Langley Research Center - Soluble Imide (LaRC-SI)

by

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Abstract

This report is about my LARSS experiences at NASA Langley Research Center. My experiences entailed experimenting and developing uses for the new thermal plastic developed by Dr. Robert Bryant called the "Langley Research Center - Soluble Imide" (LaRC-SI). The three developments that I worked on are the use of the LaRC-SI as a dielectric for thin film sensors, as an adhesive to place diamonds on surfaces to increase thermal conductivity, and as an intermediate layer to allow the placement of metal on aluminum nitride.

Introduction

The LaRC-SI was developed by Dr. Robert G. Bryant, a chemical engineer at NASA Langley Research Center. This new multi-purpose, self-bonding thermoplastic material has won the R&D 100 award for being one of the 100 most significant new technical products of 1994. The R&D 100 award is presented annually by *Research and Development* magazine to those who have contributed the most significant products for advancing science and technology during the year.

The unique properties of this material is that it is an amorphous thermoplastic. This means that it can be reformed at elevated temperature and pressures. It can be applied in the form of a spray, spin, dip coating, paint, or spread with a doctors blade. The LaRC-SI has excellent adhesive and dielectric properties. It can also be recycled. Potential applications for this material are resin for mechanical parts such as gears, bearings and valves, advanced composites like carbon fiber, high strength adhesives, thin film circuits, and as a dielectric film for placing electrical components on conductive materials.

My stay as a LARSS student at Langley Research Center was involved in helping develop applications for the LaRC-SI. There were three areas of concentration. The first was the application of using the LaRC-SI as a dielectric to place thin film sensors on models for use in wind tunnels. The second area of concentration was to use the LaRC-SI to apply diamonds to a surface in order to transmit heat energy away from the heat source. The last experiment was the use of LaRC-SI as an intermediate layer to allow metal to be placed on aluminum nitride.

Thin Film Sensors

Thin film sensors are used on models to measure air speed while in a wind tunnel. This measurement is used to find out when laminar air flow changes to turbulent air flow on a model. The sensor consist of a thin conductive film with a constant current passing though it. With a flow of air across it, the thin conductive film cools and the resistance goes down. This results in a smaller voltage across the film. When the air flow is reduced, the thin conductive film heats up and the resistance goes up resulting in a larger voltage drop The voltage drop is therefore an indication of the air speed

The best way to put thin film sensors on a model is to deposit the metal sensors directly onto the model The idea is to make the sensors as thin and smooth as possible so that it is not intrusive to the flow of air In order to deposit metal on the model, a layer of dielectric material needs to be placed on the model to prevent the thin film sensors from shorting out on the model. The current material used for a dielectric is a polyimide made by DuPont. It works but it causes some problems because it cures at 500°F (Solder melts at ≈450°F and some model materials melt at < 500°F) My project consisted of determining if the LaRC-SI could be used as a dielectric for thin film sensors and then finding the lowest curing temperature.

Most of the time spent on this project was consumed by polishing small aluminum coupon plates by hand. There is definitely an art to polishing aluminum so that it has small uniform scratches that all go the same way Also a large number of coupons were polished because every failed attempt of fabricating the thin film sensors resulted in re-polishing the coupon. The reason for the coupon surface needing to be so smooth and flat was due to the small size of the sensors

The next problem was spraying the LaRC-SI in a humid environment. The LaRC-SI was dissolved in a solvent called NMP (1-Methyl-2-pyrrolidinone). In a humid environment, the NMP would react with the moisture in the air and would glob up resulting in clogging the air gun nozzle or depositing on the coupon. This was undesirable since a smooth even layer of LaRC-SI was needed on the coupon Different ratios of LaRC-SI and NMP were tried Also experiments of mixing NMP and Xylene was tried. The recipe that resulted was to mix the LaRC-SI and NMP in a 1:9 ratio by weight. This solution was mixed until all of the LaRC-SI was dissolved This mixture was diluted by mixing 2:1 solution and NMP by volume. Finally, 10 drops of Xylene was added for every 10 mL of solution This mixture was also sprayed early in the morning when the air conditioner was not tasked beyond its ability to minimize the moisture in the air

Another problem was the forced air convection ovens in which the coupons were placed to cure the LaRC-SI The forced air caused two problems First it would make ripples in the LaRC-SI as it was drying due to the air currents. Second, it would circulate dust around in the oven which would eventually deposit on the LaRC-SI as it was drying The first attempt was to cover the coupons with aluminum shelves to minimize the air flow on the coupons. This solved the ripple problem, but dust was still being deposited on the coupons Next, large petri dishes were used in which the coupon could be placed in the dish. The first attempt did not work because the petri dish cover did not allow enough air flow to allow the NMP to escape as it was evaporating. After some thought, cardboard spacers were added to the petri dish to raise the cover up to allow the NMP to escape, but not to far up to allow a large amount of air flow (and thus dust) into the dish This modification worked when the baking temperature and time were raised from 125°F for 30 minutes to 140°F for 45 minutes between each layer of LaRC-SI applied

In my final batch of coupons, the LaRC-SI mixture was prepared as described above. Four layers of LaRC-SI were applied to four polished coupons with each layer consisting of two coats each. Each layer was dried at 140°F for 45 minutes in a large petri dish. After the four layers were applied, each coupon was baked for two hours at 150°F, 200°F, 300°F, and 400°F respectfully. The coupons were placed in an E-Beam evaporator. 3,000 Angstroms of nickel and then 10,000 Angstroms of copper were evaporated onto the coupons while simultaneously using the ion beam gun. The thin film sensors were then patterned using standard photolithography procedures. The sensors where checked for quality control with a Reichert microscope and an ohmmeter. Hard wire leads were then soldered onto the copper sensor leads. This allowed the sensors to be hooked up to an anemometer. The anemometer stresses the sensor electrically. The results from the anemometer test are shown in tables 1 through 4.

Coupon cured at 400°F.

Sensor	R_G (Ω)	R_T (Ω)	R_S (Ω)	R_L (Ω)	R_{TA} (Ω)	Set (%)	R_{TA}(Set) (Ω)	VA (V)	R_{TA}(After) (Ω)	R_D (Ω)	Comments
1	>20M	11.8	8.5	3.3	10.48	120	11.82	?	14.50	4.1	
					14.50	130	16.74	569			blown open
2	>20M	12.2	9.9	2.3	10.97	120	12.70	.63			blown open
3	>20M	11.6	9.9	1.7	11.75	120	13.76	9.3	11.75	0.00	
					11.75	130	14.77	10.9	11.75	0.00	
					11 75	140	15.77	12 1	11.70	0 05	
					11.70	150	16.70	13.0	11.64	0.06	
					11.64	160	17.60	13.7	11.63	0.01	
4	5.6M	11.8	9.8	2.0	11.81	120	13.77	11.8	11.81	0.00	
					11.81	130	14.75	13.7	11.85	0.04	
					11.85	140	15 79	15.3	11.86	0.01	
					11.86	150	16.79	15 6	11.97	0.11	
					11.97	160	17.90	8 1	12.01	0.04	
5	>20M	11.1	9.9	1.2	10.95	120	12.90	9.6	10.95	0.00	
					10.95	130	13.88	11.1	:0.96	0.01	
					10.96	140	14.86	12 4	10 94	0.02	
					10.94	150	15.81	12 4	10.89	0.05	
					10.89	160	16 70	14.2	10.84	0.05	
6	>20M	9.4	9.8	2 0	10.36	120	12.30	10 6	10.34	0.02	
					10.34	130	13.23	12.3	10.35	0.01	
					10.35	140	14.21	13.7	10 36	0.01	
					10.36	150	15.19	14.7	10 37	0.01	
					10.37	160	16.17	8.5	10.35	0.02	

Table 1

Coupon cured at 300°F

Sensor	R_G (Ω)	R_T (Ω)	R_S (Ω)	R_L (Ω)	R_{TA} (Ω)	Set (%)	R_{TA}(Set) (Ω)	VA (V)	R_{TA}(After) (Ω)	R_D (Ω)	Comments
1	>20M	11 2	8.6	2.6	11.02	120	12.70	9 3	11.03	0.01	
					11.03	130	13.56	10 8	11 04	0.01	
					~~11.04~~	140	14.42	12.0	11 01	0.03	
					11 01	150	15.22	13.0	11 01	0.01	
					11 01	160	16.06	13 9	12 98	1 97	Unstable

2											Not measured
3	>20M	10.8	9 0	1.8	10.62	120	12.38	9.2	10.63	0.01	
					10.63	130	13.56	10.8	10 63	0.00	
					10.63	140	14.16	12.0	10.61	0.02	
					10.61	150	15.02	13.0	10.56	0.05	
					10.56	160	15.82	13.9	10.48	0.08	
4	>20M	10.7	10 1	0.6	10.46	120	12.43	9.8	10 46	0.00	
					10.46	130	13.42	11.5	10.46	0.00	
					10.46	140	14.40	12 6	10.46	0.00	
					10.46	150	15.39	13.6	10.46	0.00	
					10.46	160	16.38	8.7	10.45	0.01	
5	>20M	11.1	9 9	1.2	11.01	120	12.97	8.3	11.01	0 00	
					11.01	130	13.95	9.7	11.02	0.01	
					11.02	140	14.95	10.8	11.02	0.00	
					11.02	150	15.93	11.7	11.07	0.05	
					11.07	160	16.99	12.6	11.13	0.06	
6	>20M	11.0	10.2	0.8	10.91	120	12.93	8.0	10.89	0.02	
					10.89	130	13.92	9.3	10.95	0.06	
					10.95	140	15.01	10.4	10.94	0.01	
					10.94	150	16.01	11.1	10.83	0.11	
					10.83	160	16.85				ope - 844Ω

Table 2

Coupon cured at 200°F

Sensor	R_G (Ω)	R_T (Ω)	R_S (Ω)	R_L (Ω)	R_{TA} (Ω)	Set (%)	R_{TA}(Set) (Ω)	VA (V)	R_{TA}(After) (Ω)	R_D (Ω)	Comments
1	>20M	11 7	9.5	2.2	11.50	120	13.36	7.6	11.51	0.01	
					11.51	130	14.30	8.9	11.52	0.01	
					11.52	140	15.25	9.8	11.51	0.01	
					11.51	150	16.17	10.6	11.48	0.03	
					11.48	160	17.05	11 4	11.48	0.00	
2	>20M	11.9	10.1	1 8	11 77	120	13.76	6.94	11 77	0.00	
					11.77	130	14.76	8 10	11 77	0.00	
					11.77	140	15.76	9.0	11.96	0.19	
					11.96	150	17 04	9.8	17.18	5.22	Unstable
3	>20M	11.7	10 1	1.6	11.71	120	13.73	6 4	11.71	0.00	
					11.71	130	14.74	7.5	11.71	0.00	
					11 71	140	15.75	8.2	11.94	0.23	
					11.94	150	17.11	9.0	16.46	4.52	Unstable
4	>20M	11.5	10 1	1 4	11 31	120	13.29	6.7	11.31	0.00	
					11.31	130	14.28	7.7	11.31	0.00	
					11.31	140	15.27	8.6	11.57	0.26	
					11.57	150	16.66	9.3	11.82	0 25	
					11 82	160	17 08	8 9	11 57	0 25	
5	>20M	11 8	10 8	1.0	11.59	120	13.71	5 4	11 66	0 07	
					11.66	130	14.86	6.24	11.63	0.03	
					11 63	140	15.88	3 7	12.28	0.65	
					12 28	150	17.92				blown-1kΩ
6	>20M	10 8	10 0	0 8	10.58	120	12 54	6.3	10.58	0 00	
					10 58	130	13.51	7 3	10.58	0.00	
					10.58	140	14.49	7.4	13.48	2.9	Unstable

Table 3

Coupon cured at 150°F

Sensor	R_G (Ω)	R_T (Ω)	R_S (Ω)	R_L (Ω)	R_{TA} (Ω)	Set (%)	R_{TA}(Set) (Ω)	VA (V)	R_{TA}(After) (Ω)	R_D (Ω)	Comments
1	>20M	12.1	9.7	2.4	11.92	120	13.82	7.7	13.26	1.34	
					13.26	130	16.52	4.3	14.15	0.89	
					14.15	140	18.85				open >20MΩ
2											not measured
3	>20M	11.9	10.1	1.8	11.96	120	13.99	5.6	13.75	1.79	
					13.75	130	17.34	9.7	26.7	12.9	Unstable
4	>20M	11.8	10.3	1.5	11.70	120	13.74	3.8	16.30	4.6	
5	>20M	11.1	10.0	1.1	10.96	120	12.93	7.2	12.20	1.24	
					12.20	130	15.53	3.80	12.61	0.41	
					12.61	140	17.21				open >20MΩ
6	>20M	10.1	9.3	0.8	9.94	120	11.77	6.6	10.96	1.02	
					10.96	130	14.01	8.7	13.20	2.24	Unstable

Table 4

Notes:

- R_G: Resistance from sensor to aluminum coupon
- R_T: Total resistance
- R_S: Sensor resistance
- R_{TA}: Total resistance as measured from anemometer
- Set: Rating at which anemometer is set to cause overheating in sensor
- VA: Voltage measurement from anemometer
- R_D: Change in resistance after overheating setting by anemometer

The 160% set on the anemometer is a severe test. Taking this into consideration, the coupons that where cured at 400°F and 300°F were successful. The coupon cured at 200°F had 4 of the 6 sensors go bad when tested at 150%. More tests can be conducted later to determine a more exact temperature at which the LaRC-SI can be cured. This should be in the range of 200°F to 300°F

Electrically, the LaRC-SI material has allowed the placement of thin film sensors on aluminum at curing temperatures as low as 300°F. Two other items need to be taken into consideration: one, how well the LaRC-SI adheres to the model, and two, the smoothness of the finish of the LaRC-SI

The LaRC-SI needs to adhere well to the model It should not flake off. In the 150°F coupon, the LaRC-SI flaked off in large chunks when using an instrument to scrape it off. Although not as severe, the LaRC-SI also flaked off on all of the other coupons in smaller pieces.

The surface of the LaRC-SI also needs to be smooth so that it does not disrupt the flow of air over the model All of the coupons tested had ripples due to the LaRC-SI shrinking when it was drying. The coupon baked at 150°F was the worst while the others had ripples that were not as severe

Thermal Properties

The idea for this project came from the Fall 1994 issue of *UAB Scientist* It stated that diamonds are the best known thermal conductors, but it is difficult to adhere to metals, ceramic, and plastics. The heat transfer capability of diamond is >1200 W/mK and recently it has been manufactured with a heat transfer capability of >2000W/mK. This is compared to 326W/mK of copper which is an electrical conductor - a problem in some applications. An improvement in thermal heat transfer and dissipation is needed in the electronics community. After being handed some diamond dust by Dr Robert Bryant, I wrote a proposal to investigate the heat transfer properties of diamond dust adhered to a surface with LaRC-SI. The experiment was to show that diamonds could be applied to a surface to transfer heat energy from a heat source to a heat sink

An obstacle to this experiment is that the LaRC-SI is a thermal insulator. Since it is used as an adhesive, it will exist as a thin boundary between the diamonds. This thin layer will minimize the undesirable thermal properties of the LaRC-SI. Also, the thermal heat transfer capability of diamonds is so large that if the number is reduced by a factor of five, the heat transfer capability will still be large

The experiment was conducted as follows. 600 Grit and 1200 Grit diamonds were placed on 2" x 2" 60 mil alumina ceramic plates by using LaRC-SI. Thermal measurements were taken after using the following methods of placing the diamonds on the ceramic plates

1) Sprayed LaRC-SI and then sprayed the 600 or 1200 grit diamonds Cured at 125°F for 30 minutes between layers. Placed three layers of LaRC-SI and diamonds onto the ceramic plates.
2) Followed the procedure in step 1 but also baked at 500°F for 2 hours after the third layer was placed on the ceramic plate.
3) Followed the procedure in step 1 but then baked at 500°F for 2 hours in a vacuum The surrounding air pressed the diamonds while the LaRC-SI was curing so that the diamonds are closer together.
4) Sprayed the LaRC-SI on the ceramic plate but then sprinkled the diamonds on. Used the baking methods in steps 1 and 3.
5) Mixed the 600 grit and 1200 grit diamonds together. Sprayed the LaRC-SI on the ceramic plate but then placed the diamonds on by using forced air to agitate the diamonds onto the ceramic plates. Used baking methods described in steps 1 and 3.
6) Mixed the LaRC and diamond mix together and painted the mixture onto the ceramic plates. Followed the curing methods as described in steps 1 and 3.

The thermal properties were measured by using the following setup. A 10Ω power resistor was mounted on the test plate using a silver paint (Dynalot 340) Three thermocouples were used to take measurements at various locations at 1 cm apart. Two versions of the placement of thermocouples were used They are shown in figure 1. Measurement were taken every 20 seconds for 20 minutes. The power resistor was operated at 5.0 Volts which resulted in a heat source of 2.5 watts. A box was placed over the plate to minimize the effects of thermal variations due to drafts.

The temperature differences between the thermocouples was used for comparing the rate of heat transfer. The results were compared to a plain alumina plate and to a alumina plate with three layers of LaRC-SI that was cured at 500°F

This method of measuring heat transfer is crude, but it does give an indication of heat transfer and can be later sent to VMI which has the proper equipment setup to measure the heat transfer coefficient.

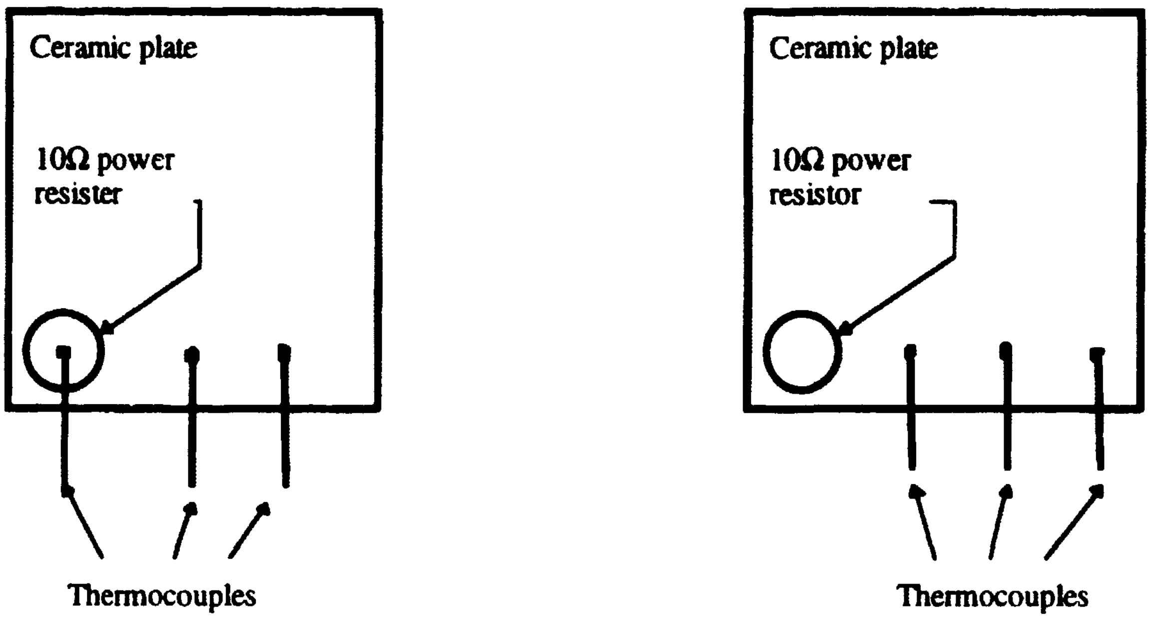

Figure 1. Thermocouple placement on ceramic plates.

Due to a limited amount of diamond dust and time, a more thorough experiment was not performed. Results were positive but not as good as expected The heat transfer increased when the sample was heated to 500°F and was the best when baked with a vacuum. A graphical result of the increase in heat transfer is shown in Figure 2. The best size diamonds to use was inconclusive. One of the problems was that too much diamond dust was deposited on some samples. The key for best heat transfer is going to be when there is just enough diamonds so that they are all touching with just enough LaRC-SI to pull them all together. Too much diamond dust and the LaRC-SI does not pull all the diamond dust together and leaves air pockets Too much LaRC-SI and the heat does not get transferred by the diamonds.

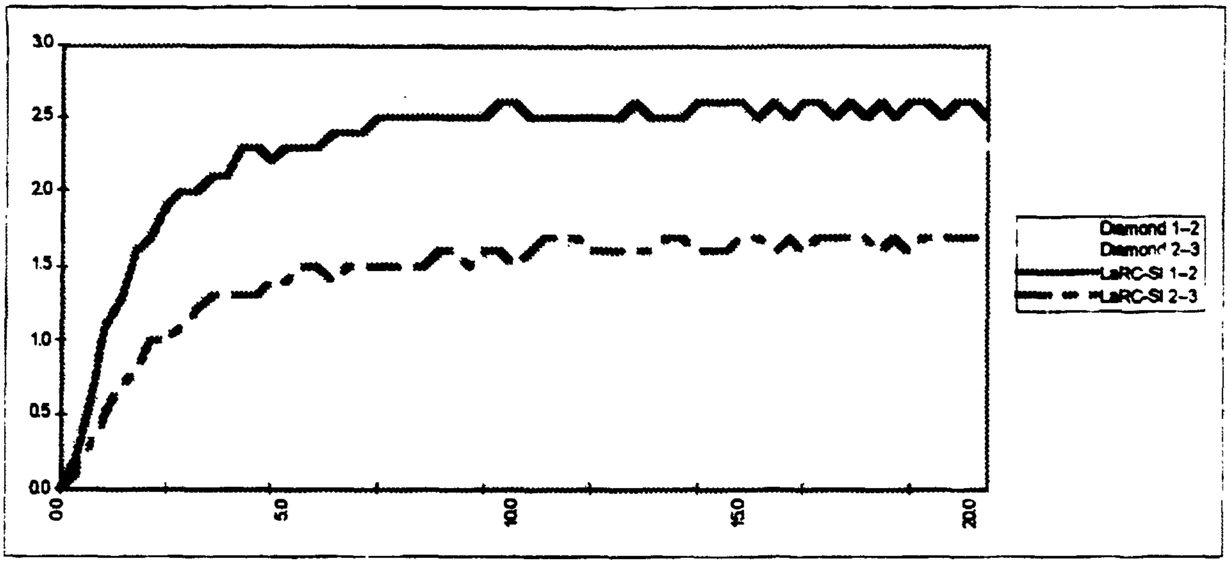

Figure 2. A Graphical Representation of Increased Heat Transfer

Applying Metal to Aluminum Nitride

Aluminum nitride is a ceramic that metal traditionally does not adhere. It is also a good heat conductor. Because of its thermal properties, it has been considered as a substrate for microelectronics. My experiment was to see if LaRC-SI could be used as an intermediate boundary to place metal on aluminum nitride.

Three layers of LaRC-SI was sprayed onto aluminum nitride plates. The mixture that was used was the same as used for the thin film sensors. Between each layer, the sample was dried at 125°F for 30 minutes. The ceramic plates were then baked at 500°F for two hours. 3,000 Angstroms of nickel and 10,000 Angstroms of copper were then evaporated onto the plates

As a check to see that the LaRC-SI really held the metal to the aluminum nitride, a tape pull test was performed. Also a more severe test was done by performing a tape pull test after the test plates were submerged in liquid nitrogen. Both test resulted positively. No metal was pulled off.

Conclusion

The LaRC-SI is an exciting new thermal plastic that has many potential applications. My stay at Langley Research Center involved exploring three of these applications. Due to the time limit of 10 weeks, all of the possibilities on these applications could not be explored.

This is also the first time that I have researched anything that is this complex and time consuming. Thanks to my Mentor, Carl Voglewede, he allowed me a great amount of freedom that I have not experience before in the workplace. I was allowed to make the decision on what equipment was needed and how an experiment was to be performed. This has definitely has been a learning experience.

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