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Conformal Coatings for 225° C Applications

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Abstract

Sandia has conducted a number of tests in search of conformal coating products that function acceptably at 225º C or higher. This paper documents the work associated with the initial testing of organic and organic-inorganic materials for this purpose and provides information on materials found which failed initial testing and those materials which show promise. The report also provides insight into our testing process, which is designed to represent the wellbore environment.

Keywords

High temperature Conformal coating, Epoxy, High temperature adhesive

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Introduction

The function of any conformal coating product is to insure that components used to construct a circuit board are protected from vibration, water vapor and oxygen. Additionally, within any high-temperature well there are small amounts of free hydrogen. Therefore, we require wellbore instrumentation to operate within a hydrogen rich environment. The development of high temperature drilling tools such as the Sandia Diagnostic While Drilling (DWD) tool created a need to develop high temperature conformal coating and adhesive technologies.

History

Preliminary testing was performed on a number of compounds, which are commonly available. This testing was done in a narrow temperature range of 230° C to 250° C. The very earliest work was done in air, but the results were unsatisfactory. All further testing was conducted in an argon-based atmosphere.

Initial test results

Table 1 shows some early conformal coating test results. Note that most coatings exhibited cracking on the first temperature cycle. Cracking is an unsatisfactory behavior for conformal coating in downhole tools. Other materials need to be investigated.

Table 1 Early Test Results

Material	99 hour 230ºC in argon	Notes
Green Mountain DEG	Minor cracking after 99 hour run. Fails in brittle mode; Remains Clear	Useful for coating for high temperature fiberglass wire. Turns black and brittle after long exposures
Cotronics 868	Significant cracking after 99-hour run. Fails in brittle mode. Stays fairly clear in color	Cracks on initial exposure to temperature. Cracks do not appear to worsen with further exposure
Cotronics 863	Significant cracking after 99-hour run. Fails in brittle mode. Darkens slightly in color	Cracks on initial exposure to temperature. Cracks do not appear to worsen with further exposure
Cotronics 4525	No cracking noted after 99 hour run	Black in color, Material is brittle.

Test Design

We began our search for a suitable conformal coating by searching the web for flexible epoxies, which we felt might be a way to accomplish our objectives for conformal coating. We located one epoxy compound from Cotronics Corporation in Brooklyn, NY that seemed interesting. It was an epoxy called 4538 that could be mixed with varying ratios of resin to hardener to achieve a finished epoxy that could be varied from hard to quite flexible depending on the mix ratios. In our discussions with the company we were informed that Cotronics has a new formulation of the resin that should tolerate higher temperatures that the 232 º C that was listed in the literature. The use of an epoxy that could be mixed at varying ratios somewhat complicated the test design, because we needed to evaluate many more samples. The supplier recommends that the epoxy can be used in mix ratios of 100 parts resin to 80 parts hardener for a more hard final product to 100 parts resin to 300 parts hardener for a flexible final product.

We developed the following nomenclature system to identify the various products and mixes used: 4538 or 4538S to identify the product. The former is the standard product from Cotronics and the latter is the Special high temperature resin formulation. Add a dash for readability and the parts of hardener; 80,100,200,300. An example sample designator would be 4538S-300 for an epoxy that used the 4538 high temperature resin mixed at 100 parts resin and 300 parts hardener.

To initially qualify the material we tested each sample under the following conditions:.

1) 500 hours at 230º C in an atmosphere of argon.
2) 240 hours at 230° C. in atmosphere consisting of 5% hydrogen and the remainder argon.
3) 280 hours at 230° C in an atmosphere of 100 ppm water and the remainder argon.

Test Setup

The test hardware is designed to allow a low volume flow of the test gas through the experimental samples at all times. The gas flows from a standard gas bottle through a two-stage regulator/ flow meter. A flow restricting orifice is added to the regulator for safety reasons. The regulator is set for 5 psi. and the flow rate is adjusted to 0.007 cuft/min . From there the gas flows to a manifold where it can be further regulated. Four separate sample lines exit the manifold each connects through a check valve and stainless steel tubing to a sample chamber inside the oven. Each sample chamber has a stainless steel exhaust tube that exits the oven and is discharges into a water bath.. The water bath is used to maintain a very low backpressure on the system and ensure that air does not enter the test chamber in the case of pressure loss at the gas bottle. Figure 1 documents the gas flow through one of the four sample chambers in a typical test

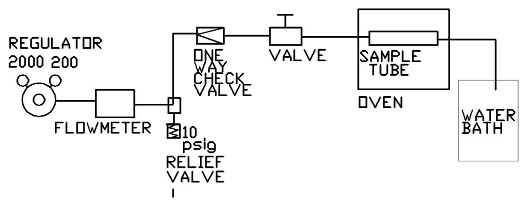

Figure 1 Gas flow diagram for testing

The oven used is a Thelco laboratory oven (catalog # 51221157) which accommodates up to four test chambers. The oven is capable of reaching 250ºC and provided a timing function which can be programmed up to 99 hours in 1 hour increments. Figure 2 shows the oven and associated equipment.

Figure 2 Test oven

Sample preparation

Each sample was mixed in a small aluminum container. The samples were then air cured in an oven at 125° C for 1 hour and the oven allowed to return to ambient temperature overnight. Next, the samples were cut into usable sized pieces and placed in aluminum sample dishes. The sample dishes were then placed in the sample chambers and connected to the gas source. The first sub test performed was done in an argon atmosphere lasting a total of 500 hours. The test included one temperature cycle. The samples were then evaluated and the second test started. The second test consisted of the same samples being subjected to 5% H_2 in argon for 240 hours. The samples were again examined. Finally the samples were subjected to 280 hours at 230° C in an atmosphere of 100 ppm water in argon. In the water test, a slight amount of dark colored liquid was noted on flanges of the sample chambers. It is unknown if this dark liquid was related to changes in the test materials, or simply water that had condensed from the gas.

Test results

The results of the tests were encouraging ,all of the samples survived the three oven tests. After each of the tests, the samples were visually evaluated as to there performance. The combined results are shown in Table 2. Notice that the samples of 4538-80 and 4538S-80 showed some brittle nature. This is expected as the manufacture specifies this mix ratio as producing a rather inflexible epoxy. Figure 3 shows a test sample after completion of the three test sequence. As a result of this test further testing was planned to characterize the mass loss that could be expected in the epoxies as a result of long term exposure to high temperatures.

Figure 3 4538-300 Sample after testing

Mass loss testing

One known problem with epoxy-based materials is the loss of mass over time at temperature. In order to evaluate the amount of mass loss by the various epoxy mixtures, a longer-term test was performed. For this test approximately 100 gram test samples were mixed, vacuum degassed, and then poured into ice cube trays that had been sprayed with a mold release that had been recommended by the

Table 2
Results for various epoxies in selected atmospheres

Material	500 Hours in argon at 230º C	240 Hours 5% H_2 95% argon at 230ºC	280 hours 100ppm H_2O remainder argon at 230 ºC
4538-80	Brittle; Dark in color; No Cracks	Hard; Black; Some chipping on edge	Hard; Black ;Brittle
4538-100	Somewhat flexible; Dark in color; No Cracks	Hard; Black; Little chipping on edge	Hard; Black ;Brittle
4538-120	Somewhat flexible; Dark in color; No Cracks	Hard; Black; Little chipping on edge	Hard; Black; Brittle
4538-200	Flexible; Darker in Color; Non-tacky; No Cracks	Slightly flexible; Black; No cracking	Hard; Black; Does not dent with pen
4538-300	Flexible; Darker in Color; Non-tacky; No Cracks	Slightly flexible; Black; No cracking	Black, Slightly soft
4538S-80	Brittle; Dark in color; No Cracks	Hard; Black; Some chipping on edge	Hard; Black; Brittle
4538S-100	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

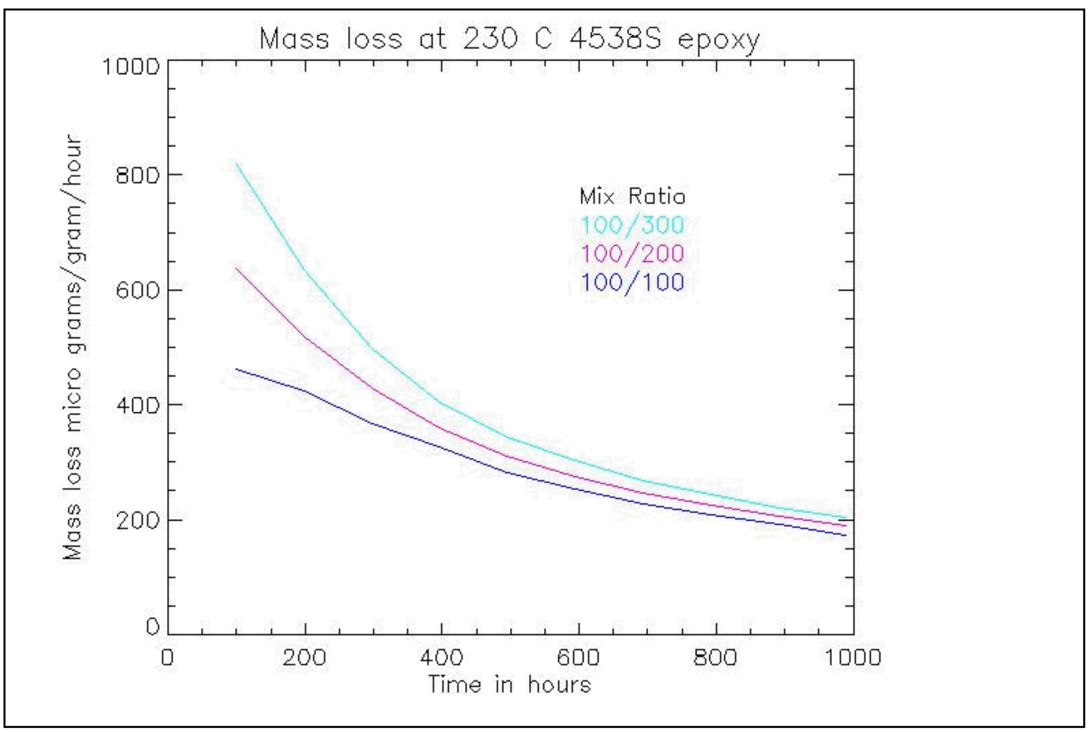

Figure 4 Epoxy mass loss as a function of time at temperature

manufacture. The resulting epoxy cubes had a mass of approximately 20 grams.

Mass loss measurements

Twenty gram ice cube samples of 4538S-100, 4538S-200 and 4538S-300 were prepared as described previously. The samples were allowed to cure over night, removed from the ice cube trays and then loaded into small aluminum sample dishes, weighed and placed in the sample chambers. Each chamber contained three samples, one from each mixture of 4538S. The sample chambers were then placed in the oven and plumbed to allow a very slow rate of flow of low-pressure (~ 5-psi) argon gas continuously through the each of sample chambers. The gas flow path and oven setup is similar to that explained earlier. The samples were heated from room temperature to 230°C as rapidly as possible and soaked at 230ºC for 99 hours. After each 99-hour cycle the samples were cooled over night to room temperature, removed from the sample chambers and weighted. At each 99 hour increment the results were tabulated as total mass lost divided by the total starting mass divided by the total number of hours at temperature. The three samples of each mix were averaged and the results plotted vs. time. Figure 4 shows the results of the test in micrograms per gram per hour (µgm/gm/hr).

Mass loss experiment analysis

The loss of mass in these epoxies is most sever in the first 100 hours of high temperature exposure. The more hardener used with 4538S the more mass loss the mixture will experience on a gram/gram basis. The epoxies losses at 99 hours were 427µgm/gm/hr for the 4538S-100 material, 594 µgm/gm/hr for the 4538S-200 and 761 µgm/gm/hr for the 4538S-300 material. The losses at 990 hours for the materials were160 µgm/gm/hr for 4536S-100, 177 µgm/gm/hr for 4538S-200 and 190 µgm/gm/hr for 4538S-300. Figure 4 indicates that baking the conformal coating for approximately 500 hours would significantly reduce the out gassing inside the tool and would stabilize the coating material.

Summary

We have found that the 4538S-80 mix is too brittle for most of our applications. We have been most encouraged by the 4538S-300 mixture. It does not crack through 1000 hours of the testing that we have done yet it cures to a kind of tacky jellylike state on first cure. It can be shaped by various methods before baking it out at 230° C. It is important to provide an inert atmosphere when using 4538S at temperatures above 125ºC. There is limited evidence that the epoxy can withstand short excursions to 250ºC. Exposure of this material to 290ºC will cause it to fail.

When 4538S is used as a potting or conformal coating material in electronics assemblies which are to be sealed, as in our case for downhole tools, it is important to assure that the 4538S is cured at a temperature that is somewhat higher than the expected operating temperature for at least 100 hours to limit the amount mass loss that the epoxy experiences while sealed into the tool. Secondly, it is important to fill the tool with an inert atmosphere to limit the damage that oxygen does to the epoxy and other electronic and mechanical components.

Notes on using 4538S-300

Weigh both the resin and hardener components to achieve a good 100/300 ratio. Mix the components thoroughly. Vacuum degassing of the mixed 4538S must be carried out to obtain the flexible nature the epoxy . Pour the mixed material on the object of interest; try to avoid entraining air if possible. We found it useful to allow the epoxy to air dry overnight before baking material in air for four hours at 125° C. After the initial curing, the material will still be somewhat tacky and can be shaped at this point. When shaping is complete, harden the material by heating it to 230ºC for 4 hours in an inert atmosphere. The final temperature rating of 4538S has not yet been determined, but limited testing indicates that this product may be useful for short term exposures up to 250ºC. This product is unsatisfactory for applications above 250ºC and in fact at 290ºC it melts. We have experimented with the mixing ratio above 300 parts hardener. The results were not satisfactory as the epoxy never sets up. Please feel free to contact the author for more detailed instructions on 4538S usage.

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