Quick Doff Hood
(Problem Solving for Apparel Manufacturers)
91-33

For:

Clemson Apparel Research and The Defense Logistics Agency

Contract Number: DLA 900-87-0017 Order Number: 0025

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The U.S. Army currently seals its Quick Doof Hoods using a solvent-based liquid adhesive that is hand-painted onto the garment. The liquid adhesive takes several minutes to cure, releases solvent fumes, and produces inconsistent results. After evaluating a large number of commercially available adhesives, Clemson Apparel Research located an adhesive (Worthen E-9) that cures instantly, does not release harmful fumes, and can be consistently applied using conventional seam sealing technology. With associated hood design modifications, the adhesive can be used to improve performance and production.

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TABLE OF CONTENTS

List of Figures ..... Page vi

Introduction 1

Background 1

Problem 1

Need 2

Solution 2

Tasks List 4

Task 1 4

Task 2 4

Task 3 4

Butyl Rubber Research 5

Reason For Butyl Rubber Research 5

Welding Technologies Don't Work on Mil. Spec. Butyl Fabric 5

Common Fabric Adhesive Technology Does Not Work Either 5

Where Does One Go For Answers To Butyl Rubber Questions? 6

Why Are Powders Put On Cured Butyl Rubber? 6

One Reason - Microbial Resistance 6

Another Reason - Blocking Prevention 7

What Types Of Powders Are Available? 8

Mineral Powders - Mica and Talc 8
Organic Powders - Starches 9
Which Powder Does The Military Use And Why? 9
Are There Any Other Alternatives? 10
Proprietary Powders 10
Release Papers 10
Why Use Cured Butyl Rubber At All? 11
Why Uncured Butyl Rubber Cannot Be Used 13

Task 1A - Locating/Developing Adhesives for Sealing Butyl Rubber 14
Adhesives Characteristics Available - Adhesive Classifications 14
Function 14
Physical Form 15
Activation Requirements 15
Chemical Composition 16
Pressure Sensitives 17
Hot-melts 18
Adhesive Characteristics CAR Wanted 19
Why Commercial Butyl Rubber Adhesives Were Eliminated 20
How Commercial Butyl Rubber Adhesive Work 20
Adhesive Characteristics CAR Needed 21
Evaluation of Commercial Pressure Sensitives 22
3M Acrylic Pressure Sensitives 22
Bond Strength Versus Solvent Resistance 22
926 And 9500PC 24

Application Method 24
Test Results - 92/02/19 Test 5100 25
Mace Urethane Pressure Sensitives 26
SSI And SSII 26
SSIII 27
SSIV - Mace 2451 28
Initial Performance Evaluation 28
Experimental Hoods Made 29
Queen Light QHP-778L 31
Hood Design Change Recommendations 32
Seam Tape Optimization 33
Are Multiple Layers of SSIV Needed? 34
How Does Width Affect SSIV Seam Tapes?35
Mace 2451, Run XU256 (SSIV) 36
Production Hoods Made 36
Problems Encountered 37
Substandard Butyl Rubber 37
Was Adhesive The Problem? 38
Did Improperr Adhesive Application
Contribute To The Problem? 38
Evaluation of Commercial Hot-melts 40
Polyester And Polyamide Thermoplastics Ruled Out 40
Hot-melts - Starensier PuddleCoat™ 40
Application Method 41
Problems Encountered 41

Starensier's Answer And CAR 's Response 42
Return To Pressure Sensitives 43
Mace 2882, Run XU270 43
Application Method - Modified QHP 778L 43
Production Hoods Made 44
Recommendations 46
Test Results - SEM's - 92/11/XX 47
Mace 2451, Run XU??? (SSIV) 49
Alloyed Adhesives = Pressure Sensitive + Hot-melt 49
Worthen E-9 49
Application Methods 50
Test Results 51
Final Adhesive Solution 56

Task 1B - Survey Of The Commercial Findings Market 57
Buckles 57
Design Characteristics 58
Design Trade-offs 58
Unique Commercial Designs 59
Custom Buckle Design May Be Needed 63
Strapping 64
Other Findings 65

Task 2A - Main Quick Doff Hood Seam Construction 66
Sewn and Taped Seams 66

Ultrasonically Welded and Taped Seams 66

Task 2B - Quick Doff Hood Elastic Cord Case Construction 67

Task 2C - Quick Doff Hood Elastic Face Openning Construction 68

Task 3 - Final Quick Doff Hood Recommendations 69

Raw Mateial Recommendations 69

Butyl Rubber 69

Thread 69

Adhesives 70

General Characteristics 70

Specific Characteristics 70

Commercial Findings 71

Buckles 71

Strappping 71

Other Findings 72

Production Equipment Recommendations 72

Sewing Machines 72

Heat Sealing Machines 72

Process Recommendations 73

Sewing Related 73

Adhesive Related 73

Hood Design Change Recommendations 75

Notes 76

Appendixes 77

INTRODUCTION

Background

The U.S. Army currently utilizes butyl rubber coated nylon fabrics for a number of chemical protective garments. The garments are typically sewn using conventional needle and thread construction. The sewn seams must be sealed to prevent penetration of liquids and vapors through the needle holes. A toluene-based adhesive is hand-painted onto the garment to seal the holes.

Problem

This construction method suffers from a number of disadvantages. First, the process of forming the seams requires two operations: sewing and sealing. The double operations add cost to the manufacturing process. Second, there is no guarantee of uniformity in the sealing operation. Because the painting is performed by hand, there is no expectation that a good seal will exist along the entire seam. This situation means that extensive post fabrication testing must be performed to ensure that the seam does not contain pinhole defects. Third, the manufacturing working conditions tend to be marginal due to the presence of large quantities of solvent fumes in the workplace.

Need

The Army needs a new way to seam/seal butyl rubber garments like the Quick Doff Hood. The new method should be easier, more reliable, and solvent free.

Solution

As part of an ongoing contract with the US Army Natick RD&E Labs, Clemson Apparel Research (CAR) has been investigating stitchless fabrication techniques for the Army's Chemical Protective Overgarment. Most of the work has focused on finding and developing adhesive products that can be readily integrated into conventional seaming/sealing methods. A majority of the adhesives CAR has developed are in the form of adhesive-coated fabric-reinforced tapes. These heat-seal tapes can be loaded onto conventional seam sealing machines and used to seal sewn seams.

One of the early adhesives used by CAR was a very aggressive pressure sensitive adhesive developed for the commercial roofing industry. The requirements of the commercial roofing industry are strong, heat resistant, moisture resistant bonds between pieces of coated roofing fabric. CAR felt that such an adhesive might be used to seam and seal military, butyl rubber, coated, fabrics.

Initial experiments showed that the liquid roofing adhesive, which is normally painted onto large, straight, stationary pieces of fabric, did not lend itself to conventional apparel material handling techniques. Attempts

to integrate commercially available adhesive delivery technology with sewing technology proved unsuccessful. By pre-curing the roofing adhesive onto a base material and then slitting the material to form adhesive tapes (like Scotch Tape®) CAR was able to develop the first of a series of butyl rubber adhesive tapes for potential use in military butyl rubber seam-sealing applications.

The latest version of the butyl-rubber seam-sealing tape uses a modified version of the original roofing cement. The new adhesive softens and becomes sticky when exposed to high temperatures but is not sticky at room temperature. The new adhesive can be applied using conventional heat-sealing equipment and does not produce harmful fumes. All other adhesive systems require that the butyl be washed and preferably abraded. The new adhesive is aggressive enough to bond un-washed un-abraded butyl rubber and can be tailored to meet specific application requirements.

Readers should note that at the time this report was written, all of the industry processes, equipment, and opinions listed in this report were current and are therefore discussed in the present tense. Developments made during the course of the Quick Doff Hood Project are reported in the conventional manner, in the past tense.

TASK LIST

Task 1

Under DLA900-87-D-0017 the Quick Doff Hood Project was divided into three tasks. Task 1 involved locating and developing the appropriate adhesives for sealing the butyl rubber fabric. Preference was given to adhesives which maintained the cleanest working conditions. Task 1 also involved finding buckles, strapping, and other components which would improve the existing quick doff hood by making it easier use, less expensive, lighter in weight, and less bulky.

Task 2

Task 2 involved constructing Quick Doff Hoods using two different seam techniques. Some hoods were constructed using adhesive-sealed sewn-seams while the remainder of the hoods were to have ultrasonic seams. The two techniques were investigated in order to determine which process yielded stronger, more uniform seams. Task 2 also involved evaluating the use of adhesives to form the casing for the elastic cord at the neck and the elastic cord at the face opening.

Task 3

Task 3 involved recommending design modifications for the quick doff hood. These modifications were to be presented as revisions in the purchase description and patterns for the hood.

BUTYL RUBBER RESEARCH

Reason For Butyl Rubber Research

Welding Technologies Don't Work on Mil. Spec. Butyl Fabric

Of all the Quick Doff Hood Project tasks, the most difficult task involved locating and developing new sealing technologies for use on Quick Doff Hood butyl rubber fabric (often called butyl rubber, for short). What made sealing butyl rubber so difficult were the Military Specifications (Mil. Specs.) C51251 and 12189 which dictate how butyl rubber fabric is made, seamed, sealed, and used.

For example, the Mil. Specs. call for the butyl rubber to be made of a nylon scrim fabric coated on both sides with fully cured butyl rubber. Cured butyl rubber does not melt in the presence of heat, but nylon does. Materials that melt (thermoplastics, see p. 16) can be welded. Simple manual tests showed that, as specified, the Quick Doff Hood fabric does not contain enough thermoplastic material to be weldable. So, at the start of the project, the Mil. Specs. ruled out the use of ultrasonic welding for sealing the Quick Doff Hoods. To complicate matters, other tests showed that, as specified, the butyl rubber would be difficult to bond.

Common Fabric Adhesive Technology Does Not Work Either

CAR's failure to bond washed and unwashed butyl rubber using a polyester based fabric-adhesive, Pellon SP28, and a polyamide (nylon) based fabric-adhesive, from ElectroSeal, made CAR realize that to effectively bond butyl

rubber fabric would require a better understanding of butyl rubber. CAR began its butyl rubber investigation by trying to find out what kind of powder was being used on the butyl rubber and why. With this information CAR could determine how to remove, replace, or avoid the powder so that adhesives could have a better chance of bonding to butyl rubber surfaces.

Where Does One Go For Answers to Butyl Rubber Questions?

To learn more about the powder being used in military butyl rubber, CAR arranged a visit to Archer Rubber Company, one of the military's butyl rubber suppliers. A tour of the plant and conversations with Peter Franco, president of Archer Rubber Co., brought some interesting butyl information to light.

Mr. Franco and a colleague were on the committee responsible for writing the current military specifications concerning butyl rubber. The military specifications that pertain to the Quick-Doff Hood butyl coated nylon are Mil. Spec. 12189, which is a light weight butyl coated nylon specification, and Mil. Spec. C51251, which is a medium weight butyl fabric specification.

Why Are Powders Put On Cured Butyl Rubber?

One Reason - Microbial Resistance

At the time the specifications were written common military practice was to inventory large quantities of raw materials and finished goods in the event of sudden military mobilization. Since most warehouses do not have

controlled environments, and since the only naturally occurring environmental threats to butyl coated fabrics are extreme temperatures and long term biological exposure, the Mil. Specs. for butyl rubber call for anti-microbial agents to be added to the butyl materials.

Since biological attack of the butyl rubber is most likely to start on an exposed surface of the material, the best place to put anti-microbial agents is on the surface of the butyl rubber. By making the anti-microbial agent an anti-sticking agent as well, the butyl rubber manufacturers solved the problem of long-term storage and a manufacturing problem as well. To understand how the anti-microbial anti-sticking agent solved a common manufacturing problem requires a basic understanding of how butyl rubber fabrics are made.

Another Reason - Blocking Prevention

In preparing a butyl rubber "recipe" one starts with uncooked (uncured) butyl rubber. Just as cake batter is made with raw eggs, butyl rubber compounds are made with uncured butyl rubber. Since uncured butyl rubber polymers are inherently sticky it is often necessary to add anti-sticking (anti-blocking) ingredients to butyl rubber compounds. The anti-blocking ingredients, which are mixed into the butyl rubber, prevent the butyl rubber from sticking to the mixing, rolling, and coating equipment.

Once the butyl rubber compound is made, it is spread (coated) onto a base fabric. In the Military's butyl rubber the butyl compound is coated onto both

sides of a nylon base fabric. At this point the butyl rubber is still uncured and as such has a doughy consistency.

Although it is theoretically possible to coat and then fully cure the butyl fabric in a continuous operation few, if any, manufacturers have the money or space for back to back continuous processing equipment. Therefore, a majority of butyl rubber coating facilities must, at some point, store the butyl coated fabric in roll form between process operations. Since uncured butyl rubber can still stick to itself and since the butyl coated fabric must be rolled up before it is fully cured, it is necessary for manufacturers to coat the uncured (or partially cured) butyl fabric with an anti-blocking compound to prevent the butyl fabric from sticking to itself.

As mentioned earlier, the military specifications for the manufacture of butyl rubber call for anti-microbial agents to be added to the butyl materials. Since the butyl rubber also needs to be coated with an anti-blocking compound, the butyl rubber manufacturers decided to "kill two birds with one stone" by making the anti-blocking compound an anti-microbial compound as well.

What Types Of Powders Are Available?

Mineral Powders - Mica and Talc

Traditionally, two of the most readily available and least expensive anti-blocking compounds used by the rubber industry are mica powder and talcum powder. Mica powder is made by grinding up mica, an abundant

non-water soluble mineral. Mica powder is used in most of Archer's commercial applications. Talcum powder is made by grinding up talc, a soft, inexpensive, non-water soluble, mineral.

Organic Powders - Starches

A third type of powder is starch powder. Starches are commonly obtained from plants (i.e. corn starch, potato starch, etc.) and can be ground into inexpensive organic powders. From an adhesive perspective starch powders have the desirable characteristic that they can be readily removed from any fabric surface. A starch coated fabric washed in a warm, concentrated, water based, enzyme bath will have all the starch removed from its surface by the enzymes in approximately 15 minute. Unfortunately, the same process that allows enzymes to remove starch allows microbes to digest starch as a food. Microbes that eat starch produce waste products which can, with prolonged exposure, damage butyl rubber.

Which Powder Does The Military Use And Why?

Mica and talc, on the other hand, are basically inert and are not affected by microbial action. Talc's inert nature, low cost, and absorption characteristics (which allow talc to be treated with anti-microbial agents) make talc the preferred choice in Military butyl rubber applications. Unfortunately, the same properties which make both talc and mica inert also make them non-water soluble and therefore very difficult to wash off. Even after five detergent washes in a domestic washing machine, a square yard of the butyl material still produced white talc rings where talc filled

rinse water evaporated from the butyl fabric surface. Discussions with the Clemson University Textile Department revealed that although other methods may exist, a majority of butyl rubber users remove the talc from butyl rubber by washing the butyl in water and then wiping the butyl with petroleum based solvents.

Are There Any Other Anti-Blocking Alternatives?

Proprietary Powders

With a better understanding of why talcum powder is present on the surface of military butyl rubber, and knowing that starch powder could not be used as an alternative, CAR wondered whether any other anti-blocking compound might be used instead of powders. According to Peter Franco, president of Archer Rubber Co., several rubber companies were trying to develop proprietary powders with better wash characteristics than talcum powders and better anti-microbial characteristics than starch powders. Unfortunately, additional information was not available on these proprietary powders.

Release Papers

Knowing that the adhesive industry uses a wide variety of release papers in adhesive coating operations, CAR wondered whether the same release papers might not be used in butyl coating operations. CAR contacted Tom Carrig, an adhesive chemist for the Bemis Adhesive Company, and asked him for a sample of the release paper he had found to be the most effective as a non-stick surface. Tom sent CAR a roll of industrial grade paper

heavily impregnated with silicon. Silicone release papers, frequently used in the processing of film coatings, work in much the same way as wax papers. Silicones are generally more heat resistant than waxes, however, and since many polymers (including butyl rubber) are processed hot, the heat resistance of silicon papers is important.

CAR requested that Archer Rubber Company prepare a butyl fabric sample using Tom's silicon release paper instead of talcum powder. When the butyl samples were returned to CAR it was apparent that even though silicon is heat resistant, the temperatures used to cure the butyl rubber fabric were sufficiently high that some of the silicon liquefied and the uncured butyl rubber penetrated the paper fiber substrate of the silicon paper. When CAR tried to separate the silicon paper from the butyl fabric, portions of the silicon paper would not separate from the fabric, and the experiment was deemed a failure.

Why Use Cured Butyl Rubber At All?

Discouraged by a lack of options on how to process cured butyl rubber, CAR then asked if it was necessary to use cured butyl rubber at all. A conversation with Rodney Kreps of Life-Guard Systems, the world's largest manufacturer of hazardous material (HAZMAT) protective garments, found that Life-Guard Systems uses more uncured butyl fabrics than cured butyl fabrics. Life-Guard receives rolls of powdered, uncured, butyl rubber fabric on silicon release paper. The fabric is die cut, and seam areas are abraded to remove the surface powder. The butyl fabric pieces are then

seamed using pressure rollers. Once the fabric pieces are pre-assembled, thermal curing is used to cross-link the butyl rubber making permanent, high strength, leak proof seams. According to Adriel Harrod, a similar technique is used by Lion Apparel to make certain fireman's turn-out gear.

To gain a better understanding of how uncured butyl rubber and other forms of butyl rubber could be handled, CAR asked Archer Rubber for samples of powdered and un-powdered butyl rubber in both cured and uncured form. (CAR did not request partially cured butyl rubber because there is no way to produce repeatable samples except in a strictly controlled laboratory environment.) CAR was already familiar with the cured powdered butyl rubber. The cured un-powdered butyl rubber was manageable at first, but over time the roll of butyl fabric began to stick to itself (block) making unrolling difficult. According to Peter Franco the poor storage characteristics of cured un-powdered butyl rubber prevent it from being used in a majority of butyl rubber applications. The uncured un-powdered butyl rubber had such bad blocking problems that it even stuck to the silicon release paper it was delivered on. The uncured powdered butyl rubber (used extensively in the protective clothing industry) was quite manageable, however. The only assembly drawback with the powdered uncured butyl fabric was that all the seams had to be abraded to expose the sticky uncured butyl.

Why Uncured Butyl Rubber Cannot Be Used

Based on the initial evaluations discussed above, CAR felt that the uncured powdered butyl rubber had potential for use in the Quick Doff Hood. Discussions with Peter Franco, who assisted in writing the military butyl rubber specifications, showed CAR's optimism to unfounded. According to Peter, and verified by Gayanne Basham at CRDEC, the military does not allow butyl seams to be abraded. Abrasion of butyl coating risks compromising the butyl fabric's structural integrity, and so abrasion, as a form of seam preparation, is not allowed. Not only does the abrasion restriction prevent uncured powdered butyl rubber from being used to make the Quick Doff Hoods, the abrasion restriction also makes effective gluing of the butyl rubber more difficult.

TASK 1A - LOCATING/DEVELOPING ADHESIVES FOR SEALING BUTYL RUBBER

While CAR was evaluating conventional butyl rubber related technologies, CAR was also looking for alternative butyl rubber adhesives. Recognizing that welding technologies were not a seaming/sealing possibility (see page 5), and having exhausted all of the butyl rubber fabric possibilities, CAR focused all of its efforts on finding alternate butyl rubber seaming/sealing adhesives. Since there are literally tens of thousands of different commercially available adhesives, several necessary adhesive characteristics had to be identified so that CAR could reduce the number of potential candidates. Identifying which adhesive characteristics were the most important involved an understanding of how adhesive are classified.

ADHESIVES CHARACTERISTICS AND CLASSIFICATIONS[1]

FUNCTION

Adhesives can be classified in any number of ways. They can be classified by function; structural or non-structural. Structural adhesives serve as critical load bearing components. Non-structural adhesives usually serve to fill gaps or carry light loads. In the case of the Quick Doff Hood an adhesive could carry all of the seam stress (i.e. be a structural adhesive) or could be used to fill the holes made in sewing the seams (i.e. be a non-structural adhesive).

PHYSICAL FORM

Adhesives can be classified by physical form. The physical state of the adhesive has a dramatic impact on the way the adhesive is handled during application. Liquid adhesives can be pumped, spread, sprayed, rolled or brushed into place. Liquid adhesives must be applied horizontally, however, to avoid dripping and running. Paste adhesives have higher viscosities than liquid adhesives. Paste adhesives can also be spread, rolled, and brushed but without the risk of dripping. Pumping and spraying viscous pastes is difficult, however, and frequently high pressures are involved. Tape and film adhesives are the easiest form of adhesive to manage, but tape and film delivery systems dramatically limit joint complexity. Powder and granule adhesives are easy to handle, but require either heating or solvent-activation during use and can only be applied to flat, horizontal surfaces. An electrostatic process for depositing powdered adhesives does exit, but is too costly to consider for use in all but the largest manufacturing operations.

ACTIVATION REQUIREMENTS

Adhesives can be classified by activation requirements. Adhesives can be made to stick (activated) with heat, pressure, atmospheric exposure (time curing), catalytic curing, chemical cross-linking (vulcanizing), and reactivation (via heat, a solvent, or a second coating). Classification by activation requirements is a little more ambiguous than some of the other classification methods because there are large numbers of adhesives whose bonds are affected by several of the activation parameters. In fabric

seaming applications any adhesive which requires more than a few second to become fully activated is not capable of meeting conventional fabric seaming expectations and can only be accommodated using unconventional seam manufacturing methods.

CHEMICAL COMPOSITION

Another way of classifying adhesives focuses on adhesive chemical composition. Of all the classification methods, classification by chemical composition is the most fundamental. There are four basic chemical adhesive families; elastomeric, thermosetting, thermoplastic, and alloyed (combinations of the first three). Elastomeric adhesives are adhesives based on polymers, both natural and synthetic, which have superior toughness and elongation characteristics (i.e. elastomeric adhesives are rubbery). In the elastomeric adhesive family is a sub-family of adhesive called "pressure sensitive adhesives" which will be discussed later.

Thermosetting adhesives are any adhesives which harden (or set) by undergoing a non-reversible chemical reaction. Usually the chemical reaction is initiated by heat hence the name thermo-setting.

Thermoplastic adhesives are adhesives which can be softened and re-hardened repeatedly. No irreversible chemical changes take place in thermoplastic adhesives. Usually softening is achieved using heat hence the name thermo-plastic. A sub-family of the thermoplastic adhesive family is the "hot-melt adhesive" family which will also be discussed later.

The final main chemical family of adhesives is the alloyed adhesive family. Just as the combination of different metals creates a metal alloy, the combination of different adhesives creates an adhesive alloy. Alloys are made by combining adhesives from two or more of the other three basic adhesive families.

Pressure Sensitives

Although all adhesive can be classified by one or more of the systems listed above, two general categories of adhesives which warrant additional discussion are pressure sensitive adhesives and hot-melt adhesives. As mentioned above, pressure sensitive adhesives are a member of the elastomeric adhesive family. Pressure sensitive adhesives are sticky by nature. Their "stickiness" can vary dramatically depending on their chemical formulation. An example of an aggressive pressure sensitive is two-sided foam tape used to stick posters to walls. A weak pressure sensitive is the adhesive placed on the back of "Post-It" notes used in secretaries' offices. Although pressure sensitives have a broad range of sticky characteristics they do share certain chemical properties.

Most pressure sensitives are made from acrylic polymers. Acrylics are inexpensive, and can be easily formulated to have different levels of bonding aggressiveness. Most pressure sensitives do not chemically react to form bonds. The adhesives rely on molecular entanglement between the surface of the adhesive and the surface of the adherends (the pieces being stuck together). Because the chemical composition of the adherends is not

generally critical, pressure sensitive adhesives can be formulated to stick to just about any surface. The ability to join dissimilar materials is the key to the commercial success of pressure sensitive adhesives. Unfortunately, in textile and sewn products, acrylic pressure sensitive adhesives have one major drawback, they are partly, if not completely, water soluble (i.e. they dissolve in water). Hot-melt adhesives, on the other hand, are not usually water soluble.

Hot-melts

As mentioned previously, hot-melt adhesives are a member of the thermoplastic adhesive family. Like all thermoplastics, hot-melts are solids at room temperature. What separates hot-melts from other thermoplastic adhesives is the dramatic change in viscosity that occurs when hot-melts are heated. Other thermoplastic resins turn into a viscous paste-like form when heated. Most hot-melts, on the other hand, turn into liquids when heated. The liquefaction of hot-melts makes them easy to apply when hot, and the fact that hot-melts are solid at room temperatures makes them easy to handle when cool. Another important characteristic of hot-melt compounds is that the performance characteristics of hot-melts can be modified with the addition of special compounds. *Stabilizers* can be used to retard oxidation, *tackifiers* to improve bond strength, *wax* to reduce viscosity and to alter surface characteristics, and various *fillers* to increase viscosity, melting point, and bond strength.[2]

ADHESIVE CHARACTERISTICS CAR WANTED

Knowing that modifications could be made to adhesives to adjust their performance characteristics, CAR set out to established a list of desirable adhesive characteristics and began using the list to screen potential adhesive candidates. The list below is a list of minimum adhesive requirements that CAR used to screen adhesive candidates. The adhesive,

- Did not need to be a structural adhesive. Sewing thread could carry all the structural loads.
- Had to be either be a paste, tape, or film adhesive. Liquid adhesives were already being used, required excessive curing times, and outgassed, and powder or granule adhesives could only be applied to flat stationary fabric seams.
- Had to cure using heat, pressure, or reactivation techniques. Time curing took too long. Catalyst curing and vulcanizing were not viable options either because they require expensive mixing and metering equipment.
- Had to be elastomeric because the butyl rubber itself is an elastomer. Matching adhesive and adherend physical characteristics produced more compatible and hence more durable bonds.

WHY COMMERCIAL BUTYL RUBBER ADHESIVES WERE ELIMINATED

Using the above criteria as a basis for evaluating adhesives CAR was able to eliminate a large percentage of commercially available adhesives. Unfortunately, avoidance of liquid adhesives, because of outgassing, eliminated all of the commonly available butyl rubber adhesives. To understand why butyl rubber adhesives are liquids that release fumes as they cure, one must understand how butyl rubber adhesives work.

HOW COMMERCIAL BUTYL RUBBER ADHESIVES WORK

The reason butyl rubber adhesives release fumes is because they contain volatile hydrocarbon solvents. The solvent molecules are smaller than the butyl rubber molecules, so the solvent molecules can fill the spaces between the butyl molecules. If adhesive molecules are suspended in the solvents (as with butyl rubber adhesives) both the solvent and adhesive molecules fill the spaces between the butyl rubber molecules. Molecular entanglement occurs between the adhesive and butyl rubber molecules occurs, and strong adhesive bonds are formed.[3]

Since molecular entanglement forms strong adhesive bonds, and since solvent based adhesives were ruled out, CAR needed to find an adhesive that would either get entangled with or react to butyl rubber molecules. Finding an adhesive to react with butyl rubber was deemed impractical because butyl rubber does not react with most chemicals (which is why butyl rubber is used to protect wearers from hazardous chemical

environments). Any adhesive that could chemically react with butyl rubber would probably be so chemically active that it would require special handling and precautionary measures to use. CAR felt it was easier (and safer) to find an adhesive that would entangle itself about the butyl rubber molecules.

ADHESIVE CHARACTERISTICS CAR NEEDED

Since solvent induced entanglement was unacceptable, CAR needed to find an non-solvent based adhesive that would encourage molecular entanglement as much possible. The only other ways for molecular entanglement to take place were if an adhesive,

- Had, at the point of application, similar flow characteristics to hydrocarbon solvents (i.e. initial low viscosity).
- Was compatible with the molecular structure to butyl rubber (i.e. was elastomeric).
- Was applied hot so its molecules were highly agitated.

and

- Was applied under pressure so that the adhesive molecules were squeezed into the butyl rubber molecules.

These additional restrictions on adhesive performance, combined with the original restrictions that eliminated conventional butyl rubber cements,

meant that the adhesive had to have the viscosity characteristics of a hot-melt and the elastomeric characteristics of a pressure sensitive.

EVALUATION OF COMMERCIAL PRESSURE SENSITIVES

3M ACRYLIC PRESSURE SENSITIVES

CAR began the search for new butyl rubber adhesives with the world's largest manufacturer of pressure sensitive adhesives, 3M. 3M has a broad line of commercially available pressure sensitive tapes and tape dispensers. Five adhesives that 3M recommended for bonding cloth were; A25, A40, A60, R40 and R50. Four adhesives that 3M recommended for bonding rubber were; A60, R30, R40 and R50. The "A" adhesives were acrylic based, the "R" adhesives, rubber based. Since CAR was trying to glue butyl rubber, CAR focused on 3M's rubber adhesives first.

Unfortunately, 3M described all of the rubber cements as having "medium" to "low" solvent resistance. The same was true for the A60 family of adhesives which were the only other adhesive family 3M recommended for bonding rubber. This left CAR with a difficult choice. Should CAR use an adhesive designed for bonding rubber but with low solvent resistance, or should CAR use an adhesive not designed for bonding rubber but with better solvent resistance?

Bond Strength Versus Solvent Resistance

To resolve the bond strength versus solvent resistance issue, CAR re-examined seaming/sealing techniques. CAR had already determined that

the hoods could not be seamed and/or sealed using welding technology. This left three options. As a first option, the hoods could seamed and sealed using one adhesive. As a second option, the hoods could seamed using one adhesive and sealed using another adhesive. And, as a third option, the hoods could be seamed by sewing and sealed using an adhesive.

The first option represented the ideal one-step seam/seal production solution. As such, CAR wanted to find (or formulate) an adhesive that could both seam butyl rubber and simultaneously seal butyl rubber. CAR hoped that such an adhesive would have enough bond strength to hold butyl rubber pieces together and enough solvent resistance to withstand all (or most) of the same solvent attacks that the butyl rubber could withstand. An evaluation of 3M's pressure sensitive adhesives had already shown that using 3M pressure sensitives required a trade-off between bond strength and solvent resistance. So, the first seaming/sealing option was ruled out for 3M adhesives.

The second option, of using one adhesive to seam butyl rubber and another to seal butyl rubber, could have been accommodated using 3M's adhesives. However, CAR's experience with the Stitchless Chemical Protective Suit had shown that, in all but simple straight seams, adhesive seaming was much more difficult than sewing. Given the difficulties associated with adhesive seaming, CAR decided that the third option was best for 3M adhesives.

As a third option, the hoods could be seamed by sewing and sealed using an adhesive. If the hoods were sewn, the sewn seams would carry all the stress loads, and the adhesive would simply seal the sewn seams. As such, the adhesive's sealing abilities and solvent resistance would be more important than the adhesive's ultimate bond strength. Having identified that 3M's A25 family of adhesives were recommended for fabric application and had "medium" to "high" solvent resistance, CAR stopped evaluating 3M's rubber adhesives and began evaluating 3M's acrylic based fabric adhesives.

926 And 9500 PC

In the A25 family of adhesives were five products, namely; 926, 922 XL, 9482 PC, 9485 PC, and 9500 PC. In 3M's adhesive literature (Designer's Reference Guide to High-Performance Bonding Tapes for Product Design and Assembly) only 926 was recommended for bonding fabric, and only 9500 PC had a carrier core. CAR decided that 926 was the best adhesive 3M had to offer, and CAR decided to test 9500 PC in the hope that its polyester carrier might improve the A25's handling and butyl bonding characteristics.

Application Method

To apply the 926 and 9500 PC adhesives CAR had three choices. The adhesives could be applied by hand. The 926 could be applied with an inexpensive hand-held dispenser offered by 3M. And, the 9500 PC could be applied with a standard packaging tape dispenser. CAR decided to

purchase the hand-held dispenser (3M part: ATG 752 Adhesive Applicator) because not only could it apply the 926, it could simultaneously remove the 926's release paper backing. CAR used the ATG 752 dispenser to make one inch wide lapped seams out of washed butyl rubber and 926. CAR made similar seams using 9500 PC, and both adhesives were tested for grab strength according to Federal Test Method Standard Number 191a Method 5100 (see Appendix A).

Test Results

Despite 3M's claim that 9500 PC had "very high" ultimate sheer and peel strengths, the 9500 PC did not produce adequate butyl rubber seams. Even the best seams made with 9500 PC could be readily peeled apart by hand. Billed by 3M as a "high performance adhesive with excellent temperature and solvent resistance", 926 seam samples performed better than seam samples made with 9500 PC. Although an average butyl-to-butyl seam grab-strength of 54.2 pounds per linear inch (lbs./linear inch) was obtained using 926, all of the butyl samples failed at the seams. Also, 54.2 lbs./linear inch average bond strength was only obtained after the samples were "aged" for two days according to 3M's recommendations. Seams made of freshly applied the 926 were easy to pull apart by hand. With 926 as a reference, CAR decided to look for a pressure sensitive which produced better short-term and ultimate bond strengths and had better temperature and solvent resistance.

To narrow the search, CAR tried to think of an everyday adhesive applications that would required temperature and moisture resistance, flexibility, and good storage characteristics. Industrial roofing is flexible, exposed to seasonal temperature extremes, exposed to water and cleaning solutions, and requires simple and straight forward installation methods. CAR felt that an adhesive used by industrial roofing industry might have the performance characteristics needed for bonding/sealing butyl rubber. After making several phone calls to industrial roofing installers, CAR found the name of a company which made a line of adhesive backed industrial roofing products. The company's name was KemTek Specialties Corporation.

MACE URETHANE PRESSURE SENSITIVES

SSI And SSII

Conversations with Jack Rustico, the production manager at KemTek, showed that KemTek is essentially a textile converter. KemTek takes base materials and coats them with polymer films including adhesive films. The adhesive that KemTek uses on its industrial roofing product is supplied by Mace Adhesives and Coatings. Discussions with Joe Fenarra, who at the time worked for Mace, revealed that the adhesive Mace supplies to KemTek is a toluene-solvent based, liquid, fully cross-linked, urethane, pressure-sensitive adhesive (CAR called it Sticky Stuff I or SSI for short.). KemTek took Mace's liquid adhesive, knife coated a thin layer of the adhesive onto a roofing substrate and then, using heat, boiled off all of the solvent in the adhesive leaving a yellow, transparent, extremely sticky, urethane film.

CAR obtained samples of Mace's adhesive in both liquid (SSI) and solid (SSII) form. The SSI was a low viscosity, yellow, sticky liquid which looked a lot like thin maple syrup. SSI smelled strongly of toluene and had the annoying tendency of getting everywhere. The SSII, on the other hand, was a high viscosity, yellow, sticky solid. Unlike SSI, SSII had very little solvent smell and, as a solid, was much easier to handle than SSI. Once SSII stuck to a surface, however, it was difficult to remove. CAR did find that isopropyl alcohol and elbow grease could remove SSI from table and floor surfaces without dissolving the surfaces (unlike toluene, acetone, and methyl ethyl ketone).

SSIII

When CAR made butyl rubber seam samples with the SSII the bond strengths were not much better than any of the other adhesives CAR had tried. So, CAR asked Joe Fenarra to supply a different dried adhesive. The new adhesive Joe sent was identified as SSIII. In manual tests the SSIII produced stronger seams than the SSII. Knowing that Mace's urethane adhesives were non-water soluble and that their performance could be modified, CAR decided to give urethane pressure sensitives additional attention.

Continuing to investigate the performance capabilities of SSIII, CAR made butyl rubber seams using washed, unwashed, abraded, unabraded, light weight, and heavy weight butyl rubber. CAR tested the butyl rubber seams

by hand using the harshest adhesive test possible. CAR subjected the seams to peel stresses. In general adhesives are weakest in peel (as opposed to shear and pure tension), and SSIII was no exception to the rule.

SSIV - Mace 2451

When placed in peel the SSIII produced long stringy strands in much the same way that chewing gum on hot asphalt produces long stringy strands when you step in it. CAR hypothesized that SSIII has low peel resistance because the adhesive strands (legs) reduced SSIII's ability to distribute forces between adherends. The less distributed the forces, the more likely the adhesive will fail. To improve SSIII's peel resistance CAR asked Joe Fenarra to have Mace produce a less "leggy" urethane pressure sensitive. The result of Mace's efforts was Mace 2451 adhesive (CAR called it SSIV). The SSIV was sent to CAR as a 12 inch wide 3-4 mil (three to four thousandths of an inch) thick film coated onto silicon release paper.

Initial Performance Evaluation

CAR's first experiment with SSIV (see Appendix B) was aimed at determining what kind of performance could be expected from the new adhesive to see if it was worth investigating further. The experiment was performed on the assumption that since single layers of SSIII had produced only marginal results the same would hold for SSIV. Consequently, multiple layers of SSIV would have to be used to obtain reasonable bond strengths. Using washed butyl rubber as the adherends,

one inch seams were made with different quantities of SSIV, and the seams were tested according to Federal Test Method 5100.

Seams made using two layers of SSIV had a disappointing average grab strength of 32 lbs./linear inch. Seams made using four layers of SSIV had a more respectable average grab strength of 53 lbs./linear inch. Adhesive tapes made with two layers of SSIV bonded to washed butyl rubber and then aged (by sitting around for several days while the Instron test equipment was being scheduled) produced bond strengths of 60 lbs./linear inch (3M's 926 had produced aged butyl seams with an average strength of 54.2 lbs./linear inch). Since Mace's urethane adhesive was more solvent resistant than 3M's acrylic adhesive, and since the tapes made with SSIV were easy to handle and produced stronger seams that the 926, CAR decided to make some experimental hoods with SSIV tapes.

Experimental Hoods Made

The process used to make the experimental hoods was as follows:

- First, one inch bias-cut strips of washed butyl rubber were coated with two layers of SSIV. This process was done by placing one layer of adhesive onto the butyl tape, applying heat, placing a second layer onto the tape, and the applying heat again. In addition to the one inch bias-cut butyl tape, 1-1/2" strips of straight-cut butyl were also coated with two layers of SSIV.

- Second, the hood pieces were sewn together using continuous filament polyester thread in order to form the preliminary garment.
- Third, the one inch bias-cut SSIV tape was then applied along the main seam of the hood using a Queen Light QHP-778L heat sealing machine (see Figure 1 on the next page). In addition to placing the tape on the main seam, tape was also placed on the front seam in order to seal the entire garment.
- Fourth, the elastic was placed around the neck of the hood. This process was done by placing the elastic onto a 1-1/2" adhesive backed butyl strip. This adhesive strip was then placed along the neck of the hood and pressed into place using a hot hand held iron.
- Fifth, the hem and the straps of the hood were sewn onto the garment using the same polyester thread. Patches coated with Sticky Stuff IV were placed onto the hood where the straps were sewn for reinforcement purposes.

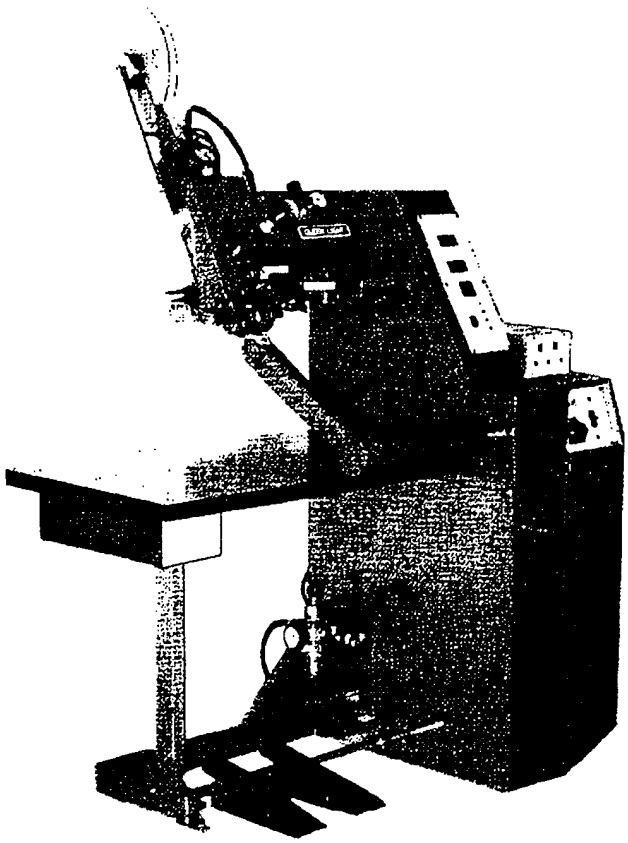

Figure 1 - Queen Light QHP-778L Heat Sealing Machine

The Queen Light machine worked as follows: As the adhesive backed butyl tape was being fed into the machine the adhesive's release-paper backing was peeled off by hand (a later modification peeled the release-paper automatically). The machine then heated the adhesive using hot air. The hot adhesive-tape contacted the hood seam and was squeezed into the hood by two heated nip-rollers. Once the tape was applied a pedal-actuated cutter trimmed the tape and automatically readied more tape for the next seam.

Hood Design Change Recommendations

In making the first Quick Doff Hoods sealed with urethane pressure sensitive adhesives, CAR made the following observations:

- A needle feed sewing machine equipped with a Teflon coated presser foot made sewing of the butyl rubber straight forward.
- The finished polyester thread that was used to sew the fabric was not correct for the application. The silicone finish tended to migrate onto the butyl fabric and could diminish the bond strength between the butyl backed adhesive tapes and the sewn seams. The thread was also stronger than necessary. CAR felt a cheaper, unfinished thread would perform as well and cost less than the thread currently being used. CAR recommended a cheaper unfinished, military-green, tex-35, spun polyester, sewing thread.
- Seaming and sealing the left and right hood pieces together first made attaching the neck elastic easier. With the ends of the neck elastic bartacked in place the hoods could be turned inside-out over a simple wooden template. The template helped stretch the elastic tight. A 1-1/2" wide, straight-cut, SSIV backed, butyl tape could then be ironed over the elastic and onto the hood. The tape not

only secured the elastic with fewer parts than previous designs, the tape also sealed the end bartacks.

- Unfortunately, attempts to glue the face elastic in place failed. The only way CAR could reliably secure the face elastic was by forming a tunnel hem using a folder and a needle feed sewing machine. A portion of the tunnel hem was left open, and the elastic cord was fed through. Once the cord was through, the ends were tied, and the tunnel hem was sewn shut. The elastic pull tabs were then sewn into place.
- The curve on the hem of the hood was difficult to sew. A sample pattern with a squared off hem was designed in order to improve the sewability and to improve the physical appearance of the hood.

Seam Tape Optimization

Making the sample hoods convinced CAR that SSIV performed well as a butyl seam tape. CAR decided to conduct a second set of experiments aimed at optimizing the seam tape to produce the best butyl seam strengths. In the second series of test CAR wanted to determine:

- If multiple layers of SSIV were necessary,
- How width affected the seam tape performance,

and

- How well SSIV resisted peeling.

Are Multiple Layers Of SSIV Necessary?

During the Chemical Protective Suit Project experiments had shown that adhesive thicknesses play a critical roll in adhesive seam performance. CAR learned that there is a fine line between providing too little and too much adhesive. CAR knew that if the adhesive was too thin, the adhesive would not bond the adherends at all. CAR also knew that if the adhesive was too thick the adhesive would become the major load bearing component between the adherends. CAR wanted the SSIV tapes to avoid being load bearing structures by distributing loads from adherend to adherend in much the same way that rivets avoid being load bearing structures by distributing loads from one riveted part to the next. Therefore, the adhesive had to be thick enough to bond the adherends but not thick enough to be load bearing.

Having already conducted tests on multiple layers of SSIV (see Appendix B) CAR decided to conduct a series of tests using single layers of SSIV. Using single layers of SSIV, CAR made one inch butyl-to-butyl seams with average grab strengths of 64.6 lbs. per linear inch (see Appendix C). With the Appendix C results in hand, CAR concluded that the best results obtained in Appendix B were due to aging and not multiple SSIV layers. This was good news since it implied seams made with SSIV would improve with age. Discussions with Jack Rustico confirmed CAR's findings and brought another interesting piece of Mace 2451 adhesive information to light.

CAR learned that the thinnest coatings of Mace 2451 KemTek could produce on its production equipment were 3-4 mils (0.003" - 0.004") thick. Since the laboratory sample of SSIV film used in Appendix C was also 3-4 mils thick, CAR was getting optimum results from a product that could be produced on a full-production basis. This meant that if CAR wanted to produce SSIV backed butyl rubber adhesive tapes CAR could have KemTek coat butyl rubber with Mace 2451 on a production basis. According to Jack, KemTek could also slit the coated butyl rubber into any widths CAR desired. To determine what widths worked best, CAR decided to conduct a test to see how tape widths affected tape performance.

How Does Width Affect SSIV Seam Tapes?

Knowing that the strength of adhesive seams would most likely increase with seam width, CAR decided to address several issues in one test. Making test specimens different widths, CAR could confirm that wider seams were stronger. CAR could also evaluate seam strength versus seam flexibility issues. Using a peel test, instead of a grab test, CAR could also learn how SSIV behaved when peeled. CAR decided to conduct a peel test (Federal Test Method Standard Number 191a Test 5950) on butyl-to-butyl seam samples varying in width from one inch to two inches in one-quarter inch increments (see Appendix D).

CAR found no surprises in the results. Taking into account random variability as measured by the standard deviation (an increase in the standard deviation implies an increase in variability), the general trend in

the data was that the seam strengths increased with seam width. The lower average peel strengths obtained from the 1-1/2" and 1-3/4" seam samples were considered the result of higher random variability. CAR also found that the thicker seams were stiffer and less accommodating of complex contours. The peel test also confirmed that, like all adhesives, SSIV had a lower peel strength than grab strength. Pleased with the overall performance of SSIV, CAR decided to try making more Quick Doff Hoods. Instead of using SSIV adhesive tapes made by hand, however, CAR decided to use SSIV tapes produced on KemTek's production equipment.

Mace 2451, Run XU256 (SSIV)

Satisfied with the performance of the finished experimental hoods, CAR decided to have 25 yards of lightweight butyl rubber coated with 4 mils of Mace 2451. KemTek coated the adhesive onto washed Quick Doff Hood butyl rubber, and the resulting coated butyl was identified as Run XU256. Following coating Jack Rustico and KemTek slit the coated butyl into various width tapes. Some of the tapes were bias cut (cut diagonally across the fabric) and some tapes were straight cut (cut along the length of the fabric). In the interim CAR digitized, cut, and sewed 25 hoods which incorporated the changes that resulted from making the first experimental hoods.

Production Hoods Made

Once all of the necessary materials had arrived CAR proceeded to make the hoods. CAR used the March 13, 1992 hood specifications but included the

were so many holes in the fabric that the material looked like a field of stars. The second quality butyl rubber, combined with the poor bond strength of the SSIV pre-formed tapes, convinced CAR not to ship the hoods and to begin trouble shooting. Acquiring and inspecting a new roll of butyl rubber solved the pin-hole problem, but finding why the SSIV tapes were defective took some time.

Was Adhesive The Problem?

Analysis and discussions with Joe Fenarra and Jack Rustico showed that insufficient drying of the SSIV film must have occurred during KemTek's coating procedure. As part of the normal processing procedure toluene is added to the liquid Mace 2451 to adjust the adhesive's viscosity so that pumping and knife coating of the adhesive is easier. After the adhesive is applied it goes through a drying process to evaporate all of the toluene. If the toluene is not completely evaporated before the release paper is applied the urethane does not become sufficiently viscous to allow proper bonding. CAR, Mace, and KemTek concluded that the 25 yards of butyl rubber used in the production run was too short to allow proper equipment set up, and that some of the toluene had not boiled out of the SSIV butyl backed tapes before the silicon paper had been applied and the roll of goods had been slit.

Did Improper Adhesive Application Contribute To The Problem?

To ensure that the toluene trapped in the butyl/SSIV tapes was indeed the source of the seam strength problems, several tests were conducted to find out if varying application parameters could dramatically affect seam

performance. In the first test seams were made using a variety of non-automated processes and tested for grab strength (see Appendix E). In the second test seams were made using various settings on the Queen Light machine, and the seams were tested for peel strength (Appendix F). Despite the small sample sizes used for the tests, several general conclusions could be drawn.

- Within each test, application parameters did not affect seam performance enough to be considered the major cause of variability between the first experimental hoods and the failed hoods.
- The original SSIV (tested in Appendix C) produced seams with much higher grab strengths than the new SSIV-Run XU256 (tested in Appendix E).
- Considering experimental variability, it is likely that the peel strength of the original SSIV (tested in Appendix D) is higher than the peel strength of SSIV-Run XU256 (tested in Appendix F). A designed experiment would have to be performed to confirm this hypothesis, however.
- Unless SSIV is improved, even with proper drying, the Quick Doff Hood seams would have to be sewn to meet the minimum strength requirements.

Having confirmed that the SSIV-Run XU256 pressure sensitive coating was indeed weaker than the original SSIV, CAR decided to evaluate a

completely different family of adhesives with the hope of finding an alternative adhesive with more uniform manufacturing characteristics. Having determined early on that two desirable adhesive characteristics were an initial low viscosity and the ability to be applied hot (see page 21), CAR decided to investigate the thermoplastic family of adhesives (see page 16).

EVALUATION OF COMMERCIAL HOT-MELTS

POLYESTER AND POLYAMIDE THERMOPLASTICS RULED OUT

As discussed on page 5, CAR failed to bond butyl rubber using a polyester based fabric-adhesive, Pellon SP28, and a polyamide (nylon) based fabric-adhesive, from ElectroSeal. Being thermoplastic in nature, both adhesive were designed to be applied using heat, but neither adhesive was designed to have the low melt viscosities. The failure of these two fabric adhesives led CAR to pursue pressure sensitive adhesives, but with the failure of the SSIV Run XU256, CAR decided to re-evaluate thermoplastic adhesive technology. Once again, CAR tried to think of an everyday adhesive application requiring temperature and moisture resistance, flexibility, and good storage characteristics. This time, however, the adhesive had to have a very low melt viscosity (i.e. the adhesive had to be a hot-melt, see page 18).

HOT-MELTS - STARENSIER PUDDLE COAT™

At the September 1991 Bobbin Show CAR had been introduced to one of the largest adhesive manufacturers and textile converters in the shoe industry, Starensier. Recognizing that the adhesive systems in shoes are subjected to

harsh treatment, CAR decided to re-establish contact with Starensier to see if they had any adhesives which might work on butyl rubber. Discussions with Mr. Hy Lamb, Vice President of Sales for Starensier, revealed that Starensier specializes in coating fabrics with hot-melt adhesives, just what CAR was looking for. When asked to recommend a hot-melt for butyl rubber applications Hy suggested Starensier Puddle Coat™.

Application Method

Puddle Coat™ is a non-solvent based, hot-melt adhesive, that is normally pre-formed into dry film sheets and heat fused between shoe uppers and foam liners to form durable, breathable, composite fabrics. CAR asked for samples of Puddle Coat™ film which were cut into strips and used on the Queen Light machine to bond washed, solvent wiped, lightweight, butyl rubber pieces together. The initial bonds were not as strong as the original SSIV bonds, but the Puddle Coat™ was easier to handle because it was not sticky. Hy Lamb suggested that the Puddle Coat™ would make a better seam/seal tape if it were coated onto butyl rubber that was sewn at the end of a full production run. CAR agreed, and arrangements were made to provide Starensier with some washed and solvent wiped butyl rubber samples which Starensier would then coat with a layer of Puddle Coat™.

Problems Encountered

Because Puddle Coat™ is a hot-melt adhesive which sets on cooling, Starensier assumed that, once cooled, the coated butyl rubber would be completely stable and safe to ship. Under this assumption, Starensier

folded all the coated butyl rubber and shipped it to CAR. Upon receipt, the adhesive coating had stuck to itself in several places. Whether this was because the butyl had gotten hot during shipment or because the coating had not fully cured is uncertain. Separating the goods only damaged the coating because the coating to coating bond was stronger than the coating to butyl bond. The butyl coating was sufficiently damaged that slitting was impractical. Seam samples were made with the salvaged portions of the undamaged coated butyl, but simple hand tests showed that the seam samples were not as strong and peel resistant as early SSIV samples.

Starensier's Answer And CAR's Response

Aware of CAR's seam test-results, Hy Lamb stated that residual talcum powder on the washed butyl rubber was probably responsible for the poor performance of the Puddle Coat™ adhesive. Hy recommended that CAR send Starensier 100 linear yards of *starch* powdered butyl rubber instead. Hy believed Starensier could process the 100 yards of butyl continuously by washing the starch off the butyl rubber with Starensier's portable wash box, drying the butyl in an in-line continuous curing oven, and then coating the butyl on an in-line coating range.

Discussions with Dr. Aspland at the Clemson University' School of Textiles revealed that to effectively remove starch from any fabric requires at least ten to fifteen minutes exposure to an enzymatic bath, something which no portable wash box is large enough to do. Since CAR had already sent Starensier butyl rubber that was as clean as reasonably possible, and since

Starensier did not have the means for making cleaner butyl rubber, and since none of the Puddle Coat™ samples produced bonds better than original SSIV, CAR abandoned Puddle Coat™ as an alternative to SSIV.

RETURN TO PRESSURE SENSITIVES

MACE 2882, RUN XU270

While CAR was investigating Puddle Coat™ as an alternative to SSIV, Joe Fenarra had been busy formulating his own alternative called Mace 2882 . Using more than 75 linear yards of joined fabric, including 15 linear yards of washed hood butyl rubber and 15 liner yards of unwashed hood butyl rubber, KemTek ran a full production run (run XU270) coating the joined fabrics with a 4-6 mil thick film of Mace 2882. This time Jack Rustico made certain that all the toluene was removed from the adhesive coating before the goods were slit. Jack also made sure that all of the goods were straight cut, per CAR's instructions (see page 38 for the reason).

APPLICATION METHOD - MODIFIED QUEEN LIGHT QHP 778L

To make sure that tape application was not a source of production problems, CAR made a special attachment for the Queen Light QHP-778L heat sealer (see page 31). The attachment automatically peeled the release paper away from the Mace 2882 adhesive tapes as the tapes were fed into place. Automatic preparation of the adhesive tapes allowed CAR to minimize tape tension thus preventing seam puckering and subsequent tape failure (see page 38). By eliminating application parameters as a

source of seam tape failure CAR ensured that the burden of seam tape success (or failure) lay with the Mace 2882 adhesive tapes.

PRODUCTION HOODS MADE

Given the pass/fail nature of the Mace 2882 adhesive performance, CAR jumped straight into manufacturing hoods. To give CRDEC a chance to evaluate Mace 2882 application parameters, CAR decided to make five groups of hoods with three hoods per group. In groups one through four the hood pieces were washed three times in a commercial washing machine. In group five the hood pieces were unnecessarily washed in a caustic solution.

At the time, CAR was under the false impression that Archer Rubber used zinc stearate in the manufacture of military butyl rubber. Zinc stearate is a lubricating agent frequently used in rubber compounding. If zinc stearate had been present on the surface of the butyl rubber the caustic solution would have reacted with it forming an easy to rinse-off detergent. Since Archer's butyl rubber does not contain zinc stearate the caustic bath did nothing more than remove trace surface oils from the hood pieces. The removal of trace surface oils was a better preparation than some of the Mace 2882 butyl tapes received, however.

To see if the Mace 2882 was sufficiently robust to handle poor tape manufacturing, CAR decided to make the first two groups of hoods using the Mace 2882 that had been coated onto unwashed butyl rubber. CAR

reasoned that, for a purchaser of Mace 2882 tapes, the worst thing that could happen, beyond insufficient drying, was coating of the adhesive onto unwashed butyl rubber. To ensure a well rounded test, the remaining three groups of hoods were made using Mace 2882 coated onto washed butyl rubber.

To help keep track of which components were made of washed butyl rubber and which components weren't, CAR made the following list:

- Group 1 - This group was taped on the outside with unwashed butyl coated with SSIV.

- Group 2 - This group was taped on both the outside and inside with unwashed butyl coated with SSIV.

- Group 3 - This group is taped on the outside with washed butyl coated with SSIV.

- Group 4 - This group was taped on both the outside and inside with washed butyl coated with SSIV.

- Group 5 - This group was cleaned in a caustic solution and taped on the outside with washed butyl coated with SSIV.

RECOMMENDATIONS

In making the five groups of hoods CAR came to the following conclusions:

- If the military chooses to tape seal the Quick Doff Hoods, the military should follow industry's lead and only use straight cut adhesive seam tapes.

- Commercially available tape sealing machines should be modified with better prefeed devices to ensure tension-free tape application.

- If adhesives with release paper backings are going to be used release paper waste will be generated equivalent to the yardage of adhesive tape used. Ways of controlling the waste paper production will have to be established.

- With the Mace 2882, or any other sticky adhesives, once the release paper is removed handling errors become difficult to correct. Therefore, the release paper should be removed at the last possible opportunity.

- Heating the adhesive tape with the release paper still attached makes removing the release paper much easier. If the pre-feed zones could also be turned into pre-heat

zones, the entire tape sealing process would be dramatically improved.

TEST RESULTS

Once the hoods were completed CAR submitted the hoods to CRDEC for evaluation. While CAR was waiting for CRDEC's test results, CAR made additional seam samples for long-term evaluation. After two months of aging the seams showed no visible signs of deterioration. To verify that the seam tapes were still doing the job of sealing the sewn seams, CAR examined the samples under a scanning electron microscope (see Figure 2).

Figure 2 - Composited Scanning Electron Micrographs of a Taped Butyl Fabric Seam

Starting at the bottom of Figure 2 and working up: The lower-most grey layer is part of a seam overlap on the inside of a Quick Doff Hood. The next grey layer is the outer seam fabric layer of a Quick Doff Hood. In between the inside and outside seam layers is a black void which is an air-gap. The top-most grey layer is the butyl backing of a Mace 2882 adhesive tape. Between the outside fabric seam layer and the adhesive tape backing are several items of interest. To the left is a film layer of Mace 2882. In the center is a triangular shaped, black, air-gap. To the right (in the upper right-hand corner of Figure 2) is a sectioned sewing thread.

Close examination of Figure 2 shows that the Mace 2882 does not form a continuous bond between the outside butyl fabric layer and the butyl tape backing. Instead, an air-gap forms where the tape must pass over the sewing thread. As long as the Mace 2882 is well bonded to the areas away from the sewing thread, however, a strong hermetic seal should be formed.

Figure 2 also shows that the seam sample was not sewn correctly. The nylon scrim in the butyl rubber is clearly visible, and shows that the thick butyl coating of one of the fabric layers is facing to the inside of the hood when it should be facing to the outside. Following this observation, CAR took more care in assembling experimental hoods to ensure that the thick butyl layer always faced to the outside.

Figure 2 also convinced CAR that the Mace 2882 Run XU270 had good aging characteristics. The lack of voids in the adhesive film, except near the thread, shows that the Mace 2882 did not separating from the butyl rubber even after two months. With visual evidence that Mace 2882 was working properly, CAR was ready to bring Task 1A to a close, when Joe Fenarra came back into the development picture.

MACE 2451, RUN XU???

While CAR was waiting for confirmation from CRDEC that the Mace 2882 had worked properly, Joe Fenarra was busy himself. Still displeased that the Mace 2451 had not been properly processed, Joe convinced Jack Rustico to have another try at coating the Mace 2451 onto washed butyl rubber. Jack agreed, and CAR received a new batch of Mace 2451 for evaluation. But, before CAR could evaluate the adhesive, Joe switched adhesive companies and used his new company's expertise to produce a radically new butyl rubber adhesive.

ALLOYED ADHESIVE = PRESSURE SENSITIVE + HOT-MELT

WORTHEN E-9

When Joe left Mace he began work for Worthen Industries, an adhesive company which manufactures hot-melt adhesives (as opposed to Mace's pressure sensitive adhesives). Joe took the knowledge he gained from working with Mace's pressure sensitive urethanes, and used it to help Worthen produce a new urethane-based hot-melt adhesive. The new urethane hot-melt (Worthen Product E-9 6/29/93) had many of the same

properties as Mace's urethanes. The hot-melt was honey yellow and moderately sticky. Instead of staying a sticky solid, however, the new adhesive turned into low viscosity liquid when heated according to the following schedule (see Figure 3):

Worthen Industry's E-9	
Temperature (^{0}F)	Viscosity (Cps)
300	10,000
325	6,500
350	4,200
375	THERMAL DEGRADATION

Figure 3 - Worthen E-9 Temperature vs. Viscosity Chart

APPLICATION METHODS

When heated the liquid E-9 could be coated onto fabrics just like the Mace adhesives. The only difference in the coating process was that when the E-9 cooled a flexible, solvent free, film was formed without the need for any post curing. Butyl seam samples made with Worthen E-9 showed that its low melt viscosity allowed it to bond aggressively to even unwashed butyl rubber. In addition, E-9 tapes could be finished with a powder coating made of the same powder that was used as a filler in the E-9's formulation. The powder coating acted just like release paper only the powder lost its effectiveness when heated. This meant that Worthen's E-9 was easier to handle than any of Mace's adhesives because there was no release paper to deal with. Also, the fact that E-9 was not as sticky as Mace's adhesives added to E-9's ease-of-use.

TEST RESULTS

To verify that E-9 could produce seam strengths comparable to the best seams constructed with any of the other adhesives, Mr. Madhu Nagaraja conducted a large number of tests (see Appendixes G through M). In the initial tests LSa seams were constructed with one inch wide adhesive overlaps according to Fed. Test Method Std. No. 191a Method 5100. Test 5100 specified that the seam samples be made with fabric pieces six inches long and four inches wide. Unfortunately, the initial seam samples were accidentally cut six inches long and three inches wide. For consistency, all subsequent test specimens were also cut six inches long and three inches wide.

The first seams tested were made as shown in Figure 4.

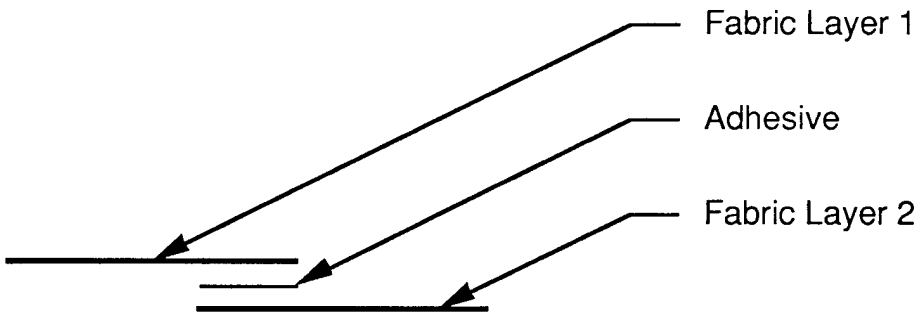

Figure 4 - LSa Adhesive Seam

Unfortunately, LSa seams cannot be constructed using adhesive backed butyl rubber tapes. Since no specific test method exists for taped seams, CAR decided to conduct additional experiments using Test 5100 on the two taped seam configurations shown in Figures 5 and 6.

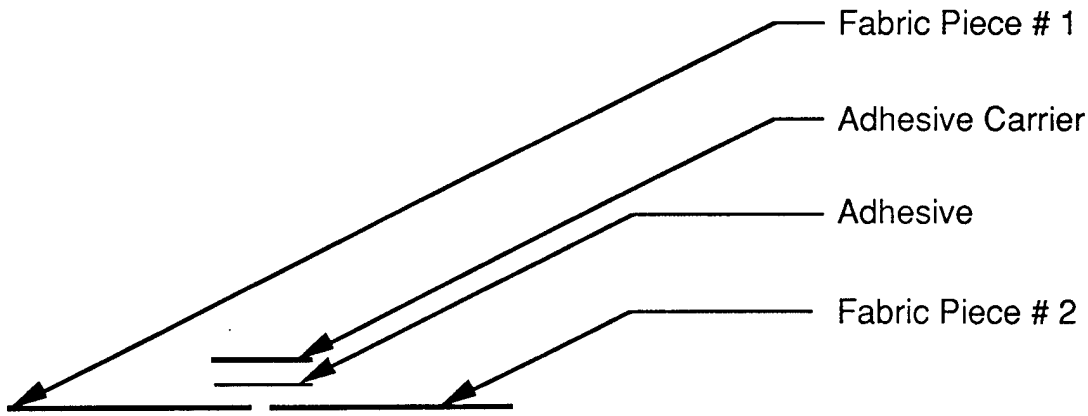

Figure 5 - LSp Adhesive Seam

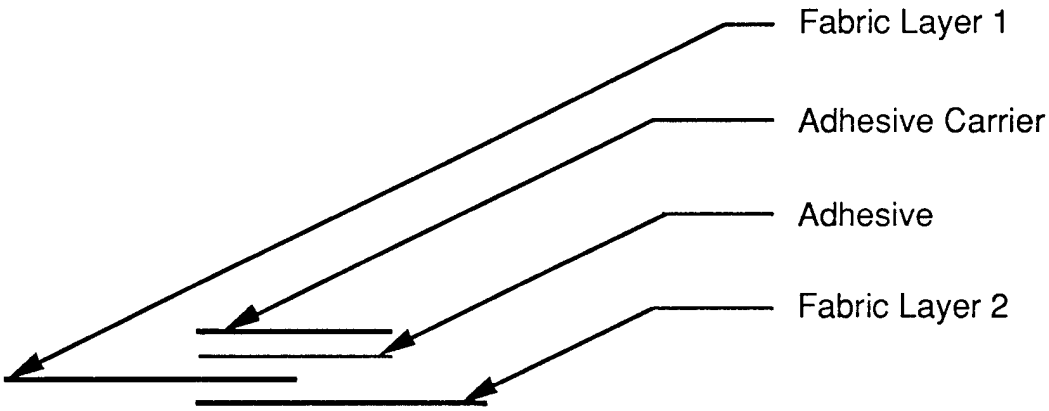

Figure 6 - LSaa Adhesive Seam

Figure 6's LSaa seam is very similar to the sewn and taped seams originally designed into the Quick Doff Hood. The difference is that in the original Quick Doff Hood, Fabric Layers 1 and 2 were sewn together and then taped. CAR, therefore, decided to add to its list of experiments by testing seams made according to Figure 7.

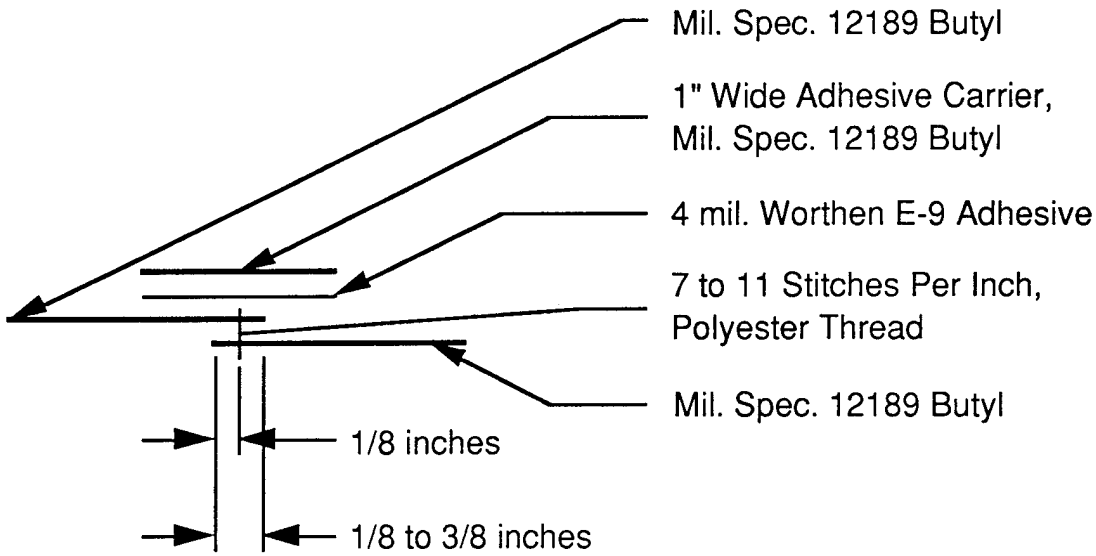

Figure 7 - Quick Doff Hood LSaa Sewn and Taped Seam

Although the original hood design called for 1-1/2" wide seam tapes, the Queen Light machine could only accommodate one inch wide tapes, and so one inch wide seam tapes were used. CAR also used straight-cut tapes instead of the bias-cut tapes specified in the original hood design (see page 38 for the reason). All the seams were constructed using washed and un-washed butyl rubber and a variety of other application parameters (see Appendixes G through M for more details). The final test results were condensed into Figure 8 on the next page.

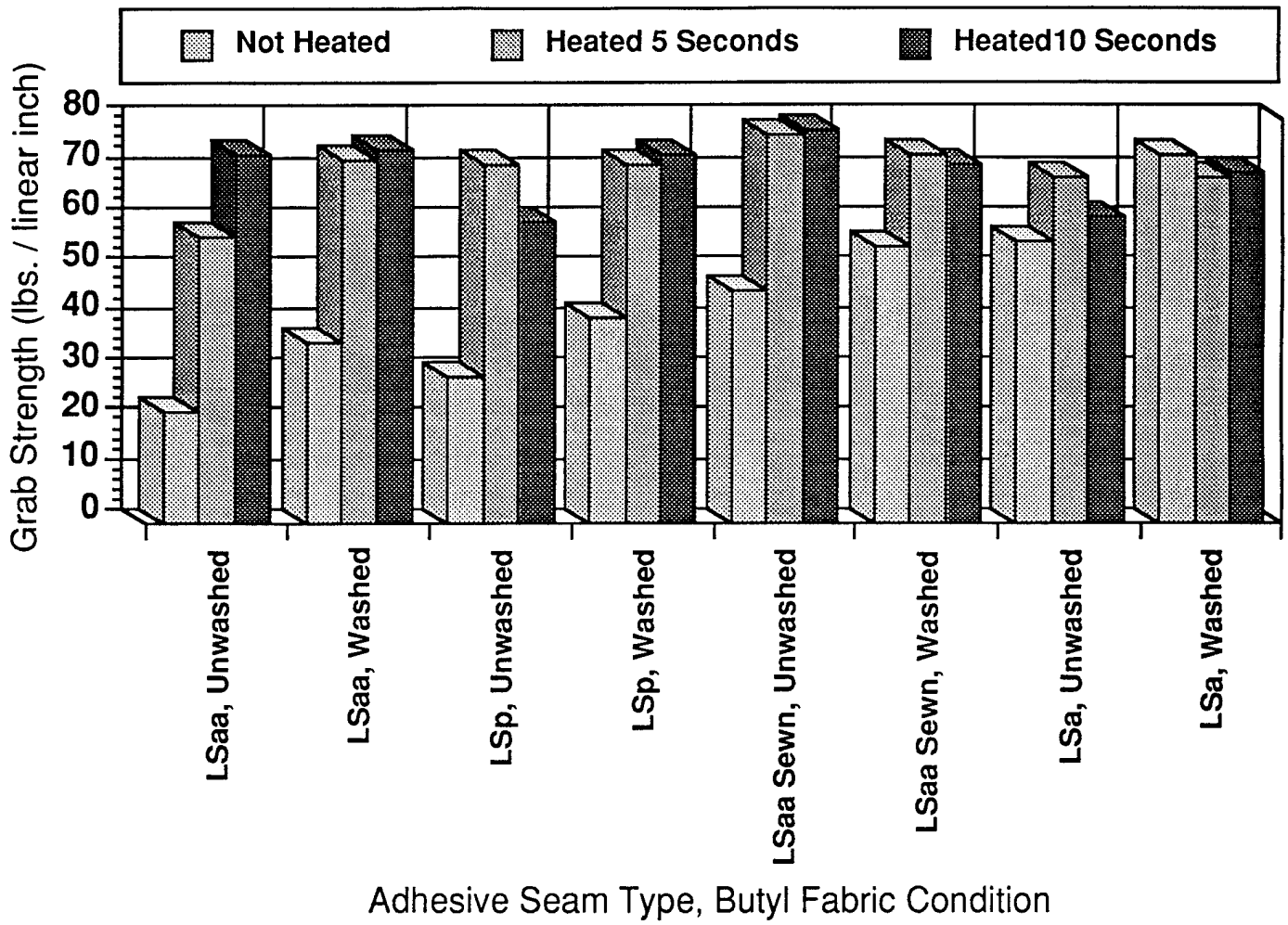

Figure 8: Worthen E-9 Seam Strength Data

Note: Because of their construction, the LSa seams could not be made with adhesive tapes. In the LSa seams the E-9 adhesive was manually coated onto the butyl rubber. As a result, the adhesive film thicknesses were inconsistent, and only general conclusions can be drawn about the relationships between seam strength, adhesive film thicknesses, and seam constructions.

Looking at Figure 8, the following conclusions were made:

- A majority of the seams failed above 70 lbs./linear inch proving that the E-9 seam strengths were much higher than the seam strengths produced by any other adhesive.

- In most cases, the butyl rubber failed before the four mil thick by one inch wide adhesive seams fail.

- The maximum seam strength was limited by the strength of the butyl rubber which broke at approximately 72 lbs. / linear inch.

- Sewing did not dramatically improve the seam strength. However, sewing did improve the garment assembly process and probably improves the garment's storage life.

- The adhesive stuck better to washed butyl rubber, but heat selection and adhesive thickness could minimize the difference in seam strength between washed and unwashed butyl.

- For four mil thick E-9 adhesive films, heating improved the adhesive performance. Too much heat could damage the adhesive bond strength, however.

- Again, film thickness and seam construction play a critical role in how well the adhesive performs.

FINAL ADHESIVE SOLUTION

Given the excellent performance of Worthen's E-9 adhesive, as demonstrated in Figure 8, CAR concluded that E-9 is the butyl rubber adhesive the military should use in all of its butyl rubber applications. Although aging tests have not been conducted, E-9's fully cross-linked urethane and high melt temperature should make E-9 seams impervious to all but the most extreme storage and use environments. CAR encourages the military to contact Worthen Industries and KemTek to arrange a large scale production run of E-9 on 100+ yards of washed butyl rubber. The resulting straight-cut tapes, if used in a large Quick Doff Hood production run, should verify that E-9 is easy to apply with conventional equipment, produces no hazardous fumes or waste, and can make one inch wide seams that are stronger than the Quick Doff Hood's butyl rubber.

TASK 1B - SURVEY OF THE COMMERCIAL FINDINGS MARKET

While CAR was waiting for adhesive samples in Task 1A, CAR completed Task 1B by conducting a survey of the commercial findings market to locate buckles, strapping, and other items which would improve the existing quick doff hood. The most important criteria for findings selection were, ease of use by a soldier outfitted with gloves, cost, weight, and bulk. CAR began its survey of the commercial findings market by attempting to locate newer buckle designs.

BUCKLES

Knowing that metal buckle designs existed well before the Quick Doff Hood was first developed, CAR decided to search for new buckle designs based on plastics technology. For a guide to apparel suppliers, CAR used the 1990 Suppliers Sourcing Issue published by Bobbin Media Corp., 1110 Shop Rd., P.O. Box 1986, Columbia, SC 29202, (803) 771-7500. After calling all 40 buckle suppliers listed in the Sourcing Issue, and contacting two additional suppliers recommended by the competition, CAR found only two companies who manufactured plastic buckles. All of the other companies who sold plastic buckles were distributors of the following two companies' products:

- ITW Nexus
  230 West Gerry Drive
  Wood Dale, Illinois 60191
  (708) 595-1888

- American Chord and Webbing (ACW)
  88 Century Drive
  P.O. Box 1370
  Woonsocket, RI 02895
  (401) 762-5500

DESIGN CHARACTERISTICS

Both companies manufacture a large number of plastic quick-release buckles made from acetal, polypropylene, and nylon. Every buckle is different, and each buckle has it's own unique blend of design characteristics. Some buckles are small and light weight. Some buckles are large and heavy duty. Some buckles open by pulling; others open by pushing or squeezing.

DESIGN TRADE-OFFS

To select a single buckle from the large number of buckles would require making trade-offs between buckle design characteristics. For example, a small buckle is light weight and not bulky, but a small buckle is more difficult to open with gloved hands and is not as strong as heavier buckles. Since the responsibility of weighing design trade-offs lies with the military

product designers, CAR felt that finding a listing of apparel suppliers (the Suppliers Sourcing Issue) and locating product manufacturers (versus distributors and suppliers) was the best help that CAR could provide. CAR did go one step further, however.

UNIQUE COMMERCIAL DESIGNS

After reviewing the plastic buckle selection CAR asked for samples of three unique buckles. The buckles CAR selected looked like the easiest buckles to open with gloved hands. The three buckle designs CAR obtained were:

- ITW's 118-4100-5614, black, acetal, two part buckle, for one inch wide straps (see Figure 9)

- ACW's SKR 91686, black, acetal, two part buckle, for one inch wide straps (see Figure 10)

- ACW's GM 91640, black, acetal, two part buckle, for one inch wide straps (see Figure 11)

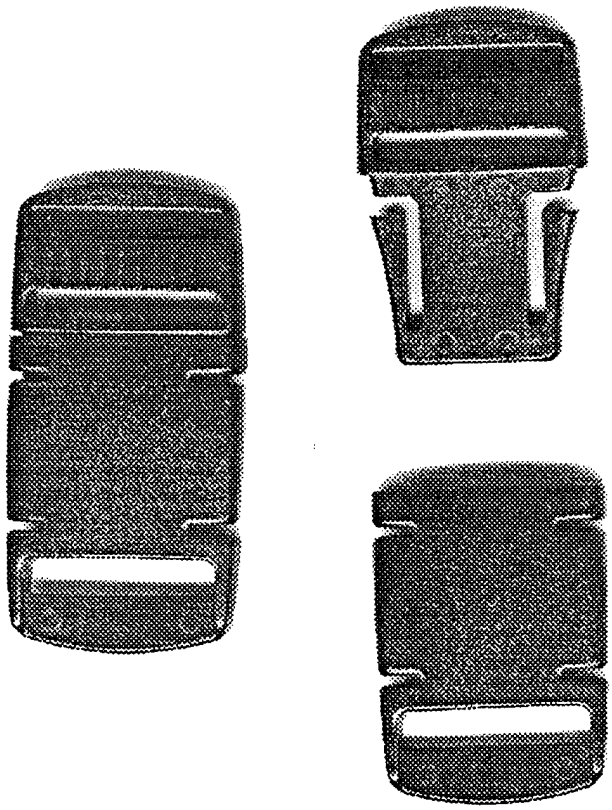

Figure 9 - ITW's 118-4100-5614 Buckle

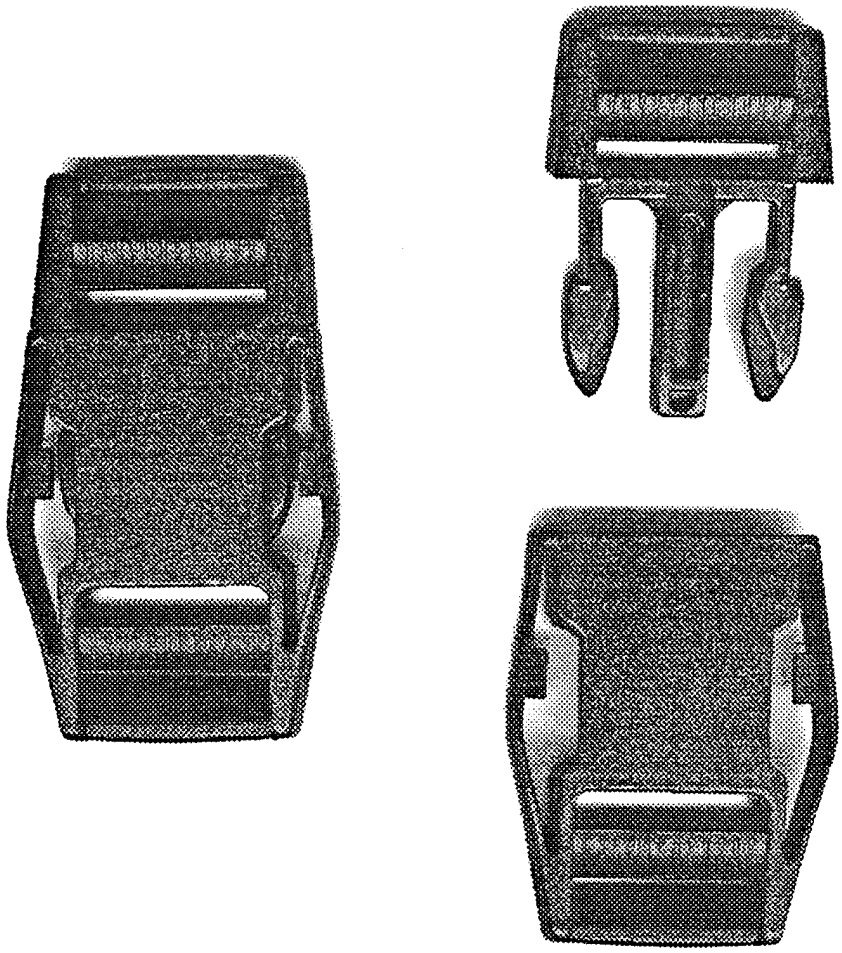

Figure 10 - ACW's SKR 91686 Buckle

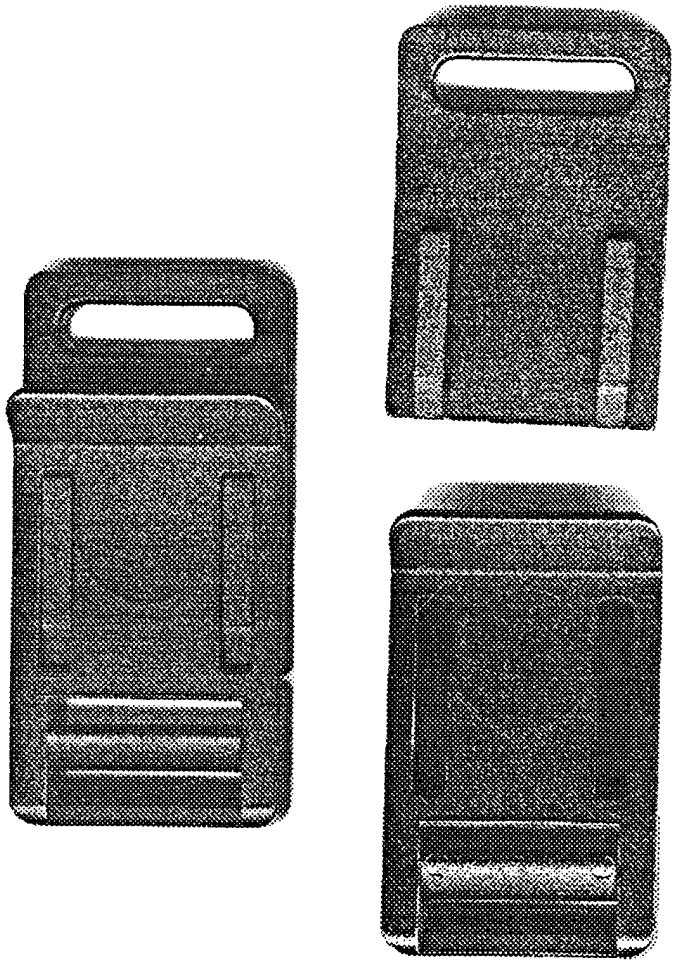

Figure 11 - ACW's 91640 Buckle

Unfortunately, only the ITW buckle is available in a size designed for smaller straps (3/4" minimum), but CAR felt that the latching devices on the three buckles was worth knowing about.

The ITW buckle releases by squeezing the sides. Unlike, other plastic buckles, there are no finger indentations on the sides of the ITW buckle. The ITW buckle can therefore be readily opened with large, heavy-gloved, hands. The same can be said for the ACW SKR-buckle which is easier to open but heavier and more bulky than the ITW buckle . The ACW GM-buckle is the only two-part buckle that opens by lifting. The lifting edge is large, but because the buckle is designed for heavy-duty use, the buckle is difficult to open, bulky, and heavy.

CUSTOM BUCKLE DESIGN MAY BE NEEDED

Although the three buckles tested were too large for the Quick Doff Hood, the buckles were the only buckles offered which could be operated with heavily gloved hands. All other commercially available plastic buckles have push-button areas designed for use with bare fingers only. If the military is interested in having a special buckle designed, however, both ITW and ACW will make custom design buckles. The military might be able to reduce the cost of custom design by arguing that the same buckle could be used in commercial applications were gloves are used to snap, unsnap, and adjust small straps. Unfortunately, CAR was not in any position to offer ITW or ACW complete buckle design specifications to be used for a cost estimate.

STRAPPING

Although CAR was unable to provide a commercial buckle solution that met the immediate needs of the Quick Doff Hood, CAR was able to come up with an improvement in the Quick Doff Hood's strapping. Having used adhesives to make adhesive based belt-loops for the Chemical Protective Suit Project, CAR quickly recognized a way to improve the Quick Doff Hood's strappings using a special piece of adhesive equipment.

The Equipment is called a TrimMaster Fuse-A-Belt machine (see Figure 12).

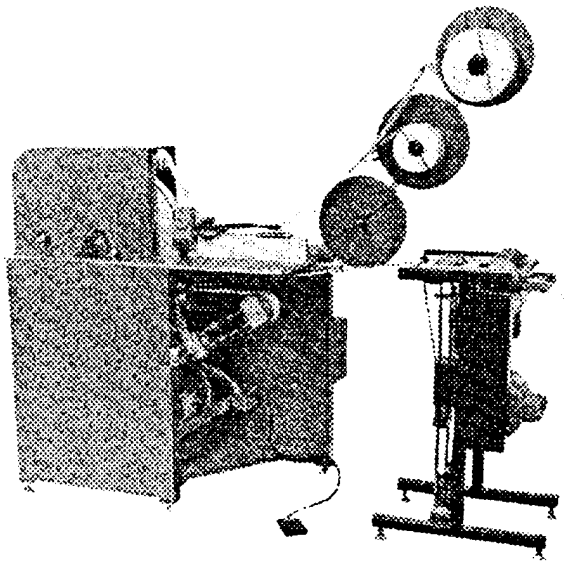

Figure 12: TrimMaster Fuse-A-Belt

The machine pulls 7/8" wide adhesive backed tapes through a folder forming a tube. The tube is then flattened by a heated drum which creases

the tube and activates the adhesive. What comes out of the machine is a flat ribbon which can be used as strapping, belt loops, and pull tabs.

Worthen E-9 backed butyl rubber (with a powder coating instead of release paper) can be used in the Fuse-A-Belt machine to produce Quick Doff Hood strapping at the rate of a foot per second. No industrial belt-loop sewing-machine can produce strapping so quickly, and the strapping is not sticky and sews nicely. Whether the strapping remains creased over long periods of time is not known, but with E-9's cross-linked urethane and high melt temperature, the strapping should survive all but the harshest storage and use environments. CAR success with the strapping stemmed from a technological advantage brought about by Worthen's E-9 adhesive, but in other findings CAR was constrained by limitations to existing technology.

OTHER FINDINGS

Having encounter many products whose selection required design trade-offs, and recognizing its own lack of authority in approving design trade-offs on behalf of the military, CAR felt that finding a guide listing most apparel suppliers (the Suppliers Sourcing Issue) was the best way to help the military intelligently evaluate the commercial findings market. CAR also recognized that the greatest help CAR could provide was in the area of butyl adhesive evaluation and development, and so, most of CAR's efforts focused on the butyl adhesives and related systems of Task 1A.

TASK 2A - MAIN QUICK DOFF HOOD SEAM CONSTRUCTION

SEWN AND TAPED SEAMS

All of the Quick Doff Hoods made by CAR were made according to the information provided on pages 29 through 30. The only major improvement to the process was the use of Worthen E-9 in place of Mace's 2451 adhesive. A detailed discussion of E-9's performance is given on pages 50 through 57.

ULTRASONICALLY WELDED AND TAPED SEAMS

As discussed on page 5, the Quick Doff Hood fabric, made according to Military Specifications, does not contain enough thermoplastic material to be weldable with any kind of welding technology. So, long before the Quick Doff Hood project began, the use of ultrasonic welding for seaming the Quick Doff Hoods was ruled out. Tests by CAR simply confirmed that the butyl fabric could not be ultrasonically welded.

TASK 2B - QUICK DOFF HOOD
ELASTIC CORD CASE CONSTRUCTION

As explained on pages 32 and 33, encasing the Quick Doff Hood elastic cord involved first seaming and sealing the left and right hood pieces together. The elastic neck cord was then bartacked at the ends. With the ends of the neck elastic bartacked in place the hoods could be turned inside-out over a simple wooden template. The template helped stretch the elastic tight. A 1-1/2" wide, straight-cut, Worthen E-9 backed, butyl, tape could then be ironed over the elastic and onto the hood. Experiments with Mace 2451 tapes not only secured the elastic with fewer parts than previous designs, but also sealed the end bartacks making the external bartack patches redundant.

TASK 2C - QUICK DOFF HOOD
ELASTIC FACE OPENING CONSTRUCTION

As explained on page 33, all attempts to secure the face elastic with adhesives failed. The only way CAR could reliably secure the face elastic was by forming a tunnel hem using a folder and a needle feed sewing machine. A portion of the tunnel hem was left open, and the elastic cord was fed through. Once the cord was through, the ends were tied, and the tunnel hem was sewn shut. The elastic pull tabs were then sewn into place.

TASK 3 - FINAL QUICK DOFF HOOD RECOMMENDATIONS

Many of the following recommendations are repeats of recommendations made earlier in this paper.

RAW MATERIAL RECOMMENDATIONS

BUTYL RUBBER

The military should re-examine its Mil. Specs. C51251 and 12189. The fact that major industry suppliers are successfully using abraded uncured butyl rubber in HAZMAT suit applications indicates that the Mil. Specs. are out of alignment with current technology. The military should also be aware of the quality problems CAR had with respect to the receipt of butyl rubber full of pin holes (**see page XXX**).

THREAD

The thread specified by the Quick Doff Hood Specification dated March 13, 1992 is not correct for the application. The silicone finish tends to migrate onto the butyl fabric and could diminish the bond strength between the butyl backed adhesive tapes and the sewn seams. The thread is also stronger than necessary. It is CAR's opinion that a cheaper, unfinished thread will perform as well and cost less than the thread currently being used. The thread CAR recommends is a cheaper unfinished, military-green, tex-35, spun polyester, sewing thread.

ADHESIVES

General Characteristics

Any butyl rubber adhesive which the military wants to use should have the following general characteristics. The adhesive should be either a paste, tape, or film adhesive. Powdered or granular adhesives can only be applied to flat stationary fabric seams. Liquid adhesives require excessive curing times, and outgas. The adhesive should cure using heat, pressure, or reactivation techniques. Time curing takes too long, and both catalyst curing and vulcanizing require exotic mixing and metering equipment. The adhesive should be elastomeric because the butyl rubber itself is an elastomer. Matching adhesive and adherend physical characteristics produces more compatible and hence more durable bonds. At the point of application the adhesive should have similar flow characteristics to hydrocarbon solvents (i.e. initial low viscosity), and the adhesive should be applied hot and under pressure to promote adhesive/adherend molecular entanglement.

Specific Characteristics

If the military chooses to tape seal the Quick Doff Hoods, the military should follow industry's lead and only use straight-cut adhesive seam tapes. In current tape-sealing equipment it is difficult to control tape tension. Unlike bias-cut seam tapes, straight-cut seam tapes do not distort when subjected to uncontrollable tension. Straight-cut seam tapes are therefore easier to handle and produce better results.

Pre-formed adhesive tapes should also have consistent adhesive film thicknesses because adhesive film thicknesses is the single most critical factor in any adhesive's performance. In CAR's experience film thicknesses around four thousandth of an inch produce strong, flexible, seams adequate for sealing the Quick Doff Hoods. Along with adhesive film thickness, the careful use of heat and pressure also improves adhesive performance, as does the use of washed butyl rubber.

Straight-cut, washed, butyl rubber, tapes coated with 4 mils of Worthen E-9 produce the strongest, cleanest, easiest to make, sealed seams without producing harmful fumes. Mace 2882 and Mace 2451 also work, but special care must be taken to ensure that the adhesive films are fully dried before the coated butyl is slit into tapes. 3M's 926 also works, but being acrylic based, 926 is, to some extent, water soluble, and 926 does not produce seam strengths as high as the Worthen and Mace adhesives.

COMMERCIAL FINDINGS

Buckles

No commercially available plastic buckles are ideally suited to the Quick Doff Hood. Both of the U.S. plastic buckle manufacturers can make custom buckles, however.

Strapping

If butyl-backed adhesive tapes are used to seal the Quick Doff Hood, the same tapes can be placed on commercial belt-loop fusing-equipment and

formed into Quick Doff Hood strapping. With the correct adhesive selection, strap-forming fusing technology is faster and more reliable than strap-forming sewing technology.

Other Findings

The best place to start a search for competitive apparel commercial findings is a current edition of Bobbin's Suppliers Sourcing Issue.

PRODUCTION EQUIPMENT RECOMMENDATIONS

SEWING MACHINES

Needle feed sewing machines equipped with Teflon coated presser feet make the sewing of the butyl rubber straight forward.

HEAT SEALING MACHINES

Commercially available tape sealing machines should be equipped (or retrofit) with a number of devices. Pre-feed devices ensure low tension adhesive-tape application. With proper design, a pre-feed device could allow bias-cut adhesive tapes to be used in Quick Doff Hood constructions. Pre-heat zones are also helpful. Pre-heating adhesives makes the removal of release-paper backings easier and makes the overall application of any adhesive faster. A final device which CAR found useful was a small feed roller to help peel release-paper backings away from some of the pressure-sensitive adhesive tapes. Adhesives that do not have release-paper backings do not require the feed roller device, however.

PROCESS RECOMMENDATIONS

SEWING RELATED

Although sewing does not improve the seam strength of Worthen E-9 adhesive seams, sewing does improve garment assembly and probably garment storage life as well. For example, the adhesive bonding of the neck elastic is easier if the left and right hood pieces are sewn and sealed first.

Unlike the neck elastic, the face elastic can not be adhesively bonded. The only way CAR could reliably secure the face elastic was by forming a tunnel hem using a folder and a needle-feed sewing machine. A portion of the tunnel hem was left open, and the elastic cord was fed through. Once the cord was through, the ends were tied, the tunnel hem was sewn shut, and the elastic pull tabs were sewn into place.

ADHESIVE RELATED

As mentioned above, the adhesive bonding of the neck elastic is easier if the left and right hood pieces are sewn and sealed first. The ends of the neck elastic can be bartacked in place, and the hoods can be turned inside-out over a simple wooden template. The template helps stretch the elastic tight. A 1-1/2" wide, straight-cut, adhesive backed, butyl tape can then be ironed over the elastic and onto the hood. Although the ironing is not necessary, the heat and pressure help ensure that the elastic is secure even though fewer parts are used. And, the heat and pressure ensure that the end bartacks are completely sealed.

Heating adhesive tapes with release-paper backings also makes removal of the release paper much easier. Turning tape sealing equipments' pre-feed zones into pre-heat zones makes the entire tape sealing process faster. Ideally, however, if no release-paper backings are present no effort is required to remove them.

For example, Worthen E-9 adhesive tapes can be prepared without release-paper backings. Instead of release paper, a special anti-stick powder can be used. The powder is part of E-9's adhesive formulation. When the E-9 tapes are heated the powder is reabsorbed into the E-9. By backing sticky adhesives with such powders several release paper problems are avoided.

The first problem is how to cope with release paper waste. Every square inch of release-paper-backed adhesive tape produces a square inch of release paper waste. If no release paper is used, no waste is generated. The second problems involves handling errors. If the release paper is removed too soon the adhesive tape may accidentally stick to the wrong surface. Exposing the sticky adhesive, or making the adhesive sticky, just before application ensures that the adhesive sticks to the correct surface thus minimizing handling errors and making adhesive bonding easier.

Although the proper use of anti-stick backings on adhesives can make adhesive bonding easier, anti-stick backings on butyl rubber make adhesive bonding more difficult. To optimize butyl-adhesive-butyl seam strengths the butyl rubber's anti-stick powder should be washed off. A single detergent-

washing of cut butyl parts in a commercial washing machine can substantially improve most adhesive-to-butyl bond strengths (E-9 seems to be an exception). However, complete powder removal does not guarantee good seam strength. Adhesive thickness also plays a significant part in determining butyl seam strength.

HOOD DESIGN CHANGE RECOMMENDATIONS

The curve on the hem of the hood is difficult to sew. Anytime a hem changes direction with respect to a fabric's grain the hem has a tendency to rope (pucker). To avoid roping, a sample pattern with a squared off hem was designed. The squared design improves the sewability of the hem and improves the hem's physical appearance.

NOTES

1. Landrock, Arthur H., Adhesive Technology Handbook, Noyes Publications, Park Ridge, New Jersey, U.S.A., 1985, pp. 126-133.

2. Landrock, Arthur H., Adhesive Technology Handbook, p. 154.

3. Interview with Dr. J. R. Aspland, Textile Color Chemist, Clemson University School of Textiles, Clemson SC, July 17, 1994.

APPENDIXES

Appendix A - 3M Pressure Sensitive, 1" Seam Grab-Strengths, Fed. Test 5100 79

Appendix B - Sticky Stuff IV 80

Appendix C - Mace 2451, 1" Seam Grab-Strengths, Fed. Test Method 5100 82

Appendix D - Mace 2451, 1" Seam Peel-Strengths, Fed. Test Method 5950 83

Appendix E - Mace 2451, 1" Seam Grab-Strengths, Fed. Test Method 5100, Application Parameters 84

Appendix F - Mace 2451, 1" Seam Grab-Strengths, Fed. Test Method 5950, Application Parameters 85

Appendix G - Worthen E-9, Fed. Test Method 5100, Test Listing 86

Appendix H - 1" Wide, 4mil Worthen E-9, Fed. Test Method 5100, Unwashed Butyl, LSp Seam Strengths 87

Appendix I - 1" Wide, 4mil Worthen E-9, Fed. Test Method 5100, Washed Butyl, LSp Seam Strengths 88

Appendix J - 1" Wide, 4mil Worthen E-9, Fed. Test Method 5100, Unwashed Butyl, LSaa Seam Strengths 89

Appendix K - 1" Wide, 4mil Worthen E-9, Fed. Test Method 5100, Washed Butyl, LSaa Seam Strengths 90

Appendix L - 1" Wide, 4mil Worthen E-9, Fed. Test Method 5100, Unwashed Butyl, Sewn Seam Strengths 91

Appendix M - 1" Wide, 4mil Worthen E-9, Fed. Test Method 5100, Washed Butyl, Sewn Seam Strengths 92

Appendix N - Adhesive Development Time-Line 93

APPENDIX A

3M Pressure Sensitive, 1" Seam Grab-Strengths, Fed. Test. 5100 (Chem. Suit Folder, Tester: Meg Watters, Test Date 2/19/92)										
Catagory	1	2	3	4	5	6	7	8	Avgerage Strength	Standard Deviation
926 Nyco - Nyco	35.0	37.5	40.0	22.5	27.5	25.2	25.1	25.9	29.8	6.63
9500 Nyco - Nyco	14.0	17.5	17.0	15.0	16.5	16.0	14.8	15.5	14.5	3.45
926 Nyco - Butyl	25.5	36.5	32.5	27.5	26.0	20.0	38.0	35.5	30.2	6.38
9500 Nyco - Butyl	14.5	16.5	16.0	16.5	20.5	17.5	17.5	16.0	16.9	1.75
926 Butyl - Butyl	62.0	60.0	42.5	50.0	49.8	59.9	45.0	64.5	54.2	8.38
9500 Butyl - Butyl	42.5	42.5	40.0	30.0	31.0	34.5	37.5	35.5	36.7	4.82

APPENDIX B

Technician: Linda Hoffman

Date: 1/23/92

Objective of Test: To test Sticky Stuff IV when bonding butyl rubber to itself.

Data:

Materials: 3 sets of tests were conducted

1) Butyl was first washed in order to remove excess talc. 2 layers of SSIV were placed on butyl and then plain piece of butyl onto sample.
2) Butyl was first washed in order to remove excess talc. 4 layers of SSIV were placed on butyl and then a plain piece of butyl was placed onto the sample.
3) 2 pieces of pre-backed butyl were placed together. Pre-backed butyl has 2 layers of SSIV on washed butyl).

All three layers were tested on the Instron in order to determine the strength of the bond.

Notes of Interest:

SSIV is extremely aggressive - difficult to precisely apply it to butyl. Pre-backed butyl was much easier to handle.

(continued on next page)

APPENDIX B - Continued

Conclusions and/or Recommendations:

Results are as follows:

2 layers - average strength of the bond was 32 lbs.

4 layers - average strength of the bond was 53 lbs.

Pre-coated - average strength of the bond was 60 lbs.

Results showed that the pre-coated butyl gives the best bond and is also the easiest to apply - Further testing is necessary.

APPENDIX C

Mace 2451 (SSIV), 1" Seam Grab-Strengths, Fed. Test. 5100
(Chem. Suit Folder, Tester: Meg Watters, Test Date 3/23/92)

Catagory	1	2	3	4	5	6	Avgerage Strength	Standard Deviation
1" Overlap Butyl - Butyl	69.0	58.0	64.0	64.0	68.0	X	64.6	4.34
1-1/4" Overlap Butyl - Butyl	68.0	62.0	49.0	63.0	66.0	66.0	62.3	6.89
1-1/2" Overlap Butyl - Butyl	56.0	58.0	65.0	64.0	69.0	65.0	62.8	4.88
1-3/4" Overlap Butyl - Butyl	62.0	52.0	64.0	60.0	63.0	64.0	60.8	4.58
2" Overlap Butyl - Butyl	62.0	59.0	66.0	63.0	44.0	53.0	57.8	8.08

Note: In all cases, the butyl rubber failed before the adhesive seams.

APPENDIX D

Mace 2451 (SSIV), 1" Seam Peel-Strengths, Fed. Test. 5950
(Chem. Suit Folder, Tester: Meg Watters, Test Date 3/23/92)

Catagory	1	2	3	4	5	6	7	8	Avgerage Strength	Standard Deviation
1" Overlap Butyl - Butyl	15	9	10.5	18.5	7.5	12	X	X	12.1	4.07
1-1/4" Overlap Butyl - Butyl	15	16	18	19	19.5	17	17.5	18	17.5	1.49
1-1/2" Overlap Butyl - Butyl	16.5	21	21	18	21	18.5	11.5	10	17.2	4.32
1-3/4" Overlap Butyl - Butyl	16.5	17	15	8	12	13.5	14.5	23.5	15	4.46
2" Overlap Butyl - Butyl	19.5	21.5	13	22.5	14.5	15.5	20	18.5	18.1	3.43

APPENDIX E

Mace 2451 (SSIV), 1" Seam Grab-Strengths, Fed. Test. 5100
(Chem. Suit Folder, Tester: Meg Watters, Test Date 04/16/92)

Catagory	1	2	3	4	5	6	Avgerage Strength	Standard Deviation
Hot Iron	45	43	40	37.5	47.5	46	43	4
No Heat	48	46	48.5	49.5	48	48	48	1
Buck Press No Heat	46	48	45	46	45	53	47	3
Buck Press 250^{0}C	44	35	29	38	31	30	35	6

APPENDIX F

Mace 2452 (SSIV), 1" Taped Seam Grab-Strengths, Fed. Test. 5950
(Chem. Suit Folder, Tester: Meg Watters, Test Date 04/16/92)

Heat / Nip Pressure	1	2	3	4	5	Avgerage Strength	Standard Deviation
No Heat / By Hand	11	11	10	11	10	10.6	0.55
No Heat / $2kg/cm^2$	11	13	13	14	X	12.8	1.26
No Heat / $4kg/cm^2$	12	12	13	12	13	12.4	0.55
No Heat / $6kg/cm^2$	14	13	14	14	14	13.8	0.45
200^0C / $6kg/cm^2$	15	15	12.5	12	13.5	13.6	1.39
400^0C / $6kg/cm^2$	12	8	10	12	8	10	2.00

Note: All seam samples made with pressure done on the Queen Light machine.

APPENDIX G

Table 1:
One Inch Wide Tape: Mil. Spec. 12189 Butyl
Adhesive: 4 mil. Worthen E-9
Coated by: KemTek
Tested Using : Fed. Test Method Std. No. 191a Method 5100*

Substrate Material	Seam Type	No. of Samples	Heat Sealing Parameters
Unwashed Light Weight Butyl	LSa	5	Cold press, hand pressure
	LSa	5	Heat press @ 190°C for 5 Seconds
	LSa	5	Heat press @ 190°C for 10 Seconds
	LSa	5	Cold press, hand pressure, specimens kept in Freezer for 1 week before testing
Washed Light Weight Butyl	LSa	5	Cold press, hand pressure
	LSa	5	Heat press @ 190°C for 5 Seconds
	LSa	5	Heat press @ 190°C for 10 Seconds
	LSa	5	Cold press, hand pressure, specimens kept in Freezer for 1 week before testing

* Test specimens were 6" x 3" with a 1" overlap. These are the only aspects of Mr. Nagaraja's tests that were not in accordance with Fed. Test Method 5100.

APPENDIX H

Table 2:
One Inch Wide Tape: Mil. Spec. 12189 Butyl, Non-bias Cut
Adhesive: 4 mil. Worthen E-9
Coated by: KemTek
Tested Using : Fed. Test Method Std. No. 191a Method 5100*
Material Being Seamed: Unwashed Mil. Spec. 12189 Butyl
Seam: LSp

	Specimen #	Max. Breaking Load (lbs)	Failure of Bond	Avg. Breaking Load (lbs)
Cold press, hand pressure	1	32	Seam Failed	29
	2	26		
	3	28		
	4	28		
	5	29		
Heat press 5 seconds	1	80	Butyl Failed	71
	2	73	"	
	3	53	Seam Failed	
	4	78	Butyl Failed	
	5	71	"	
Heat press 10 seconds	1	54	Seam Failed	60
	2	58	"	
	3	64	Butyl Failed	
	4	64	"	
	5	61	"	
Cold press, hand pressure after 1 week Freezing	1			
	2			
	3			
	4			
	5			

* Test specimens were 6" x 3" with a 1" overlap. This is not in accordance with Fed. Test Method 5100, but parallels Mr. Nagaraja's initial experiments.

APPENDIX I

Table 3:
One Inch Wide Tape: Mil. Spec. 12189 Butyl, Non-bias Cut
Adhesive: 4 mil. Worthen E-9
Coated by: KemTek
Tested Using : Fed. Test Method Std. No. 191a Method 5100*
Material Being Seamed: Washed Mil. Spec. 12189 Butyl
Seam: LSp

	Specimen #	Max. Breaking Load (lbs)	Failure of Bond	Avg. Breaking Load (lbs)
Cold press, hand pressure	1	37	Seam Failed	41
	2	36		
	3	46		
	4	41		
	5	43		
Heat press 5 seconds	1	71	Seam Failed	71
	2	70	Butyl Failed	
	3	75	"	
	4	72	"	
	5	69	Seam Failed	
Heat press 10 seconds	1	69	Butyl Failed	73
	2	77	"	
	3	76	"	
	4	69	Seam Failed	
	5	73	Butyl Failed	
Cold press, hand pressure after 1 week Freezing	1			
	2			
	3			
	4			
	5			

* Test specimens were 6" x 3" with a 1" overlap. This is not in accordance with Fed. Test Method 5100, but parallels Mr. Nagaraja's initial experiments.

APPENDIX J

Table 4:
One Inch Wide Tape: Mil. Spec. 12189 Butyl, Non-bias Cut
Adhesive: 4 mil. Worthen E-9
Coated by: KemTek
Tested Using : Fed. Test Method Std. No. 191a Method 5100*
Material Being Seamed: Unwashed Mil. Spec. 12189 Butyl
Seam: LSaa

	Specimen #	Max. Breaking Load (lbs)	Failure of Bond	Avg. Breaking Load (lbs)
Cold press, hand pressure	1	22	Seam Failed	22
	2	26		
	3	23		
	4	18		
	5	23		
Heat press 5 seconds	1	59	Seam Failed	57
	2	57		
	3	52		
	4	57		
	5	58		
Heat press 10 seconds	1	76	Butyl Failed	73
	2	74	"	
	3	68	Seam Failed	
	4	77	Butyl Failed	
	5	71	"	
Cold press, hand pressure after 1 week Freezing	1			
	2			
	3			
	4			
	5			

* Test specimens were 6" x 3" with a 1" overlap. This is not in accordance with Fed. Test Method 5100, but parallels Mr. Nagaraja's initial experiments.

APPENDIX K

Table 5:
One Inch Wide Tape: Mil. Spec. 12189 Butyl, Non-bias Cut
Adhesive: 4 mil. Worthen E-9
Coated by: KemTek
Tested Using : Fed. Test Method Std. No. 191a Method 5100*
Material Being Seamed: Washed Mil. Spec. 12189 Butyl
Seam: LSaa

	Specimen #	Max. Breaking Load (lbs)	Failure of Bond	Avg. Breaking Load (lbs)
Cold press, hand pressure	1	32	Seam Failed	36
	2	35		
	3	35		
	4	34		
	5	42		
Heat press 5 seconds	1	74	Butyl Failed	72
	2	73	"	
	3	68	Seam Failed	
	4	72	Butyl Failed	
	5	75	"	
Heat press 10 seconds	1	76	Butyl Failed	74
	2	80	"	
	3	70	Seam Failed	
	4	73	Butyl Failed	
	5	71	"	
Cold press, hand pressure after 1 week Freezing	1			
	2			
	3			
	4			
	5			

* Test specimens were 6" x 3" with a 1" overlap. This is not in accordance with Fed. Test Method 5100, but parallels Mr. Nagaraja's initial experiments.

APPENDIX L

Table 6:
One Inch Wide Tape: Mil. Spec. 12189 Butyl, Non-bias Cut
Adhesive: 4 mil. Worthen E-9
Coated by: KemTek
Tested Using : Fed. Test Method Std. No. 191a Method 5100*
Material Being Seamed: Unwashed Mil. Spec. 12189 Butyl
Seam: Sewn and Taped to Quick Doff Hood Specifications

	Specimen #	Max. Breaking Load (lbs)	Failure of Bond	Avg. Breaking Load (lbs)
Cold press, hand pressure	1	49	Butyl Failed At Seam	46
	2	44		
	3	47		
	4	46		
	5	46		
Heat press 5 seconds	1	77	Butyl Failed	77
	2	77		
	3	74		
	4	76		
	5	79		
Heat press 10 seconds	1	79	Butyl Failed	78
	2	77		
	3	74		
	4	80		
	5	78		
Cold press, hand pressure after 1 week Freezing	1			
	2			
	3			
	4			
	5			

* Test specimens were 6" x 3" with a 1" overlap. This is not in accordance with Fed. Test Method 5100, but parallels Mr. Nagaraja's initial experiments.

APPENDIX M

Table 7:
One Inch Wide Tape: Mil. Spec. 12189 Butyl, Non-bias Cut
Adhesive: 4 mil. Worthen E-9
Coated by: KemTek
Tested Using : Fed. Test Method Std. No. 191a Method 5100*
Material Being Seamed: Washed Mil. Spec. 12189 Butyl
Seam: Sewn and Taped to Quick Doff Hood Specifications

	Specimen #	Max. Breaking Load (lbs)	Failure of Bond	Avg. Breaking Load (lbs)
Cold press, hand pressure	1	59	Butyl Failed At Seam	55
	2	53		
	3	55		
	4	55		
	5	54		
Heat press 5 seconds	1	71	Butyl Failed	73
	2	72		
	3	74		
	4	74		
	5	72		
Heat press 10 seconds	1	70	Butyl Failed	71
	2	69		
	3	74		
	4	69		
	5	73		
Cold press, hand pressure after 1 week Freezing	1			
	2			
	3			
	4			
	5			

* Test specimens were 6" x 3" with a 1" overlap. This is not in accordance with Fed. Test Method 5100, but parallels Mr. Nagaraja's initial experiments.

APPENDIX N

Adhesive Evaluation Timeline			1992											1993												
			Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan
Adhesives	Pressure Sensitives	ATG	X																							
		SS I	X																							
		SS II	X																							
		SS III	X																							
		SS IV	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	X						
	Hot Melts	SP 28	X																							
		ElectroSeal	X																							
		Puddle Coat			–	–	–	–	–	–	X															
		E-9																	–	–	–	–	–	–	–	X
		Application Parameters		√		√		√							√				√				√	√	√	
Adherends		Fabric Parameters							√	√			√													
		Seam Preparation					√						√										√	√	√	
		Seam Construction																					√	√	√	