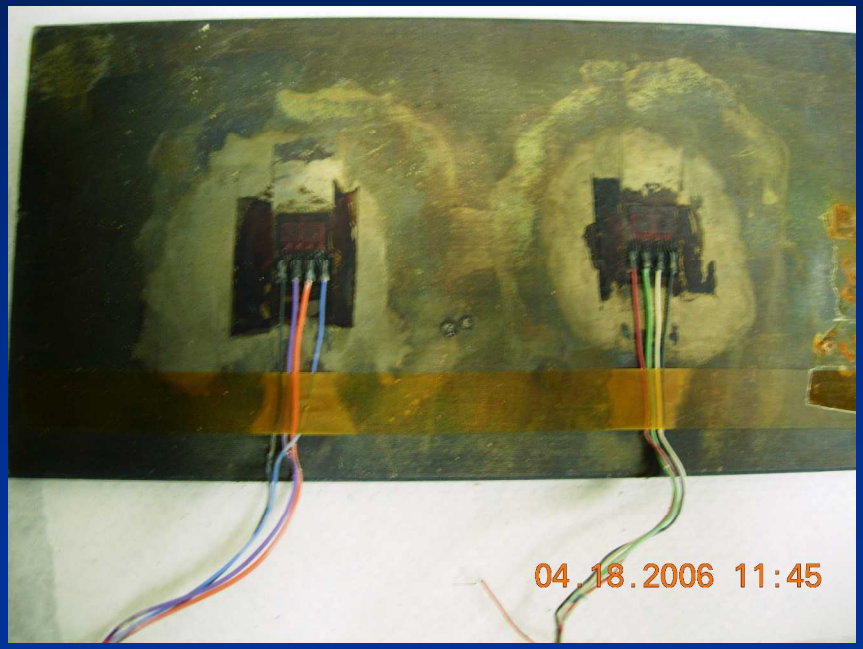
Parylene HT

- Some limited experience with this
- Available from Specialty Coatings Systems, Indianapolis, IN
- Very thin coating
- Expensive for prototype production
- Works to 250ºC, should work above that
- Does not work in parboil testing

Parylene HT



- Soaked in room temperature water for several days
- Strain gage resistances unaffected
- Very thin coatings not water resistant at high temperatures for long periods

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Silicon Based Materials

- Work well in absolutely dry environments
- Most sealing systems leak in long term deployments
- Silicon sheet turned to bubble gum in long term test at Coso, CA

RTV-60

- RTV can be used up to 225ºC
- RTV disintegrates rapidly in hot water or steam environments
- Not good for long term use

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Current Research

- Ceramic Coatings for 300ºC Electronics Packaging
- Two Areas of Interest
  - Capacitor potting
  - Wire wrap conformal coating

Ceramic Material Testing

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Ceramic Sleeving and Cloth

- Cotronics Thermeez 395 Tape and Sleeving
- Works well as padding and wire bundle
- Can be coated with DEG material if needed

Ceramic Potting for 300ºC

- Currently testing Cotronics Thermeeze 7030 silica
- Mix with water 20 to 40 parts by weight
- Hardens at room temperature
- 125ºC bake out helps at 40 parts
- Potting fly leaded capacitors for 300ºC use

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Ceramic Potting Continued

- Some limited testing on Cotronics Duralco 215
- Is a bit thicker than we like
- Did not do as well as 7030 material for potting capacitors

Ceramic Potted Wire Wrap

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Parboiled Mini Muffin
70 Hours at 200ºC

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Summary Materials

- 4538S-300 for conformal coatings to 230° C
- DEG is useful as a coating
- Parylene HT for production coating
- Ceramic materials for higher temperatures

Testing Methods Summary

- 500 hours at 230° C in Ar
- 240 hours at 230° C. 5% H_2 / Ar
- 280 hours at 230° C 100 ppm H_2O / AR
- Parboil test materials if high water content is expected

Summary

- Watch the environment
- Degas potting materials
- Inert gas fill tools
- Water is not your friend even in small amounts
- Hydrogen has little effect on potting
- You can’t stop hydrogen

Additional Resources

- Sandia's Geothermal Research Department

  Phone (505)-844-3933

  Web www.sandia.gov/geothermal/
- Qualified Components List
- Useful Equipment List

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Questions

- The only truly unacceptable question is the one you did not ask and should have

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Useful parts for high temperature testing

- The following slides contain a list some of the items used in high temperature testing at Sandia.

Tubing and Fittings

- Swagelok pre-swaged tubing sticks

Part #	Description	Price ($ ea)
SS-4-SPS-2	¼” Stainless Steel Swagelok 2”	10.00
SS-4-SPS-4	¼” Stainless Steel Swagelok 4”	10.50
SS-4-SPS-6	¼” Stainless Steel Swagelok 6”	11.00
SS-4-SPS-8	¼” Stainless Steel Swagelok 8”	11.50
SS-4-SPS-10	¼” Stainless Steel Swagelok 10”	12.00
SS-4-SPS-12	¼” Stainless Steel Swagelok 12”	12.50

Tubing and Fittings

- Albuquerque Valve and Fitting
  - Ed Whitehouse
  - 505-842-0213
- http://www.swagelok.com/

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Ovens

- Fisher Isotemp programmable oven
- Catalog Number
  13-247-851F
- 240 VAC 20 A 1θ
- Max temperature 325º C

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Ovens

- Thelco All purpose laboratory oven model 70DM
- Catalog number 51221157
- 120 VAC 20A 1θ
- Max temperature 250ºC

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Furnace

- Fisher Isotemp programmable muffle furnace
- Catalog number
  10-650-14
- 120VAC 15A 1θ
- Max temperature 1125º C

Sample tubes- Small

- Kurt J. Lesker Co. 1-800-245-1656
- http://www.lesker.com/newweb/index.cfm
- Small sample vessels: 2” OD x 12”
- 1 FN-C4134120 full nipple 12” OAL 3-3/8” UHVS 1-Fixed 1 Rotating 2”OD tube
- 1 F0337X000N CF 3-3/8 blank flange
- 1 of the following
- CF-041201B with 1 ½”x 14 ntp and 1 ¼” x 18 npt ports with 1 Swagelok SS-400-1-4 and 1 Swagelok SS-810-1-8
- or
- CF-041201A with 2 ¼”x 18 npt ports with 2 Swagelok SS-400-1-4
- 2 GA-0337 Copper gaskets (10 gaskets)
- 1 HBS31224175 Bolt set (25 bolt nut washer sets)

Sample tubes- Large

- Large sample vessels: 3” OD X 20” These fit upright in Fisher oven
- 1 FN-C6136200 full nipple 20”OAL 4-5/8” UHVS 1 Fixed 1 Rotating 3” OD tube
- 1 FO-462X000N CF 4-5/8” blank flange
- 1 CF-050308A with with 1 ½”x 14 ntp and 1 ¼” x 18 npt ports with 1 Swagelok SS-400-1-4 and 1 Swagelok SS-810-1-8
- 2 GA-0462 Copper Gaskets (10 gaskets)
- 1 HBS31224225 bolt set (25 bolt nut washer sets)

Sample Tube WARNING

- These are vacuum components, they are not intended for pressure use. Do not exceed 10psig. Make certain that copper gasket seats properly before tightening. Use nickel based anti-seize on bolts. (Loctite item # 77164)

Sample Tubes

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Regulator/Flow Meter

- http://www.mathesontrigas.com
- Or local compressed gas vendor
- 81-AF-580 for nitrogen or argon
- 81-AF-350 for H2 in argon
- Remove provided hose barb fitting and add Swagelok part # SS-400-1-2 to convert to ¼ Swagelok
- Add Swagelok part # SS-4-A-RFO-020 flow restrictor to input side of regulator

Regulator/Flow Meter

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Gas Manifold

- Build at least one manifold for each gas mix being supported
- Use Swagelok pre-swaged sticks where possible

Gas Manifold ¼ inch

Part #	#	Description
SS-1RS4	4	Needle valve
SS-4-SPS-4	4	4” pre-swaged tubing
SS-CHS4-1	4	One way valve
SS-RL3S4	1	10 psi pressure relief valve
SS-401-PC	4	Port connector
SS-400-9	2	Union elbow
SS-400-3	2	Union tee
SS-400-4	1	Union cross
SS-4-SPS-2	4	2” pre-swaged tubing
SS-4BHT-XX	1	Flex hose XX denotes length in inches

Gas Manifold

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Vibration System

- Labworks Inc. (714)-549-1981
- Labworks ET-140 vibration system
- VL-144 controller
- Fisher Isotemp oven
- Custom Frame

Vibration System

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