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Title: EVALUATION OF MS-122 MOLD RELEASES

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From/MS: M. Dirmeyer
Phone: 505-665-8778
Date: July 19th, 2018

SUBJECT: EVALUATION OF MS-122 MOLD RELEASES

Executive Summary

Miller Stephenson polytetrafluoroethylene-based mold releases were evaluated; the mold releases include MS-122E, MS-122SE, MS-122AD, MS-122ADL, and MS-122XD. The mold releases were tested for cure inhibition of Sylgard 184 90/10. These mold releases were also tested for shear flow stability through a defined gap on a uranium alloy surface. Mold releases MS-122E, MS-122SE, MS-122AD, MS-122ADL, and MS-122XD were all tested. SEM images comparing the lay down of each mold release on a flat silicon surface were also compared. MS-122 XD showed signs of cure inhibition of Sylgard 184 90/10. All mold releases were detected on uranium alloy surfaces via Infrared Spectroscopy (FT-IR) after flowing Sylgard 184 90/10 across the uranium surface indicating the mold release coating is stable under flow. All SEM images of the mold releases studied are comparable showing rough surfaces with micron-sized particles.

Introduction

Miller Stephenson (MS) polytetrafluoroethylene (PTFE)-based mold release have been used for a variety of applications. A variety of these PTFE-based mold releases have been used complex wide. While in general, these mold releases are similar, there are slight variations in formulations. The formulations of the mold releases evaluated in this study are listed in Tables 1-5.

Component	CAS #	Weight %
1,1,1,2,2,3,4,5,5,5-Decafluoropentane (HFC-43-10mee)	138495-42-8	18-23
1,1,1,3,3-Pentafluorobutane (HFC-365mfc)	406-58-6	25-30
Trans,1.2-Dichloroethylene	156-60-5	25-30
Isopropyl Alcohol	67-63-0	2-4
1,1,1,2-Tetrafluoroethane	811-97-2	18-22
Poly-TFE, Alpha-Hydro-Omega-(Methylcyclohexyl)	65530-85-0	<1.5
Polytetrafluoroethylene	9002-84-0	<0.5

Table 1: Formulation for MS-122E

Component	CAS #	Weight %
1,1,1,2,2,3,4,5,5,5-Decafluoropentane (HFC-43-10mee)	138495-42-8	40-50
Trans,1.2-Dichloroethylene	156-60-5	50-60
1,1,1,2-Tetrafluoroethane	811-97-2	18-22
Poly-TFE, Alpha-Hydro-Omega-(Methylcyclohexyl)	65530-85-0	<1.5
Polytetrafluoroethylene	9002-84-0	<0.5

Table 2: Formulation for MS-122SE

Component	CAS #	Weight %
1,1,1,2-Tetrafluoroethane	811-97-2	88-92
Isopropyl Alcohol	67-63-0	5-10
Poly-TFE, Omega-Hydro-Alpha-(Methylcyclohexyl)-	65530-85-0	1-2
Poly-Tetrafluoroethylene	9002-84-0	<1

Table 3: Formulation for MS-122AD

Component	CAS #	Weight %
Trans-1,3,3,3-Tetrafluoroprop-1-ene (HFO-1234ze)	29118-24-9	88-92
Isopropyl Alcohol	67-63-0	5-10
Poly-TFE, Omega-Hydro-Alpha-(Methylcyclohexyl)-	65530-85-0	1-2
Poly-Tetrafluoroethylene	9002-84-0	<1

Table 4: Formulation for MS-122ADL

Component	CAS #	Weight %
1,1,1,2-Tetrafluoroethane	811-97-2	80-90
1,1,1,2,2,3,4,5,5,5-Decafluoropentane (HFC-43-10mee)	138495-42-8	5-10
Poly-TFE, Omega-Hydro-Alpha-(Methylcyclohexyl)-	65530-85-0	1-2
Poly-Tetrafluoroethylene	9002-84-0	<1

Table 5: Formulation for MS-122XD

Material/Instrumentation

Sylgard 184 (90/10) was provided by Linda Meincke as three 450 gram kits. The kits were labeled as LJM2018150- 20, 21, and 22; with an expiration date of 11/30/18. Kit 20 was used for the cure inhibition study and Kit 21 was used for the Sheer Stability study. Part A and Part B of the kit were mixed in a 10:1 mixture, gravimetrically. This mixture was mixed by hand for 3 minutes. The mixture was then degassed at room temperature via repeated pump/purge cycles until no bubbles were visible; a vacuum oven at room temperature was used and samples were pumped for at most 3 minutes and vented to atmospheric pressure. This process was cycled three times or until the material appeared bubble free via visual inspection. Any mixed Sylgard was used within 15 minutes of mixing.

Fourier Transform Infrared Spectroscopy (FT-IR) was used to detect the presence of mold release on various surfaces. FT-IR spectra were collected using a Nicolet Avatar 360 FT-IR spectrometer with an ATR sample cell. Spectra were collected at 32 scans over a range of 400-4000 cm^{-1} .

SEM images were acquired using a FEI Quanta 200 SEM operating at an accelerating voltage of 20 keV, a spot size of 4, and a working distance of ~ 10 mm. Each mold release was applied to a clean silicon wafer, holding the can approximately 6-8 inches above and coating the wafer with three passes. The mold release was allowed to dry on the wafer for 10 minutes. The coated wafer was then sputtered coated with gold ($\sim$ nms thick layer) prior to electron microscopy.

Cure Inhibition Study of Sylgard 184

MS-122E, MS-122SE, MS-122AD, MS-122ADL, and MS-122XD were tested for cure inhibition of Sylgard 184 (90/10) according to RM6K0435 Section 3.2 (states 72 hours cure at room temperature, in direct contact with substrate, following cure the contacted surfaces of the cured silicone are examined for wetness, stickiness, or softness, any of which indicates cure inhibition). Three plate aluminum button molds were fabricated. These button molds produced right circular cylinders nominally 1" in diameter and 0.5" tall. A photograph of the button mold is shown in Figure 1.

Figure 1: Left to right, the top, middle, and bottom plates of the aluminum button molds

Five molds were fabricated and a single mold release was dedicated to a specific mold release to avoid cross contamination. Three buttons were produced. Mold release was administered to all three plates of the button mold, holding the can roughly 6-8 inches above the mold. The middle plate was treated to cover the walls of each button mold. The mold release was allowed to dry for two minutes, Sylgard 184 (90/10) was applied and allowed to cure for 72 hours at room temperature. The Sylgard 184 (90/10) buttons were then observed for signs of cure inhibition. These signs include wetness, stickiness, softness, each button was compared to a that was in contact with the treated metal surface will be inspected for signs of cure inhibition (wetness, stickiness, softness) as compared to cured Sylgard 184 that was not contacted by the mold release as a control. Only mold release MS-122XD showed signs of cure inhibition small sections of all three buttons treated with MS-122XD had small patches of stickiness. These results are summarized in Table 6. Table 6 also summarizes the way the buttons were removed from each mold providing a relative comparison of the efficacy of each mold release.

Mold Release	Cure Inhibition	Method for Button Removal
MS-122E	No	Released by tool, hardest to release
MS-122SE	No	Released by tool, not as hard as E to release
MS-122AD	No	Released by hand and tool
MS-122ADL	No	Released by hand
MS-122XD	Yes	Released by hand

Table 6: Summary of mold release cure inhibition of Sylgard 184 (90/10) results.

Shear Stability of Mold Release on Metal Surfaces

Mold releases were tested for stability on uranium alloy surfaces, in an assembly that mimics a relevant engineering process. The uranium alloy surface was treated with a mold release. The Sylgard 184 (90/10) was vacuum-potted via the pumping action of a small rough pump. Five shear stability apparatus were engineered and fabricated to avoid cross contamination. The apparatus is pictured in Figure 2.

Figure 2: A photograph of the shear stability apparatus (left, unassembled; right, assembled) photograph does show indentation where uranium alloy coupon is seated,

A polycarbonate (Lexan) cover having two aluminum barb fittings was secured to a bottom aluminum plate with standard nuts, washers, and bolts. A circular metal shim was used to define the gap between top and bottom plates. The bottom plate had a small indentation to hold the uranium alloy coupon level to the aluminum surface. The barb fittings were fitted with silicone Tygon tubing to vacuum pot the Sylgard 184 (90/10). Sylgard 184 (90/10) was mixed and degassed according to the above outlined procedure. The top and bottom plates were treated with mold release and allowed to dry for two minutes. Then, one of the silicone tubes was immersed in the Sylgard 184 (90/10); the other silicone tube was connected to the vacuum pump. The pump was started and the Sylgard flowed through the tube and across the uranium alloy surface. Once the tube connected to the pump began to fill with Sylgard 184 (90/10) the pump was turned off. The Sylgard 184 (90/10) was allowed to cure for 24 hours. Following cure, surfaces were analyzed for the presence of mold release via IR spectroscopy. PTFE (the active ingredient in the mold release) has two distinct peaks in the IR region ($\sim 1210\text{ cm}^{-1}$ and 1150 cm^{-1}). The collected spectra were compared to Sylgard 184 (90/10) and uranium alloy specimens not treated with mold release. These results are summarized in Table 7. FT-IR results are shown in figures 3 and 4. All Sylgard 184 (90/10) seemed to release from each uranium alloy surface treated with each mold release relatively similarly, suggesting a similar release efficacy for uranium alloy surfaces.

Mold Release	PTFE Detected on Sylgard 184 (90/10) Surface	PTFE Detected on Uranium Alloy Surface
MS-122E	Yes	Yes
MS-122SE	Yes	Yes
MS-122AD	Yes	Yes
MS-122ADL	Yes	Yes
MS-122XD	Yes	Yes

Table 7: Summary of PTFE detection on surfaces in shear stability study

Figure 3: FT-IR Spectra of Sylgard 184 (90/10) surfaces showing presence of PTFE, as compared to untreated Sylgard 184 (90/10)

Figure 4: FT-IR Spectra of uranium alloy surfaces showing presence of PTFE, as compared to untreated uranium alloy

Electron Microscopy to Compare Mold Release Microstructure

Secondary Electron Microscopy (SEM) was completed on silicon wafer surfaces treated with MS-122E, MS-122SE, MS-122AD, MS-122ADL, and MS-122XD. The images for each mold release are presented in Figures 5-9. It can be seen that all the mold releases studied are relatively similar in microstructure; each yielding a rough film with micron sized particles.

Figure 5: SEM image of MS-122E on a silicon substrate

Figure 6: SEM image of MS-122SE on a silicon substrate.

Figure 7: SEM image of MS-122ADL on a silicon substrate.

Figure 8: SEM image of MS-122AD on a silicon substrate

Figure 9: SEM image of MS-122XD on a silicon substrate

Conclusions

Miller Stephenson PTFE-based mold releases were tested for various aspects of compatibility with Sylgard 184 90/10. The mold releases tested were MS-122E, MS-122SE, MS-122AD, MS-122ADL, and MS-122XD. The mold releases were evaluated for cure inhibition of Sylgard 184 90/10, sheer stability of mold release under flow of Sylgard 184 90/10, and a comparison of the morphology on the microscale. Only MS-122XD showed signs of cure inhibition of Sylgard 184 90/10. All mold releases were stable on both the surface of the mold release and then uranium alloy surface for all mold releases evaluated. All mold releases evaluated have similar micro structures (rough nodular structure on the ~ 1-micron scale) when sprayed on a silicon surface.

APPENDIX: Statement of Work proposed

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Phone: 505-665-8778
Date: April 20th, 2018

SUBJECT: STATEMENT OF WORK FOR MS-122 MOLD RELEASE STUDIES

Both tasks of this Statement of Work will involve the analysis of Miller Stephenson Mold Releases MS-122E, MS-122SE, MS-122AD, MS-122ADL, and MS-122XD. This work will supplement SEM observations and further establish similarities and differences between the mold release formulations above, which in turn will guide revisions to the current mold release specification.

Cure Inhibition Study of Sylgard 184

This task will test each of the five aforementioned MS mold releases for cure inhibition of Sylgard 184 (90/10) according to RM6K0435 Section 3.2. Aluminum button molds will be fabricated, then treated with mold release, the mold release will be allowed to dry for two minutes, Sylgard 184 will be applied and allowed to cure for 72 hours at room temperature. The side of the Sylgard 184 that was in contact with the treated metal surface will be inspected for signs of cure inhibition (wetness, stickiness, softness) as compared to cured Sylgard 184 that was not contacted by the mold release.

Sheer Stability of Mold Release on Metal Surfaces

Mold release will be tested for stability on uranium alloy surfaces, in an assembly that mimics a relevant engineering process. These surfaces will be treated with mold release then interfaced to an aluminum surface with a relevant small gap defined between the two substrates. Sylgard 184 (90/10) will be drawn through the gap via vacuum pressure. The Sylgard will be allowed to cure for 24 hours. Following cure, surfaces will be analyzed for the presence of mold release via IR spectroscopy. Release adequacy will be qualitatively described for each mold release.

Task	Description	
1	Cure Inhibition Study of Sylgard 184	15
2	Sheer Stability of Mold Release on Metal Surfaces	15
3	Prepare Report	2
Total Budget K		32

Risks: Delays in obtaining Sylgard 184. Instrument downtime. Staffing availability.