RENEWABLE SOURCE MATERIALS PHASE II

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Abstract

In phase I, soy-based polyesters were introduced in the form of sheet molding compound (SMC) to be used in farm equipment such as combines. In phase II, soy-based polyester will be evaluated in the spray-up, infusion and resin transfer molding (RTM) processes for similar types of application. Each system was evaluated at room temperature and 120°F for surface quality, cure and molding ability. This paper will discuss shrink control for room temperature cured parts and surface quality, as compared to automotive standards. Physical property data will also be compared to standard polyesters and SMC used in these fields.

Introduction

With the success of the soy-based SMC products in the agriculture area, a study was undertaken to determine if the same base resin could be used in low temperature cured laminates. Would it be possible to maintain a Class A surface and build barcol hardness at temperatures below the 300°F used in SMC? The target temperature to mold at for this study was set from 77°F (25°C) to 140°F (60°C). Could tooling made from epoxy and/or polyester be used for Class A parts at lower temperatures? At the same time the study was started, the MACT standards were introduced and emissions became a major issue in the industry. Along with the hand lay-up work, RTM, RTM Light and infusion molding were evaluated in the study.

Development Study

The first step in the development project was to determine the lowest temperature at which the blend of soy polyester resin and low profile additive (LPA) would profile, build barcol hardness and what type of surface quality reading could be obtained. The study was set up to look at four different low profile systems and three different resin systems. The different combinations of resins and thermoplastics would hopefully give some insight as to what effect the soy resin product had on profiling and barcol hardness development. Table 1 contains some of the combinations and shows their effect on barcol hardness and surface quality. The initial study had the following results and trends:

A.) The soy base resin and one of the low profile additives was the only combination that would profile at 77°F and build barcol hardness.
B.) At 140°F, all the resin and LPA combinations would profile and build barcol hardness to some degree.
C.) At 100°F, the soy base resin and LPA profiled better and built higher barcol hardness.
D.) The LPA level needed to profile in all cases was between 15 and 25 parts.
E.) Normal inhibitor adjustments were needed to get all systems to a 20-minute gel time.
F.) The optimum temperature for profiling and barcol hardness is from 120-150°F.

The next step was to look at the soy base resin and thermoplastic blend in the RTM, RTM Light, hand lay-up and spray-up formulations.

RTM

The soy base resin and thermoplastic blend was adjusted into the standard liquid properties of a standard RTM resin that would be run as is and into one where filler would be used. A standard general purpose resin and Class A RTM system was chosen to compare the soy/LPA blend in the areas of physical properties, flow, stability, barcol hardness and surface quality.

The comparison of the three resin system was done on a 24-inch by 24-inch steel tool with surface quality of 40 using a commonly used surface analyzer. Laminates were prepared using three plies of a continuous strand mat, one 20-ounce veil and a standard 5-10 micron filler when used. All laminates were pumped through a standard positive displacement pump RTM machine. Inlet pressure to the RTM machine was kept constant for both the control and the soy resin system to collect the flow data for comparison. Water heaters connected to the steel tool were used to control temperature at 100°F, 130°F and 150°F. Table 2 shows the formulations, glass sequence and pertinent data needed in making the laminates. Table 3 has the physical property data, laminate shrinkage and surface quality data.

From the laboratory study, we were able to conclude the soy/LPA blend would produce Class A laminates at temperatures from 77-150°F. The surface quality and barcol hardness showed marked improvement as the molding temperature was increased up to 125°F and remained the same until 150°F. After post baking for 24 hours, the laminates showed little improvement in barcol hardness and no reduction in surface quality.

Field trials were then run at selected customers to confirm the laboratory results. Five-gallon samples were sent out to three customers and trials were set up on production parts. The parts were produced by the customer and Ashland's laboratory and then compared to the production standard. In all the cases, the parts had adequate barcol hardness and an improved surface quality.

RTM Light

The same soy-based resin/LPA combination was used to evaluate RTM Light and only slight viscosity modifications were needed. The biggest change needed was reducing the filler level needed in the RTM Light formulations. Filler levels for RTM were 40%, but the level was lowered to 0-20% maximum in RTM Light. The lower filler level does affect the surface quality, but it does not affect the barcol build-up. The material will still profile without filler, but there is some waviness in the panel. At filler levels of 20%, the waviness and surface quality are improved.

Hand Lay-Up

The same soy/LPA blend used in RTM and RTM Light was used for the hand lay-up development work. The LPA level was adjusted to meet the surface quality requirements, along with the performance specifications of the process. A steel plate was used as the mold to duplicate the poorest curing conditions available which would still have a surface that could be checked for surface quality. If the resin system would profile and build barcol hardness on the cool steel surface, it would build barcol and profile on plastic tooling.

Laminates were prepared using the steel tool, fiberglass and veil. The steel tool was kept in a 75°F environment and mold released with a semi-permanent, high gloss mold release. A 10-mil veil was placed on the steel tool and resin was applied. Three plies of 1 ½ -ounce mat were added, one play at a time, wet out with resin and rolled out to eliminate air. The laminate was allowed to cure for four hours and until a barcol hardness of 35 was achieved. The panel was then removed from the mold and the surface quality was checked. The surface turned white from the profiling and the surface looked good. As expected, the same formulation gave the same results when it was laid-up on a plastic tool.

Field trials were held at two fabricators to determine if the laboratory results could be verified. The trial results were good as the lab results and even better than expected in one case. The use of filler in any amount will enhance the surface quality of the part over a filled system. When no filler was used, a surface quality number of 70 was obtained, which is within the specification set by one original equipment manufacturer (OEM).

Conclusion

Renewable source resins made from materials such as soy, can be used in RTM, RTM Light, hand lay-up and spray-up applications using normal equipment and processing techniques. Surface quality using the renewable source Class A resin can and in many cases surpass the surface quality of the current material used with less secondary finishing then used today. General purpose polyesters made from soy-based polyesters will perform as any general purpose resin. Table 3 illustrates the possible finishing differences in using a Class A type resin over a standard general purpose polyester resin. The table shows that SMC and low pressure, low temperature SMC require very little, if any, finishing. RTM and RTM Light decreased the finishing time after demold compared to the extensive finishing time required by general purpose resins. The soy Class A spray-up system presently meets specifications for one of the agricultural manufactures. The soy hand lay-up material has already been used on a vehicle displayed at the auto show in Detroit, Michigan. The resin was used for a tailgate and can be seen in figure 1. In the RTM area, several fabricators are evaluating the soy Class A resin for reduced finishing time vs. cost. Table 4 lists the typical physical properties of the different Class A soy resins. Table 5 includes the surface quality evaluation.

Tables & Figures

Table 1: Starting Soy Formulations

Materials	A	B	C	D	E	F
Soy Base Resin	80.00	70.00	60.00	50.00	60.00	50.00
Styrene	20.00	20.00	20.00	20.00	20.00	20.00
LPA 1	---	10.00	20.00	30.00	---	---
LPA2	---	---	---	---	20.00	30.00
Wetting Agent	0.5	0.5	0.5	0.5	0.5	0.5
Promoters	Yes	Yes	Yes	Yes	Yes	Yes
Inhibitor, ppm	300	300	300	300	300	300
Viscosity, cps	148	148	148	148	148	148
Barcol Hardness	50	30	25	15	0	0

Table 2: Formulations and Glass Sequence Used in Test Panels

Formulations	RTM/RTM Light	Infusion	Hand/Spray Lay-Up
Soy Base Resin	55.00	55.00	60.00
LPA 1	25.00	25.00	20.00
Styrene	20.00	20.00	20.00
Wetting agent	0.5	0.5	0.5
Promoters	Yes	Yes	Yes
Inhibitor, ppm	300	300	300
Viscosity, cps	150	150	400-500
Thixotropic Index	None	None	2.5-4.0
Gel Time, Minutes	3 to 30	3 to 30	15-30

Table 3: Effect of Class A Resin on Finishing Time

Molding Temperature	SMC	LPLT SMC	RTM/ RTM Light	Infusion	Hand/Spray Lay-Up
77°F	NA	NA	3X	5X*	4X
100°F	NA	NA	3X	3X*	3X
120°F	NA	NA	2X	NA	2X
140°F	NA	NA	1X	NA	2X
200°F	NA	X	X	NA	NA
300°F	X	X	X	NA	NA

Figure 1: Vehicle Model Recently Displayed at the Auto Show in Detroit, Michigan

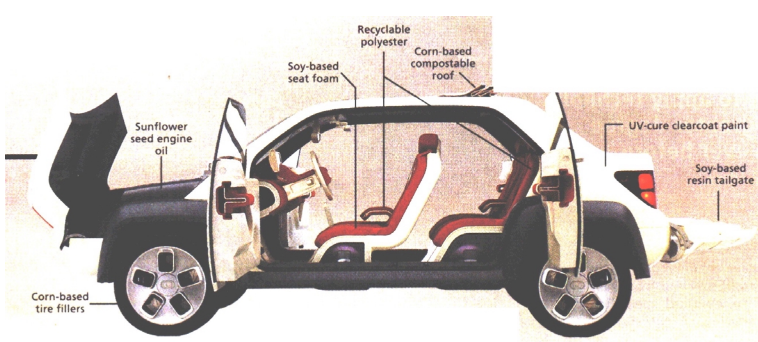

Table 4: Physical Property Comparison of Soy Resins

Test	SMC	LPLT SMC	RTM/ RTM Light	Infusion	Hand/Spray Lay-Up
Glass, %	29	28	27	46	32
Barcol Hardness	35-40	35	40	35	30
Tensile Strength, Mpa	102	91.6	91.63	110	106
Tensile Modulus, Mpa	10,789	10,239	7,315	8,652	7,280
Flexural Strength, Mpa	194	181	182	200	174
Flexural Modulus, Mpa	9,823	8,450	7,315	7,457	7,029
Impact - Notched, J/M	940	98	891	790	870
Impact – Unnotched, J/M	1,260		1,425	977	1,356

Table 5: Surface Quality Evaluation

Test for Surface Quality	SMC	LPLT SMC	RTM/ RTM Light	Infusion	Hand/Spray Lay-Up
LORIA[1] Number	40-60	50-70	50-70	125-150	90-110
OEM Standard	< 85	< 85	< 85	None	< 200

[1]Trademark of Diffracto Limited.

Figure 1 – Tensile Strength

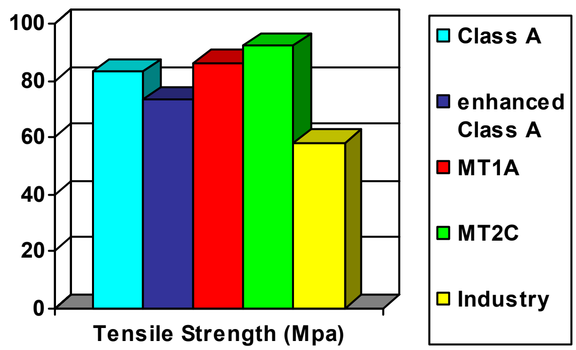

Figure 2 – Tensile Modulus

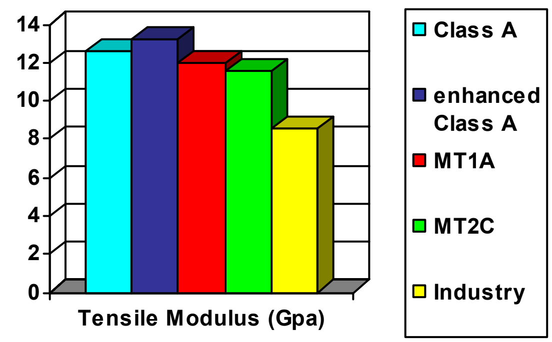

Figure 3 – Flex Strength

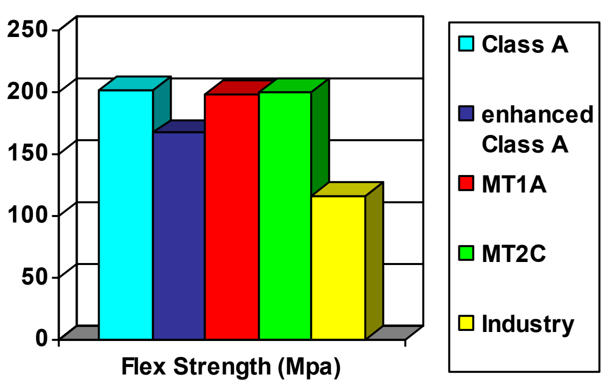

Figure 4 – Flex Modulus

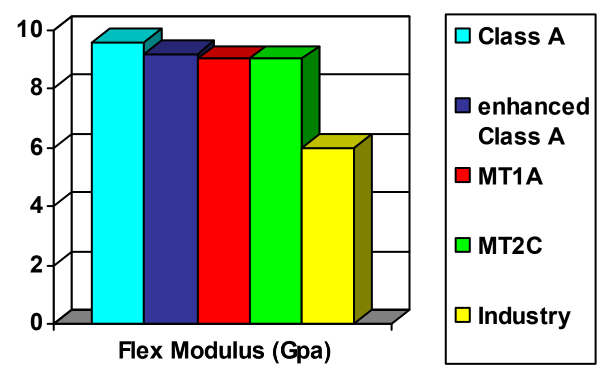

Figure 5 – Paint Pops

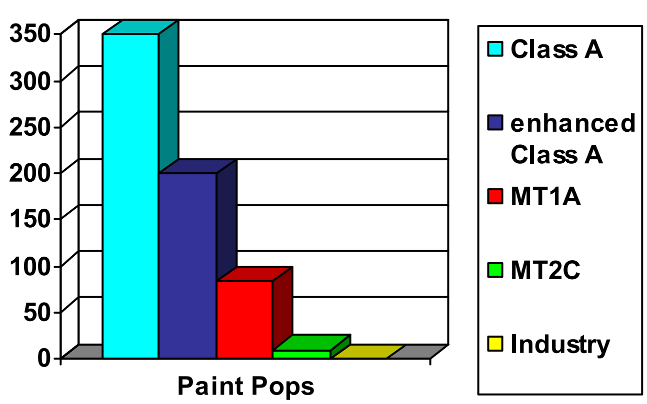

Figure 6 – Photograph of micro crack in enhanced Class A panel

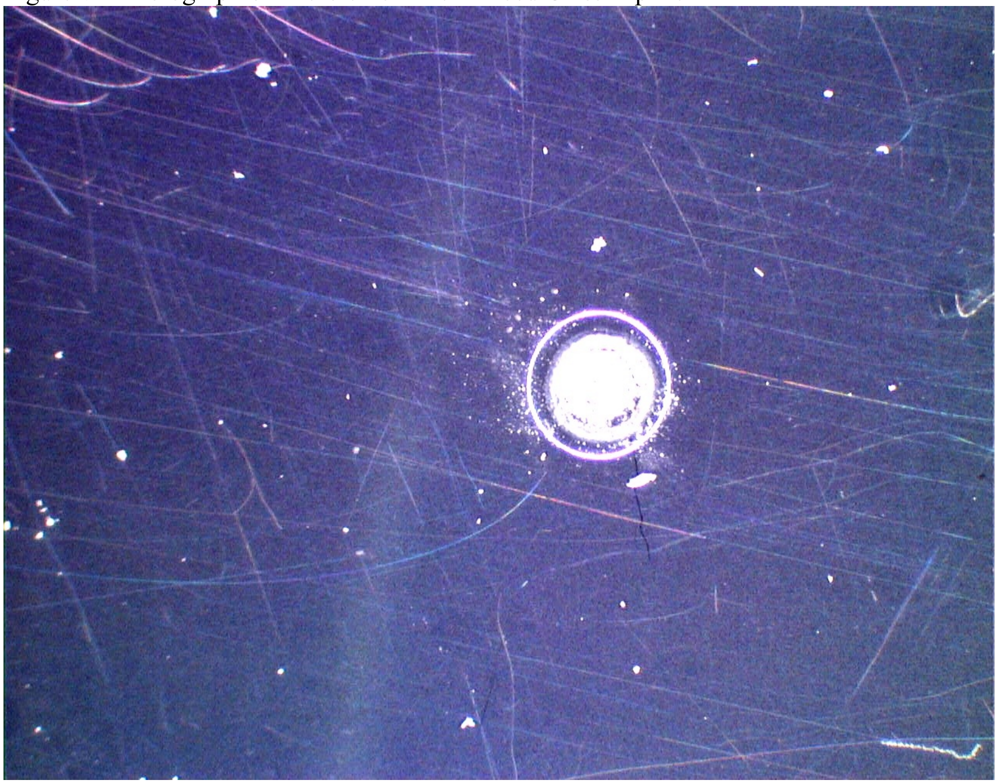

Figure 7 – Photograph of micro crack in MT1A panel

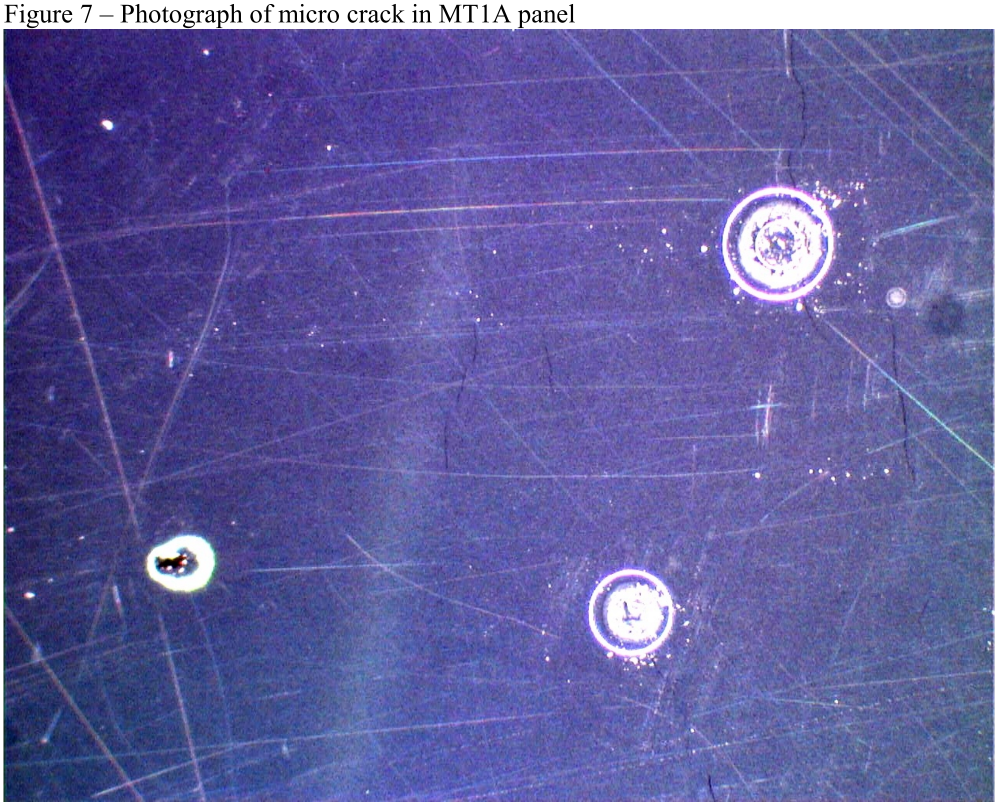

Figure 8 – Photograph of micro crack in MT2C panel

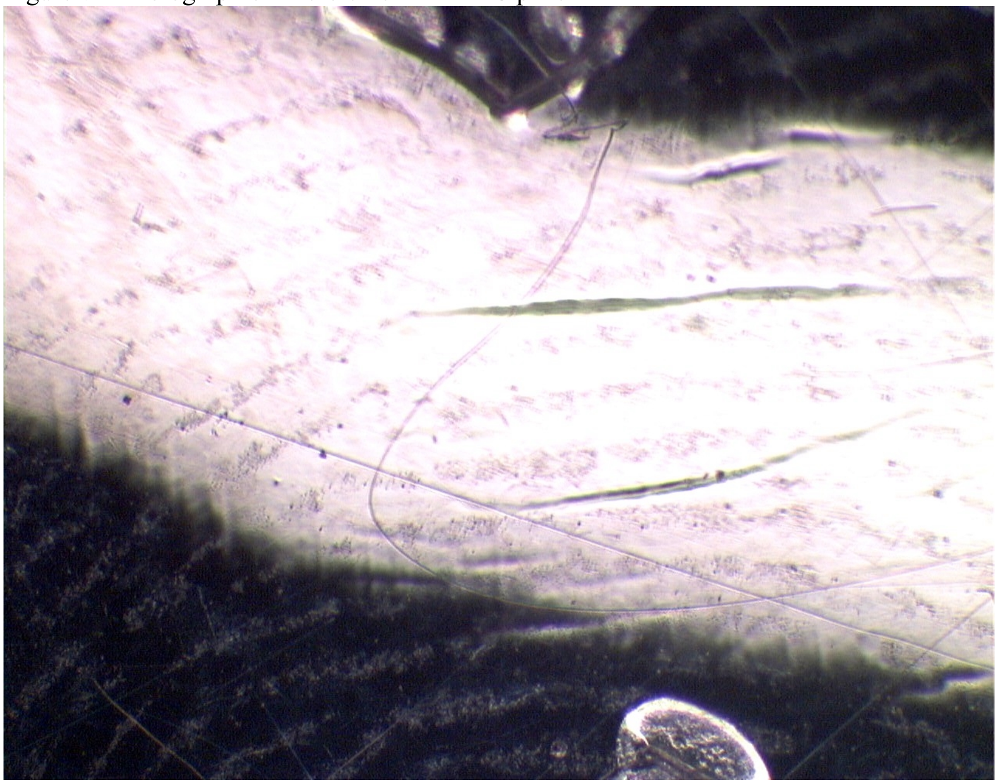

Figure 9 – SEM Micrograph of enhanced Class A panel (60x)

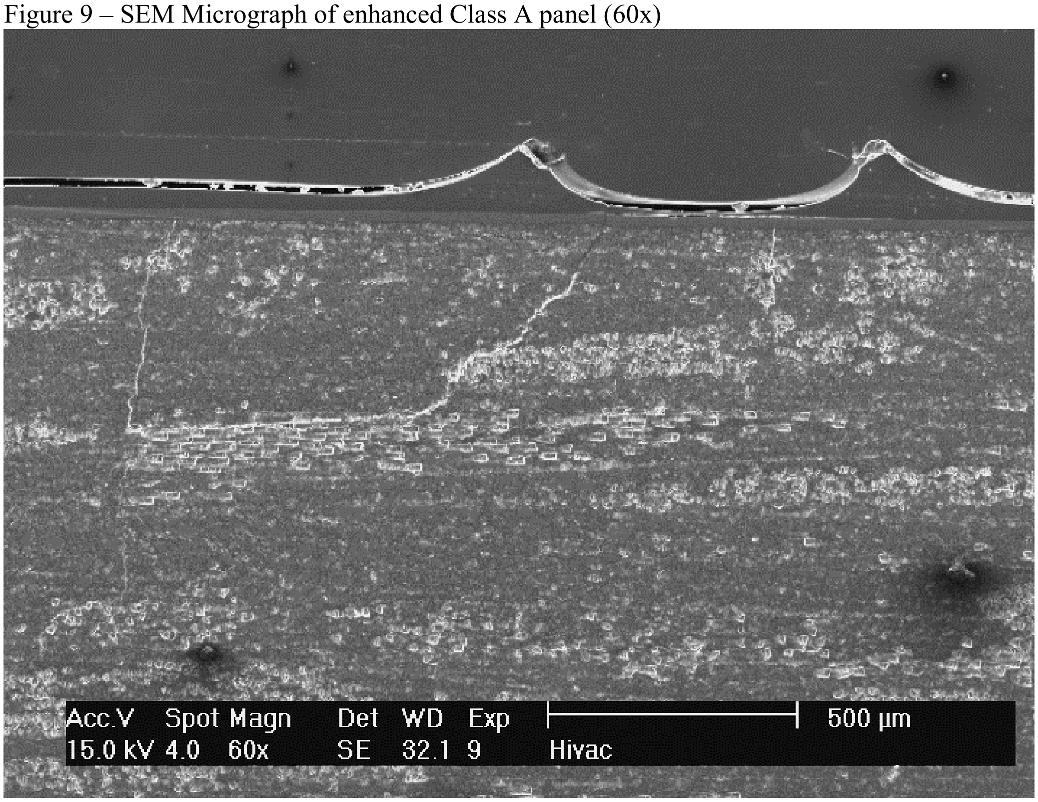

Figure 10 – SEM Micrograph of MT1A panel (60x)

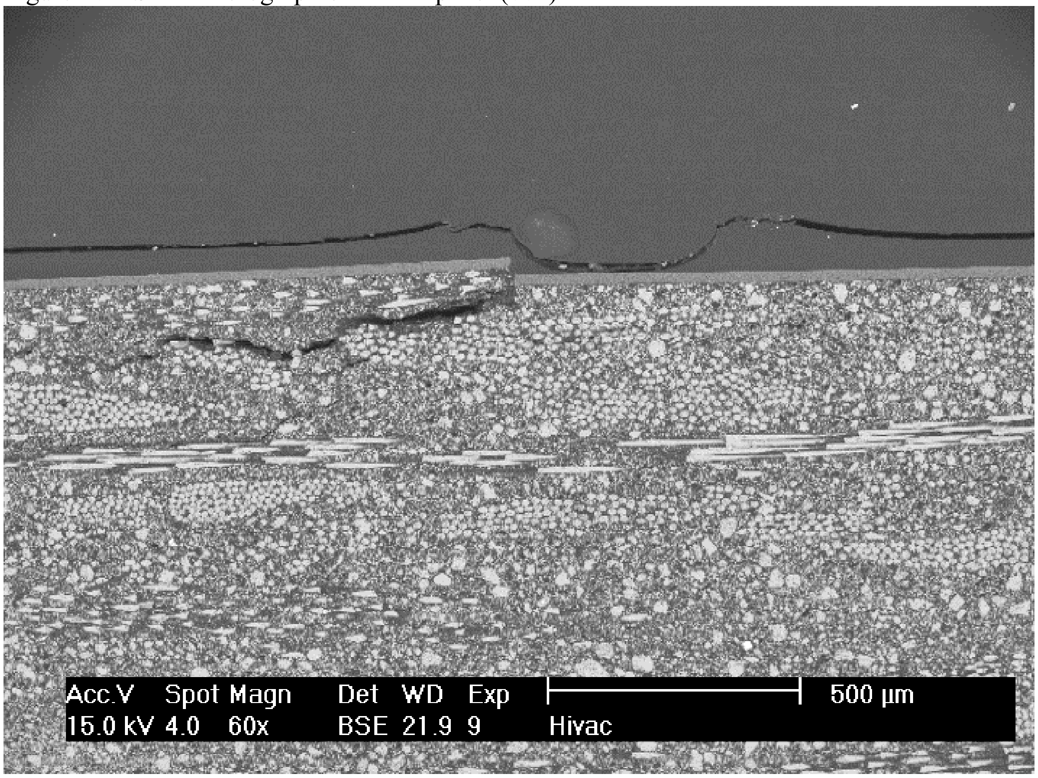

Figure 11 – SEM Micrograph of MT2C panel (60x)

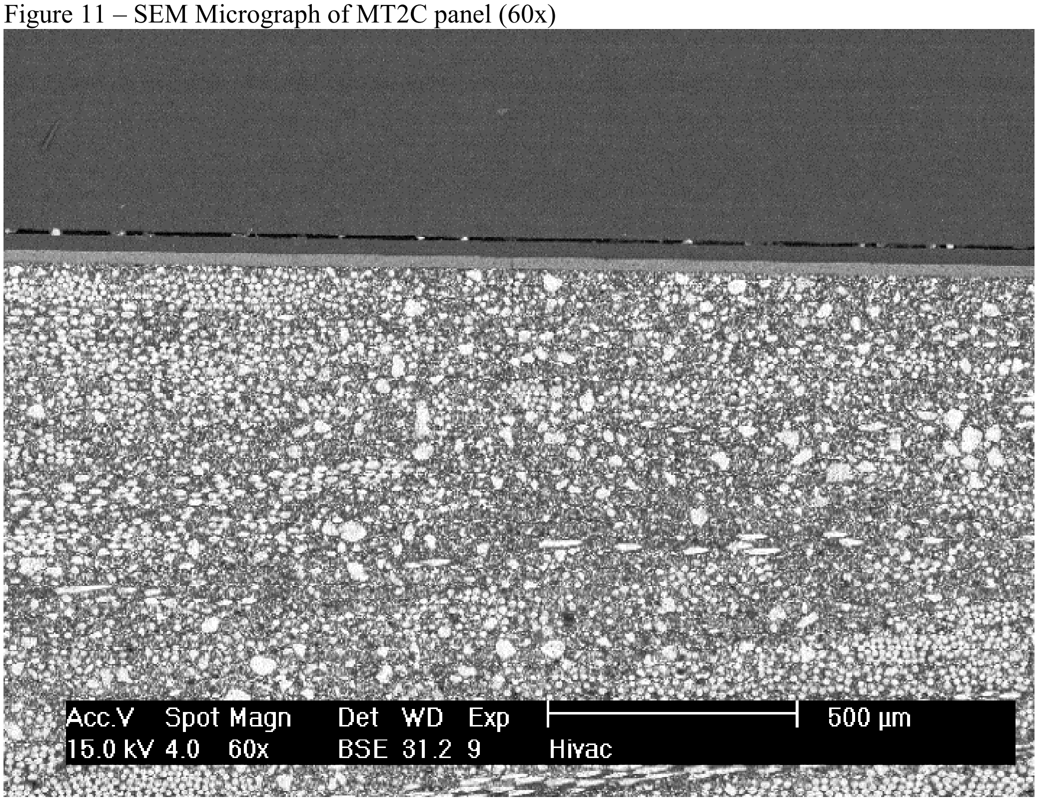

Figure 12 – Micrograph of MT2C panel (240x)

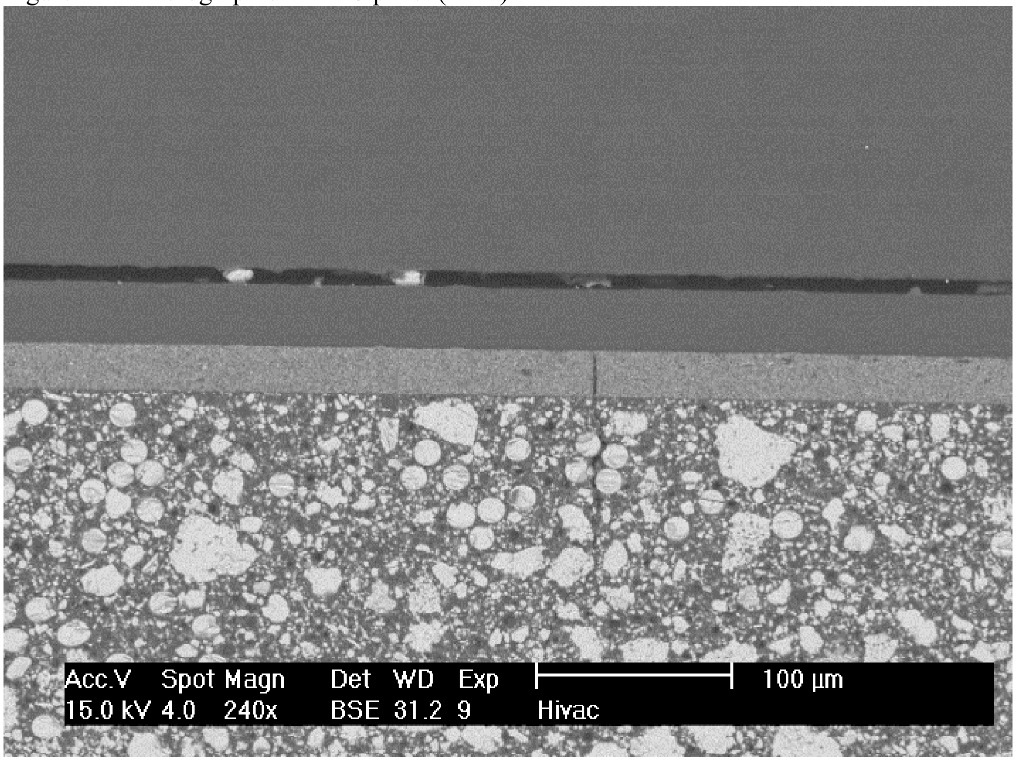

Figure 13 – Photo of paint pops – enhanced Class A top – Class A bottom

Figure 14 – Photo of paint pops – MT1A top – Class A bottom

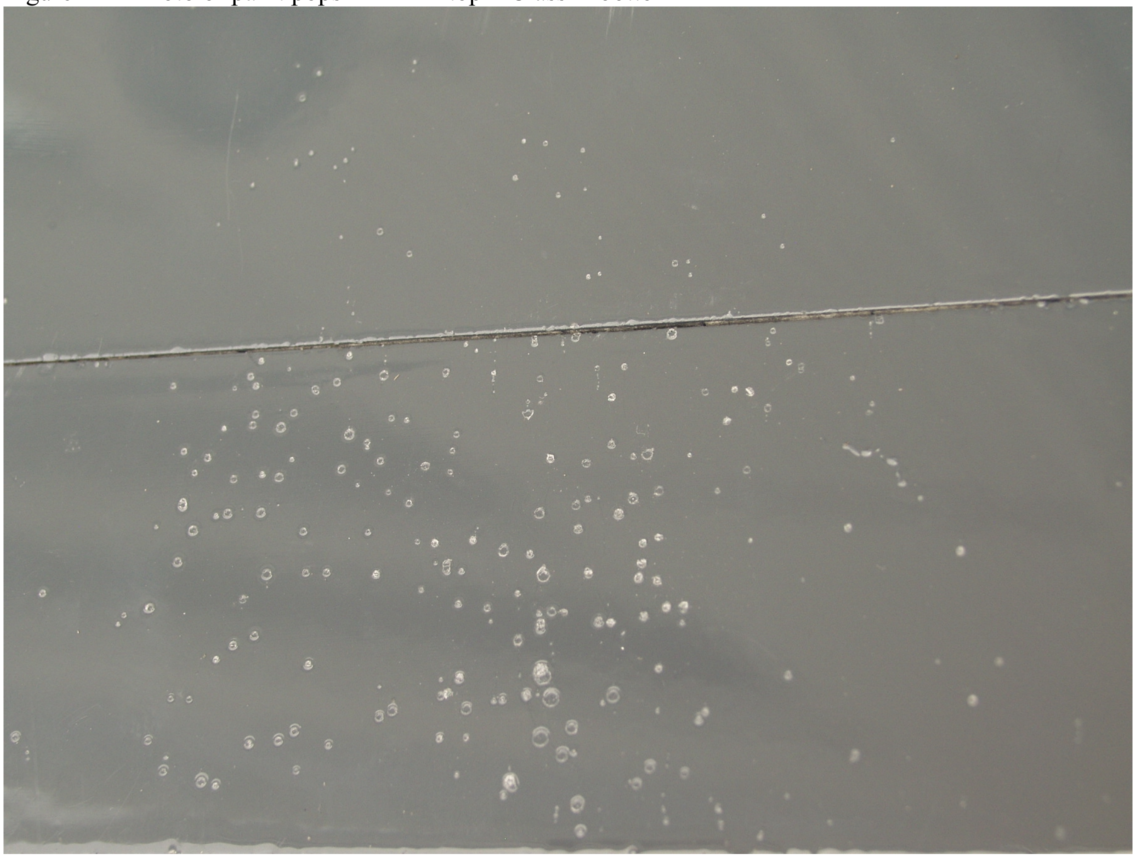

Figure 15 – Photo of paint pops – MT2C top – Class A bottom

Figure 16 – Photo of paint pops – MT2C top – MT1A bottom