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**Aluminum Tape Evaluation for Sealable Aluminum Tubes
Containing Mark 22 Fuel Tubes**

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Glossary of Terms

Acrylic resin:	polymers of acrylic or methacrylic esters, sometimes modified with nonacrylic monomers such as the acrylonitrile-butadiene-styrene (ABS) group.
Adduct:	a chemical addition product.
Adherend:	a body held to another body by an adhesive.
Block copolymer:	an essentially linear copolymer consisting of a small number of repeated segments of polymeric segments of different chemical structure.
Binder:	a component of an adhesive composition that is primarily responsible for the adhesive force.
Coagulation:	a separation or precipitation from a dispersed state of suspensoid particles resulting from their growth; may result from prolonged heating, addition of an electrolyte, or from a condensation reaction between solute and solvent; and example is the setting of a gel.
Colloid:	the phase of a colloidal system made up of particles having dimensions of 1-1000 nanometers and which is dispersed in a different phase.
Copolymer:	a long-chain molecule formed by the reaction of two or more dissimilar monomers.
Curing agent:	a catalytic or reactive agent that causes cross linking . Also called a <i>hardener</i> .
Degradation:	a deleterious change in the chemical structure, physical properties, or appearance of a plastic.
Diluent:	an ingredient added to an adhesive, usually to reduce the concentration of bonding materials.
Disproportionation:	termination by chain transfer between macroradicals to produce a saturated and an unsaturated polymer molecule.
Emulsion:	a two-phase liquid system in which small droplets of one liquid (the internal phase) are immiscible in, and are dispersed uniformly throughout, a second continuous liquid phase (the external phase).
Extender:	a substance, generally having some adhesive action, added to an adhesive to reduce the amount of primary binder required per unit area.
Filler:	a relatively nonadhesive substance added to an adhesive to improve its working properties, strength, or other qualities.

Glossary of Terms (continued)

Flocculent:	pertaining to a material that is cloud-like and noncrystalline.
Gel:	a semisolid system consisting of a network of solid aggregates in which liquid is held. In a cross-linked thermoplastic, gel is the fraction of polymeric material present in the network.
Hot-melt adhesive:	an adhesive that is applied in a molten state and forms a bond after cooling to a solid state. A bonding agent that achieves a solid state and resultant strength by cooling, in contrast to other adhesives, which achieve the solid state through evaporation of solvents or chemical cure.
Modifier:	any chemical inert ingredient added to an adhesive formulation to change its properties.
Phthalate esters:	the most widely used group of plasticizers, produced by the direct action of alcohol on phthalic anhydride.
Plasticizer:	a material incorporated in an adhesive to increase its flexibility, workability, or dispensability.
Rosin:	a resin obtained as a residue in the distillation of crude turpentine from the sap of the pine tree.
Sol:	a colloidal solution consisting of a suitable dispersion medium, which may be a gas, liquid, or solid, and the colloidal substance, the disperse phase, which is distributed throughout the dispersion medium.
Stabilizers:	chemicals used in plastics formulation to help maintain physical and chemical properties during processing and service life.
Tack:	the property of the adhesive that enables it to form a bond of measurable strength immediately after adhesive and adherend are brought into contact under low pressure.
Tackifier:	an agent applied to effect tack (adhesive force). Tackifiers provide tack and flexibility while promoting surface wetting and adhesion.

Aluminum Tape Evaluation for Sealable Aluminum Tubes Containing Mark 22 Fuel Tubes

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Summary

As part of the HEU Blend Down project, aluminum tape is required to seal aluminum tubes that will hold contaminated Mark 22 fuel tubes for dissolution. From a large field of candidate tapes, Avery Dennison's Fasson[®] 0802 tape (synthetic rubber adhesive system) was found to be acceptable for this application. This tape will disentangle in the normal H-Canyon dissolver solution and have no detrimental effect on the H-Canyon process. Upon placement of Fasson[®] 0802 tape into the dissolver solution, nitric acid will attack and disentangle the block copolymer network and destroy the adhesive nature of the material, resulting in insoluble particles that can be removed via centrifuge operations (cake weight increase of no more than 1%). The addition of the tape will not generate off-gas products and the resultant solution characteristics (surface tension, viscosity, density, and disengagement time) will be unaffected. Further, the potential effect on the down-stream evaporation system is negligible. Since the tape will not be placed in a high radiation environment, radiation stability is not an issue. Through detailed discussions with Avery Dennison chemists and based on analytical tests, a fairly detailed understanding of the constituents comprising the proprietary adhesive system has been assembled. Most importantly, chlorine was not detected in the aluminum tape (neutron activation analysis detection limit is 16 ppm). Finally, application of this tape will not impact LEU specifications.

Background

Aluminum tape is available with acrylic-based, rubber-based, and silicone-based adhesive systems. Table 1 lists general descriptions of these adhesive systems (listed in order of increasing temperature resistance). The actual compositions of manufacturer's adhesives are strictly held proprietary information. Indeed, tape manufacturers are quick to note that, as per OSHA Hazard Communication Standard, 29 CFR 1910.1200, effective 25 November 1985, their tape is defined as an "Article" (a manufactured item other than a fluid or particle). As such, material data safety sheets are not provided for aluminum tapes or their adhesives systems. The tested aluminum tapes possess a hot-melt adhesive (vs. solvent-based) that consists of 100% solids and is polymeric (natural rubber, synthetic rubber, or acrylic) in nature. However, these adhesives are rarely 100% polymers in composition. This is due to the limited adhesion of pure thermoplastics and their lack of molten properties, such as tack and wettability.¹ Table 2 illustrates typical components of hot-melt adhesives.

Table 1. Rubber-, acrylate-, and silicone-based adhesives²

Natural rubber	Natural rubber is cis-polyisoprene, which contains unsaturated double bonds susceptible to oxidative attack. Further, significant resins are required due to the poor tack and adhesion of natural rubber. Accordingly, it must be compounded with resins (e.g., wood rosin, terpene resins, and/or petroleum-based resins) and antioxidants (e.g., amines, phenolics, and dithiocarbamates).
Butyl rubber	Copolymer, composed mainly of isobutylene with a minor amount of isoprene.
Thermoplastic elastomers	Block copolymers {e.g., styrene-butadiene-styrene (SBS)} available as both solvent-coatable and hot-melt adhesives. (The SBS block copolymer is a two-phase system consisting of rigid, styrene-base end blocks and a rubbery mid segment.) Plasticizers are used to decrease the hardness, elastic modulus, and melt or solution viscosity. Other additives include fillers, pigments, and stabilizers.
Polyacrylates	Acrylic resins are colorless, thermoplastic, synthetic resins made by the polymerization of acrylic derivatives, chiefly the esters of acrylic acid and methyl acrylic acid, ethyl acrylate, and methyl acrylate. The resins can be soft, sticky semisolids to hard, brittle solids depending on the constitution of the monomers and the degree of polymerization. The lower molecular weight materials used in rubber-based adhesives are not present. Hence, migration of these materials to the surface to interfere with bonding does not occur. Since polyacrylates are saturated in structure, they are resistant to oxidative degradation.
Silicone	Silicones are more resistant to higher temperatures than those described above. Further, they are considered to possess excellent chemical and solvent resistance and flexibility. These adhesives are based on a gum and a resin. Gums are either methyl-based or phenol-modified silicones.

Table 2. Typical components of hot-melt adhesives

Additive	Purpose	Example(s)
Polymers	Provide strength and high viscosity	Polypropylene
Diluents	Promote surface wetting	Waxes
Plasticizers	Provide surface wetting and adhesion flexibility	Phthalates
Tackifiers	Provide tack and flexibility while promoting surface wetting and adhesion	Rosin, hydrocarbons, chlorocarbons, and carboxylic acids
Stabilizers	Help to maintain adhesive melt viscosity while functioning as antioxidants.	Phenols
Extenders	Lower cost and help control melt flow	Talc, clay, barites

Aluminum-foil sealant tapes feature dead soft aluminum (99.45% purity) foil with excellent adhesion and quick stick and are Underwriter Laboratories (UL) rated. The foil facestock, adhesive, and liner are typically 2.0, 2.0, and 3.5 mils thick, respectively. Tapes are typically available in widths of 2, 2.5, and 3 inches. Permissible operating temperature ranges and strength characteristics are shown in Table 3.

Table 3. Aluminum tape specifications

Manufacturer	Tape Identifier	Trade Name	Adhesive	Foil thickness (mil)	Temp. Range (°C)	US Oz. Force per in. width
Avery Dennison, Painsville, OH	0802	Fasson [®] 0802	Synthetic Rubber	2	< 107	126
	0810	Fasson [®] 0810	Acrylic	2	< 176	60
Shurtape Technologies, Hickory, NC	AF-75	Shurtape [®] AF-75	Acrylic	3	-34 to 163	55
	AF-100	Shurtape [®] AF-100	Acrylic	2	-29 to 121	60
	AF-973	Shurtape [®] AF-973	Natural rubber	2	-29 to 66	80
	SF-685	Shurtape [®] SF-685	Butyl rubber	2	-29 to 104	200+
Ideal Tape Co. Lowell, MA	488	488	Rubber	2	-12 to 82	90
	489	489	Acrylic	3	-29 to 121	75

Experimental

Dissolution tests

The aluminum tape samples described in Table 3 were tested with the objective of finding a tape that would dissolve or disentangle in the H-Canyon dissolver solution. The tape loading was about twenty times that of the dissolver solution after evaporation (see Table 4 for basis assumptions). Silicone adhesive systems were not tested due to their superior performance at elevated temperatures and in corrosive environments. The tests were conducted by placing the tape (approx. one cm²) into a 250-mL Erlenmeyer flask (containing 100 mL of 90°C, 4.4M nitric acid, and stir bar) for 20 hours. At the conclusion, the dissolution was characterized and the residue mass was determined.

Table 4. Estimated aluminum tape determination for one first-cycle batch

Number of dissolver batches	2
First cycle batch weight	48,000 to 52,000 pounds
Total fuel assemblies	32
Fuel assemblies with tape	24
Number of wraps / tube	2
Total number of wraps	48
Length of wrap	13.5 inches
Grand total length of tape	648 inches
Width of tape	2.5 inches
Total area of tape	1620 square inches
Mass of tape per sq. inch	0.00026 pound
Amount of tape (aluminum plus adhesive)	0.42 pound

To investigate the effect on plant material, coupon tests were conducted with the most promising tape (identified via initial screening tests) from each adhesive category. These tests were performed as follows. Approximately one cm² piece of aluminum tape was affixed to a 316 stainless steel coupon of the same area (0.15-mm coupon thickness) and placed in a 250 mL-Erlenmeyer flask (100 mL of 4.4M nitric acid, 103-108°C) for 20 hours. To support the semi-volatile organic compound (SVOC) analysis of the solid residue of the tape identified from the screening tests, a special dissolution test was conducted wherein the tape loading was 1700 times that expected in the dissolver solution. This special test also enabled a quantification of the mass ratio of aluminum to adhesive. Images of the residual material from these, as well as the other, tests were obtained with a digital Nikon SMZ 1500 microscope (up to 100X magnification).

Characterization of the dissolution solution and residue from Fasson[®] 0802 tape

Based on the screening tests, Fasson[®] 0802 was selected for further study. Absolute and kinematic viscosities were measured (22°C) with a Cannon-Fenske Routine Viscometer (size "50," Cannon Inst. Co.). The absolute viscosity (in centipoise) was obtained by multiplying the kinematic viscosity by the fluid specific gravity. Surface tension of the liquids was measured with the capillary tube (radius 0.40 mm) rise technique. A Fowler Sylvac precision micrometer was used to determine the actual rise. Disengagement time experiments were conducted with a Vortex test tube mixer (model K-500-2, Scientific Industries Inc.). Specifically, 5.00 milliliters of 7.5 vol. % TBP in *n*-paraffin were first added to a calibrated vortex tube. Next, 5.00 milliliters of the test solution were added. The vortex tube was mixed (medium setting) for 1 min. After the mixing period, the time for the two phases to separate was measured. Due to the subjective nature of this observation, multiple tests were performed on each sample. SVOC analyses of the liquid and solid residue were carried out on a Hewlett Packard 6890 gas chromatograph, equipped with a 30 m DB-5 column, with 0.25-mm diameter and 0.25- μ m film thickness. The solid residue was completely dissolved in methylene chloride prior to injection. Quantitation was performed using a Hewlett Packard 5973 mass selective detector. The mass spectrometer tuning was confirmed within 24 hours prior to each measurement using perfluorotributylamine. Acid molarity of the test solutions was confirmed via acid-base titration. Finally, chlorine content of the tape was determined via neutron activation analysis.

Results

Screening Study

Disintegration of the aluminum tapes generated a wide range of materials with different morphologies (see Table 5 and Figures 1-4). For all of the tapes tested, the aluminum dissolved readily (within two hours). None of the adhesives fully dissolved. The synthetic rubber adhesive system of Fasson[®] 0802 lost its tack and the resultant residue formed suspended solids possessing flocculation and sol characteristics. Minimal residue formed on the plastic stir bar (see Figure 1, plate 1 and Figure 2, plates 1 and 3) and no residue formed on the stainless steel coupon (see Figure 2, plate 2). The other rubber-based adhesives also lost their tack. The natural-rubber adhesive resulted in a coagulated mass that adhered to the stir bar (see Figure 1, plate 2 and Figure 3, plate 1).

The butyl-rubber adhesive resulted in a large coagulated/agglomerated mass that floated (see Figure 3, plate 2). In contrast, all of the acrylic-based adhesive residues formed a gel with tack retention (see Figure 4). Based on these findings, Fasson[®] 0802 was selected for further study.

Table 5. Results of screening study

Tape	Adhesive	Tack retention	Residue Color	Resultant residue ⁽¹⁾ (% of initial tape mass)			Residue Description
				Stir bar	Coupon	Soln. ⁽²⁾	
0802	Synthetic rubber	No	Yellow Orange	3	0	97	Flocculation/sol.
0810	Acrylic	Yes	White	54	N/A	46	Gel, tack retention.
AF-75	Acrylic	Yes	Clear	10	22	68	Gel, tack retention.
AF-100	Acrylic	Yes	Clear	79	N/A	21	Gel, tack retention.
AF-973	Natural rubber	No	Yellow Orange	70	8	22	Coagulation - material adhered to stir bar and did not breakup.
SF-685	Butyl rubber	No	Gray	4	0	96	Agglomerate - large (~1 cm) particles remained.
488	Rubber	No	Yellow Orange	22	N/A	78	Coagulation, some flocculation.
489	Acrylic	Yes	Clear	35	N/A	65	Gel, tack retention.

1.) N/A indicates that a coupon was not present in the test solution.

2.) By mass balance.

Detailed evaluation of Fasson[®] 0802

Based on the favorable disentanglement of Fasson[®] 0802, the tape, resultant solution, and solid residue were investigated in detail. Table 6 shows that the physical characteristics of the solution were not affected due to dissociation of the tape at a level about 20 times greater than actual process conditions. Specifically, the density, surface tension, kinematic viscosity, dynamic viscosity, and disengagement time were essentially within measurement error of 4.4M nitric acid. After dissolution operations in the plant dissolver, the nitric acid molarity will be about 0.7M. Accordingly, the effect of this shift was addressed by testing the solution after partial neutralization. As Table 6 shows, this adjustment had a negligible effect on the solution's physical properties. SVOC analysis of the solution (practical quantitation limit = 0.3 mg/L) showed only a trace amount of a plasticizer (0.21 mg/L dibutyl phthalate). {SVOC analyses detect analytes possessing boiling points between 145 to 500°C, including styrene (b.p. = 145°C)}. SVOC analysis results of the solid residue (practical quantitation limit = 0.3 mg/L) are shown in Table 7. Based on the special test with excessive tape loading, 27% of the initial tape mass was recovered as residue. Off-gas products were not observed. Neutron activation analysis of the tape showed the chlorine content to be less than the detection limit (16 ppm). Plutonium pick-up tests were not applicable since the fuel did not see reactor service. Radiation stability is not an issue due to the low level of exposure expected.

Figure 1. Resultant solutions from rubber-based adhesives

Plate 1. Synthetic rubber-based tape (Fasson[®] 0802) solution.

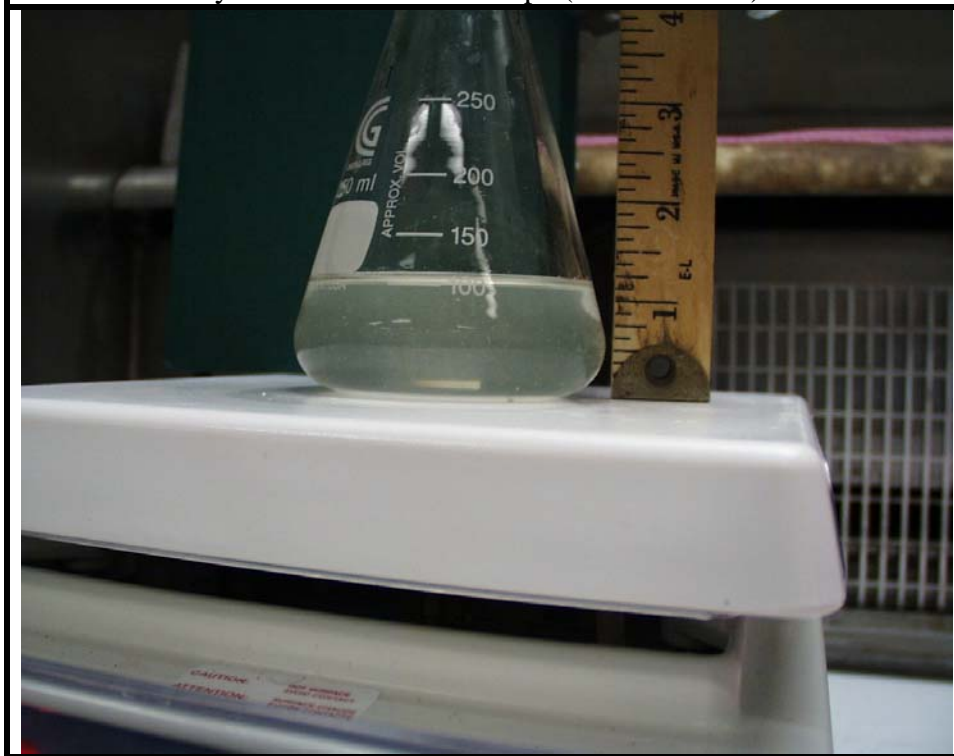


Plate 2. Natural rubber-based tape (AF-973) solution.



Figure 2. Synthetic rubber-based tape (Fasson[®] 0802) residue

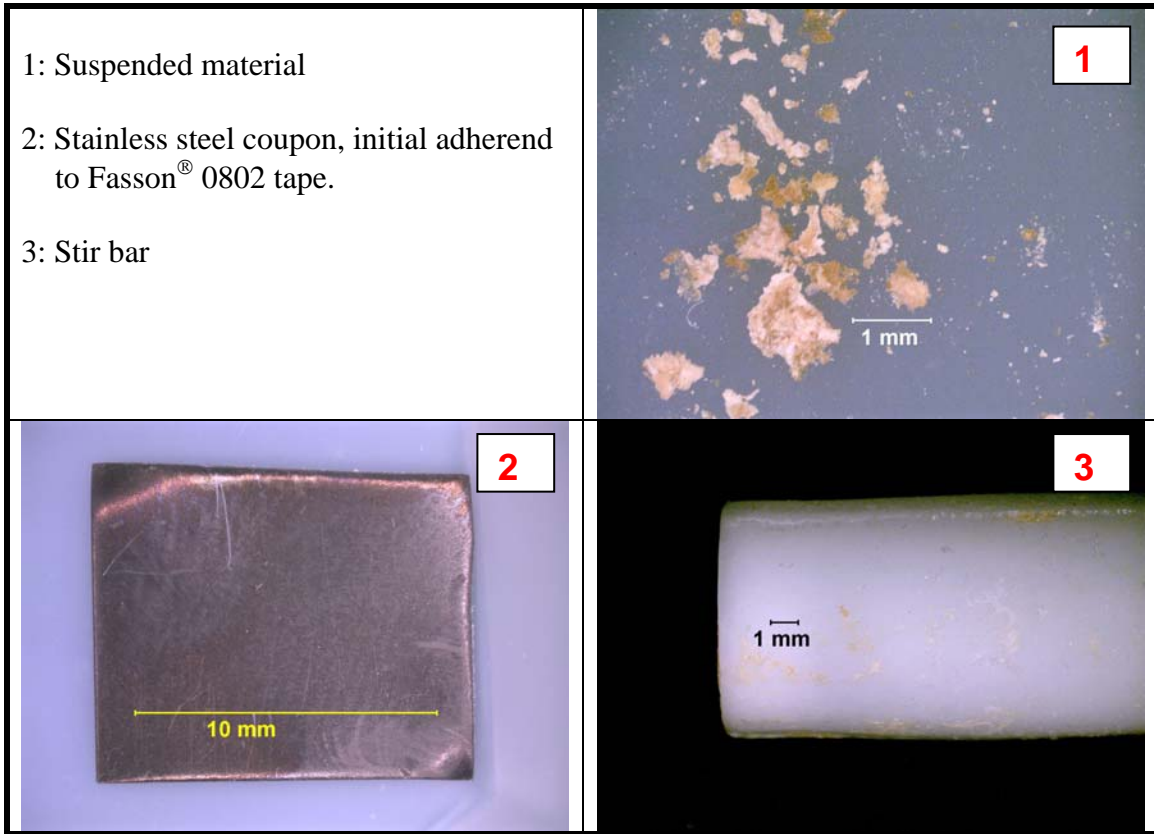


Figure 3. Natural rubber- and butyl rubber-adhesive based tape residue

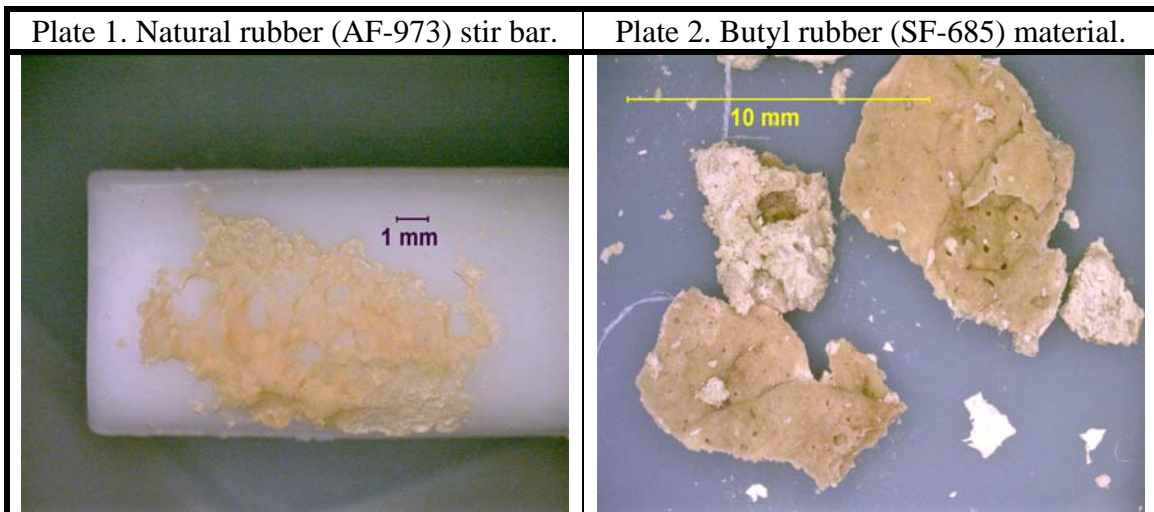


Figure 4. Acrylic-based adhesive tape residue

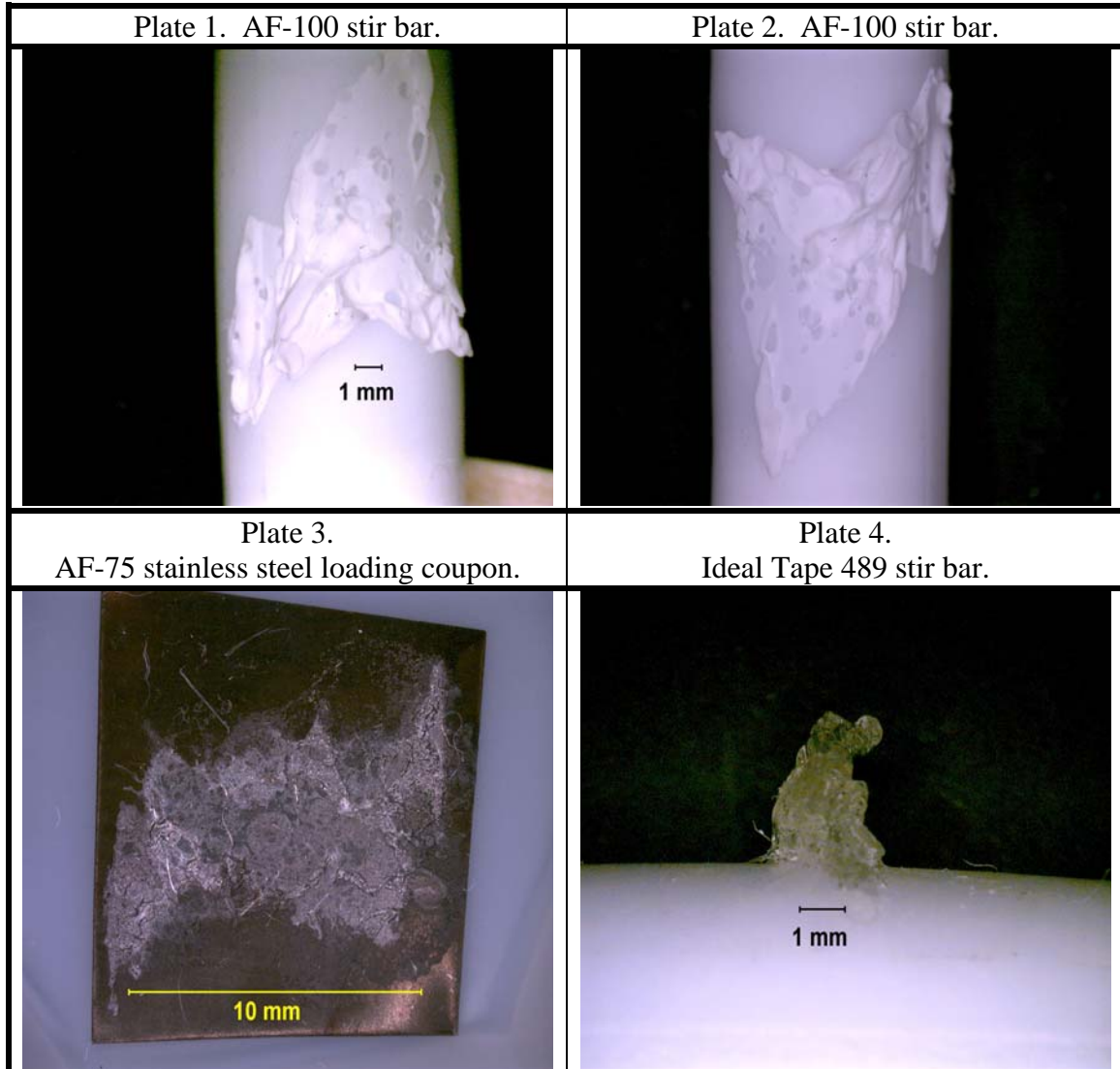


Table 6. Characterization of resultant liquid solution from Fasson[®] 0802 (22°C)

Parameter	Units	4.4M HNO ₃ (note 1)	Fasson [®] 0802 solution, <i>prior</i> to partial neutralization	Fasson [®] 0802 solution, <i>after</i> partial neutralization	Fasson [®] 0802 solution, <i>prior</i> to partial neutralization
Tape conc. ratio ⁽²⁾		N/A	22	24	1700
Liquid volume	mL	N/A	100.0	100.0	100.0
Tape mass	gram	N/A	0.0208	0.0236	1.626
Tape residue mass	gram	N/A	N/A	N/A	0.441
Mass/unit area	gram/cm ²	N/A	0.0182	0.0182	0.0182
Area of tape	cm ²	N/A	1.14	1.30	89.3
Density	gram/mL	1.15	1.15 ± 0.01	1.12 ± 0.01	1.19 ± 0.01
Surface tension	dyne/cm	69.8	70.7 ± 5.4	69.1 ± 3.4	67.7 ± 3.5
Kinematic viscosity	centistokes	1.07	1.07 ± 0.03	1.06 ± 0.03	N/A
Dynamic viscosity	centipoise	1.23	1.23 ± 0.03	1.19 ± 0.03	N/A
Disengagement time	seconds	29.9 ± 1.1	29.9 ± 1.4	29.3 ± 1.3	N/A

(1) Data (other than disengagement time) obtained from the CRC Handbook of Chemistry and Physics, 83rd edition, 2002-2003.

(2) This parameter is the ratio of the tape in the experimental solution to that in the plant dissolver.

Table 7. Characterization of resultant solid residue from Fasson[®] 0802

Analyte	mg/kg	Comment
Dodecanoic acid	230	tackifier
Unidentified phenanthroid structure	210	possible carboxylic acid and hence a tackifier
Undecanoic acid	140	tackifier
Alkene, substituted	96	adhesive component
Tributyl phosphate	94	plasticizer
Substituted 1-Phenanthrene carboxylic acid	92	tackifier
Substituted 1-Phenanthrene carboxylic acid	91	tackifier
Benzene, 1,3,5-trimethyl-2-(1-methylethenyl)-	79	polysubstituted and thus, probably not polymeric degradation products
Benzene, 1-methyl-4-(1-methylethenyl)-2-nitro-	79	
Furan, tetrahydro-3,4-dimethyl-, cis-	74	adhesive component
Diaromatic alkyl ketone	71	possible polymeric degradation product
Phthalic acid, substituted and nitrated	65	plasticizer

Discussion

Selection of tape

From the field of candidate tapes, Avery Dennison's Fasson[®] 0802 (synthetic rubber adhesive system) was found to be acceptable for the intended application. In no case did the adhesive residue completely dissolve; its disposition was the main concern. The natural rubber-based adhesive lost its tack; however, extensive cross-linking prevented sufficient disentanglement, resulting in large (> 1 cm) particles. On the other hand, the acrylic-based adhesives retained their tack, which would pose plant problems. In contrast, Fasson[®] 0802 lost its tack and exhibited sufficient disentanglement (yielding fine suspended particulate matter) for plant application.

Fasson[®] 0802 description

Through discussions with the manufacturer's senior chemist (Tom Apple) and based on laboratory analyses, a fairly detailed assessment was compiled on the constituents of Fasson[®] 0802. This tape possesses an adhesive system identified internally as "413." The main component of the hot-melt thermoplastic elastomer adhesive system is a block copolymer {styrene-butadiene-styrene (SBS)}. Plasticizers include dibutyl phthalate and tributyl phosphate (TBP). There are several tackifying resins; the manufacturer noted a rosin ester and one possessing phenolic functional groups. Stabilizers (e.g., hindered phenols) and extenders (e.g., clay, talc, and barites) are not present; however, some antioxidant agents are present. The manufacturer noted that chlorinated compounds are not applied in the manufacturing process.

Effect on H Canyon process and LEU specifications

Characterization of the resultant simulated dissolver solution, adhesive residue, and tape verified acceptable consequences of Fasson[®] 0802 application. First, solution characteristics (surface tension, viscosity, density, and disengagement time) were unaffected. Second, the particulate matter can easily be removed via centrifugal plant operations (estimated addition <1% of cake loading, based on a 20 pound loading and <50% of the initial aluminum tape forming an insoluble residue). Third, the effect on the evaporator will be insignificant. Specifically, the allowable concentration of organics in the evaporator feed is 0.5% by weight. According to Table 4, the organic material introduced via the tape will be less than 0.001% and essentially all of this will be removed in solid form prior to evaporator operations. Fourth, the absence of off gas products was expected; research with oxidation systems has shown that nitric acid (under the test conditions) is not significantly strong to induce significant, if any, oxidation of plastics or other organic materials.³ Further, this is consistent with previous research with the dissolution of nylon.⁴ Fifth, the absence of chlorine in the tape was confirmed by neutron activation analysis.

Under conditions of the H Canyon dissolving solution, the findings suggest that the high molecular weight (MW ~ 100,000s) SBS polymeric chains became disentangled but did not break down, resulting in insoluble particulate matter. The nitric acid most likely attacked the non-polymeric constituents of the adhesive system. Generation of

shorter (but still significant, MW ~ 10,000s) polymeric chains may have occurred; however, evidence for this is lacking and is not supported by SVOC analyses. Specifically, the resultant solution did not possess polymeric decomposition products (e.g., alkyl benzenes or polyphenyl olefins) - the only analyte identified was dibutyl phthalate (0.2 mg/L). Further, with the exception of a trace (71 mg/kg) quantity of a diaromatic alkyl ketone, polymeric decomposition products were not detected in the solid residue. The most significant analytes identified in the solid residue were carboxylic acids (dodecanoic and undecanoic acids, 230 and 140 mg/kg, respectively). Accordingly, based on disposition of the tape residue, negligible amount of organic material entering the solution, low level of impurities (iron and silicon) in the aluminum, and small quantity of tape expected to be applied, application of the tape will not impact the LEU specifications.⁵

The senior chemist at Avery Dennison confirmed the experimental findings and agreed that the selected tape was the most appropriate for the application. According to Tom Apple, the only liquid component will be a tackifying agent – a rosin ester that has a softening point of 10°C (much less than 1% of the adhesive system). He further noted that the remaining components would form very small particulate matter without tack that can be easily removed by centrifugation. This explanation was corroborated by the experimental findings. These findings are supported by the lack of dissociation/disentanglement observed with the natural rubber adhesive. In contrast to the SBS system in Fasson[®] 0802, natural rubber is heavily cross-linked, which provides significant resistance to nitric acid attack.

Recommendations

The aluminum tape recommended for application to the aluminum tubes is Avery Dennison “Fasson[®]0802.” This tape does not require any special preparation of the aluminum tube surface and possesses a suitable synthetic rubber-based adhesive system for the intended application. Upon placement in the head end dissolution system, the adhesive will quickly lose its adhesion characteristics and the constituents will break up into particles that can be removed via subsequent centrifuge operations. It is recommended that administrative controls be implemented to ensure the application of this tape only. It is suggested that periodic tests be performed to confirm the absence of chlorine in batches of delivered tape. Finally, it is suggested that the minimum acceptable tape width be applied.

Acknowledgement

The author acknowledges with gratitude the efforts of Dr. Stephen Crump of SRTC’s Analytical Development Section (ADS) for expert execution and evaluation of semi-volatile organic compound (SVOC) analyses of liquid and solid samples.

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⁴ Ibid.

⁵ Document Y-FDD-H-00001, Westinghouse Savannah River Company, HEU Blend Down Project, Facility Design Description, Appendix K, December 14, 2001, Revision 3.