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A Study of the Variables Affecting the Heat Seal Strength and Hot Tack of Ethylene-Vinyl Acetate Heat Sealant Copolymers

By

John Williams Spink

THESIS

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ABSTRACT

A Study of the Variables Affecting Heat Seal Strength and Hot Tack of Ethylene-Vinyl Acetate Heat Sealant Copolymers

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Heat seal parameters and their correlation with the physical and molecular properties of a series of ethylene-vinyl acetate copolymers of varying percent vinyl acetate were investigated. Seal temperature, dwell time, and pressure were statistically evaluated to determine the conditions affecting maximum seal strength and hot tack for these films. Evaluation of heat seal parameters was based on a statistically designed experiment, and was carried out on a laboratory Heat Sealer with Hot Tack Attachment. For each seal, the seal strength was evaluated by tensile stress and weighted balance technique, respectively.

Properties of the test copolymers were determined and linear regression analysis used to describe their relationship at maximum heat seal strength and hot tack. The film composition, melt temperature, and viscosity average molecular mass were found to be statistically significant parameters with respect to heat seal strength and hot tack.

DEDICATION

This thesis is dedicated to my parents, sister, grandmothers and great aunts who encouraged and supported my graduate education.

They instilled in me a desire to learn and raise myself to a higher level. They instilled in me the desire to fulfill my high self-expectations. They instill in me the idea that I can do anything.

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...and my close and special friends who kept me "in-bounds."

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1.0 INTRODUCTION

Heat sealing characteristics of a polymer are associated with fundamental properties such as composition, molecular weight, molecular weight distribution and percent crystallinity. Polyethylene and its copolymers are among the most commonly used materials for heat sealing purposes (Ryan,1949).

For example, the combination of high clarity, puncture resistance, impact strength and low heat sealing temperature makes ethylene-vinyl acetate copolymers (EVA) very desirable materials for flexible packaging. EVA is frequently specified as the extrusion coating on polypropylene, polyester and aluminum foil, to provide heat seals at high converting rates. Typical applications include frozen food, dry soup, soft drinks, dry mix pouches, and food pouches (Duncan,1988).

In addition to low density polyethylene and ethylene/ vinyl acetate copolymers (EVA), Surlyn iononomer resins are also very commonly used heat sealant polymers. The term "ionomer" denotes a thermoplastic containing both covalent and ionic bonds. The ionic inter-chain electrostatic forces are quite strong, and to an appreciable extent, are responsible for the unique spectrum of properties characteristic of ionomeric resins (Young,1968).

The heat sealing properties of polyethylene are very

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sensitive to physical and molecular properties, especially the degree of crystallinity, molecular weight, molecular weight distribution, density, and composition (Ryan,1949). This point is illustrated by comparing the melt profile of low, medium and high density polyethylene. High density polyethylene (HDPE) melts with a high and very sharp melting point, which narrows the useful range of heat sealing temperatures. Thus, the heat sealing range is rather narrow. Medium density polyethylene (MDPE) has a wider melting range, but is still high in percent crystallinity, and has a melting point approximately as high as HDPE. These factors contribute to reducing the usefulness of MDPE for heatsealing.

In contrast, low density polyethylene (LDPE) melts over a very wide range, and produces a melt endotherm which is very broad, lower in area, and which melts at a much lower temperature than either HDPE or MDPE. The practical significance is optimum seal conditions, resulting in a strong seal over a broad temperature range. These features, namely low melt initiation temperature and broad melt range,make LDPE a better choice as a heat sealing material.

In addition to seal initiation temperature and seal range, hot tack is also an important property of a heat sealant polymer, since the seal area is often still under stress after sealing in many form/fill/seal applications. Hot tack is defined as the melt strength of a heat seal when the sealing bar pressure has been removed and the seal is still

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molten and under tension, and must resist the stress caused by the weight of the packaged product (Szemplenski,1989). The amount of tack is estimated as the resistance when separating the adherents, while the adhesive or heat seal layer still exhibits viscous or plastic flow. The separation is affected without failure or deformation occurring in the adhered surroundings.

Most commonly, the major noted seal strength measurement has been the cold seal strength, or dynamic seal strength. This cold seal strength is defined as the dynamic strength of a heat seal when placed under tensile stress, after the seal temperature has cooled to ambient conditions. The seal is strained until failure (that is until the film breaks). The maximum load is recorded, and defined as the maximum seal strength. The nature of typical bond failure has been studied (Theller,1988).

The mechanical strength of a heat seal, as affected by the sorption of flavor components of a product, is also of major concern in selecting a heat sealant polymer (Harte,1990).

As indicated above, polymer processability or flow properties are dependent upon the average molecular weight and molecular weight distribution of the sealant polymer. For example, heat seal temperatures generally increase with an increase in polymer molecular weight, while the seal range decreases with increasing molecular weight. Thus, the broadly

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distributed polymer exhibits a broad seal range and provides a wider temperature range over which good heat seals can be affected (Theller,1988).

The three variables; temperature, pressure, and dwell time, control the strength of the heat seal being formed (Sherman,1976). Each of the variables has upper and lower limits. The temperature causes the interfaces between the two surfaces to disappear, the pressure assures intimate contact of the interfaces, and the dwell time brings the interfaces to the needed sealing temperature, which allows time for cooling with the surfaces held together (Ryan,1949).

The upper boundary of the temperature range is limited by damage to the material caused by heat degradation. The lower temperature boundary is limited by the amount of heat necessary to melt the interface between the two surfaces being heat sealed (Ryan,1949). To form a good heat seal, the temperature must be high enough to melt the interface between the two polymer layers, and low enough to prevent their thermal degradation.

Pressure is the second variable in the formation of a heat seal and, like temperature, pressure also has upper and lower limits. The upper limit is present because too high a pressure in the heat seal jaws will thin out the heat sealant polymer layers during formation of the heat seal. A pressure that is very high will extrude the polymer from between the heat sealing jaws (Young,1968). The lower limit of the pressure range is the minimum pressure necessary to assure intimate contact between the heat sealant material interfaces. The bond strength increases and then decreases as pressure is increased, indicating the presence of an optimum range. This supports the previous statement, that the pressure must be high enough to assure intimate contact of the interfaces, yet low enough to prevent a thinning out of the heat sealant polymer layers at the heat seal area.

It has been proposed and generally accepted, that an increase in pressure for "soft" materials like LDPE has little affect on the increase of intimate contact and the associated increase in seal strength (Theller,1988).

The third variable, dwell time, also has upper and lower limits. Miltz (1980) observed that the heat seal strength for polyethylene passes through a maximum as dwell time increased for all sets of temperature and pressure conditions. Insufficient dwell time formed weak heat seals,as did too long a dwell time.

In general, it is thought that an increase in dwell time, beyond an optimum point, has little affect on an increase in heat seal strength. The minimum dwell time (especially on form/fill/seal machines) within this optimum range, is the point used in production. Because of economic considerations, line speeds must be run as fast as possible (James River Corp.,1988).

Thus, the three variables; temperature, pressure and dwell time, are interrelated. The broadest optimum temperature range in forming a strong heat seal occurs at high temperature, short dwell times and low pressures.

Based on the complex relationship between polymer properties of density, percent crystallinity, molecular weight average and molecular weight distribution, and the heat seal characteristics, there is a need to investigate correlations between optimum heat seal parameters and fundamental properties of the heat sealant polymer.

The present study will focus specifically on optimizing heat seal and hot tack parameters for a series of ethylene-vinyl acetate heat sealant polymers, and the development of correlations between the bulk and molecular properties of the polymer structures, to include: chemical composition, density, melt flow index, molecular weight and molecular weight distribution, melt temperature profile, and mechanical properties.

Heat transfer of the sealing bars by theoretical calculation and physical test, with evaluation of pre-melting transitions and relaxations, was investigated.

Heat sensitive waxes were used to determine the interface temperatures achieved during the heat sealing process, employed specifically in this research, with this equipment.

In terms of practical significance, these studies should provide a potential method for predicting the heat seal behavior of EVA copolymers and optimum seal parameters, as a function of the known physical and molecular properties of the copolymer. This correlation will be facilitated by an understanding of selected physical and molecular properties of the heat sealant polymers and their heat sealing characteristics, to include both hot tack and cold seal strength.

2.0 LITERATURE REVIEW

[Low density polyethylene] ...is generally the softest and least crystalline of the polyethylenes, and is widely used in applications requiring clarity, inertness, processing ease, sealability, moisture barrier, and good electrical resistance (Kaus,1963).

LDPE producers directly control resin density and melt index (MI -- a measure of resin viscosity at a given rate of shear). The molecular weight distribution (MWD) is controlled to a lesser extent. An increase in density increases crystallinity, stiffness, tensile strength, hardness, melt temperature, shrinkage and chemical resistance (Kaus,1963).

An increase in melt index improves flowability, clarity, draw down, but reduces melt strength of heat seals and hot tack (Kaus,1963).

Ethylene-vinyl acetate copolymers are derived from basic low density polyethylene technology. One of the first uses of vinyl acetate as a modifier for ethylene was to reduce the splittiness [brittleness] of high clarity film resins. Typical applications include frozen food, dry soup, and soft drink mix pouches; cheese wrap; and pet food pouches (Duncan,1988).

The heat sealability of a packaging film is one of the most important properties when considering its use on wrapping or bag making equipment and, of course, the integrity of the seal is also of tremendous importance to the ultimate package. The heat sealability of a film has to be considered in relation to many other factors, including the available pressure, the dwell time, temperature and the rate of heat transfer of the sealing bars. Any test of the heat seal qualities of a particular film must, therefore, simulate the conditions under which the film will be used as closely as possible (Briston,1989).

Heat seals can be made by a number of different methods, apart from the straightforward application of heat to the layers of film. Seals

> can be made, for instance, by high frequency heating (as with PVC) or by ultrasonic welding. Regardless of how the seal is made, the strength is determined by measuring the force required to pull apart the two film layers which have been sealed together. The force can be applied in such a way as to cause the seal to fail in shear or in peel. If other factors, such as the temperature, dwell time, and pressure used in making the seal are equal, then peeling a seal will give a lower figure than shearing it. Seals are normally tested in peel, therefore, as this is the way they are likely to fail in practice (Briston,1989).

3.01 Seal Failure

An important consideration in the formation of heat seals, is the failure of the system. "The force required to pull open a seam is of obvious relevance to the integrity of a package" (ASTM F88,1985). As noted above, when part of the seal system fails, the package fails. However, many times this failure is not within the actual seal. Therefore, the effect of thermo-mechanical influences on seal strength warrants investigation, since many of these effects can lead to weakening of the regions adjacent to the immediate seal area. When two materials are heat sealed together, the seal can be described in terms of three regions, namely: (i) the seal itself; (ii) the region adjacent to the seal; and (iii) the unaffected film region far from the seal interface.

> Interfacial and cohesive failure.
>
> [For instance] It is not possible to get true hot tack values on uncoated materials such as unlaminated polyethylene film. Instead of "peeling," the seal as is intended for hot tack, one often gets a "break" in the seal. In order to achieve a peeling effect, it is necessary to first cover the film with non-thermoplastic tape, such as ordinary adhesive tape which is often based on

> polyester. The procedure is to cover the plastic film with tape first and then carefully cut the test pieces in the cutting apparatus into 15mm strips. The strips are then mounted in the normal way with the polyester nearest the sealing bars. One may always use the same tape quality in order to get reproducibility (DTC,1988).

3.01.01 Molecular Mechanisms of Heatsealing

The following section dealing with the molecular mechanism of heat sealing is taken from Theller (1989), and represents in large part the views of Voyutskii (1963).

> Voyutskii (1963) explains autohesion from the standpoint of the theory of diffusion of free and mobile ends of macromolecules on the surface of high polymers. The term autohesion, or self-adhesion, refers to the ability of two surfaces of the same material in contact with each other to form a strong bond.
>
> As the interface temperature of the two layers of material being heatsealed is increased from ambient, seals of significant strength begin to appear considerably below the melting point of the heatseal layers. How far below the melting point depends on the type of polymer. In the case of LDPE the onset of detectable heatsealing is 15-20 degrees Kelvin below the melting range.
>
> From the first appearance of measurable seal strength to a temperature almost to the melting point, bonds are formed that, when tested, fail at the seal interface --they peel apart. A peel seal is not necessarily a weak seal. Peel strengths range from just above zero up to the strength of a fused seal.
>
> The ultimate force that holds heatseals together is the same force that binds the chain molecules in the polymer structure itself: secondary valence forces. For these forces to come into play in the heatsealing process, enough movement and deformation of the surface polymer chains must take place for the molecules in the two surfaces to move into intimate contact with each other. The atomic groups on chains in the adjacent surfaces must move to within about 5 Angstrom units of each other -- a primary prerequisite for secondary valence bonds to form.

The bonds form instantaneously if the molecules have sufficient energy and the proximity condition is met.

On a molecular scale the smooth surfaces of heatseal layers are of course not smooth, but very rough. Most polymers used in heatseal layers have a high amorphous content by design, and can be expected to have surfaces exhibiting clumps of tangled disordered chains and chain ends. The surface molecular order should never the less be somewhat greater than that of the bulk structure, due to the tendency of chains to lie parallel to the air interface.

The first thing that must take place in the heatsealing process is to flatten the webs to bring the two surfaces into molecular contact over as high a percentage of the total area as possible. As the interface temperature is increased from the toe of the strength versus temperature curve, chain ends up to 20-30 carbon atoms in length diffuse across the original interface into amorphous areas of the opposite layer. This results in a shallow zone where the structure of the bulk polymer is approximated through the interweaving of chain segments. This results in much greater seal strength than bond formation alone.

Voyutskii applied the thermodynamic equation,

$$\Delta F = \Delta U - (T * \Delta S) \tag{eq. 1}$$

where F is the free energy; U is the internal energy; T is the absolute temperature; S is entropy). Applied to autohesion, it predicts that the decrease in free energy of the system -- a measure of seal strength --comes from the decrease in internal energy, due to bond formation, and the increase in entropy associated with the restoration of a zone of random structure at the original interface. Voyutskii states that "a decrease of the internal energy of the system, which ensues instantly after the contact is made, and after the polymer/air interface disappears, plays only a minor role in autohesion." He believes the entropy factor stemming from the intermingling of chain segments due to their thermal motion (microbrownian movement) is by far the overriding effect.

Increasing vigor of microbrownian movement with temperature, and the consequent increased depth of the zone of intermingling could explain the rapid increase of seal strength [in the region where the seal failure changes from peel, to break]. Heatseals in this portion of the curve are still peel seals. Inspection of the sealed surfaces after peeling reveals that much of the area appears to retain its shiny appearance, but with many rough spots where significant diffusion and sealing apparently took place --the areas of molecular contact of the original surfaces.

In order for chain ends from each surface to diffuse readily into the structure of the opposite surface, the receiving surface must already have, or must develop, holes and voids in its structure to accept incoming chain segments. Obviously an amorphous material fits this picture better than a crystalline one. An increase in temperature causes thermal expansion of the structure and a decrease in density due to additional voids and holes opening up. There is then increased probability that a chain segment with the necessary activation energy will be located so it can move into a hole and assume a new equilibrium position.

As the temperature climbs to the melting point of the polymer, to quote Voyutskii again, "a larger and larger number of segments of the individual macromolecule, with ever increasing energy values will participate in self-diffusion. Finally, at a definite temperature, at a certain moment the sum of activation energy values of individual segments would exceed the energy barrier of the macromolecules." At this point the polymer mass as a whole is capable of flow, and a merging of larger chain segments from the two layers can take place --to the extent that the seal becomes fused and it no longer peels apart. The test strip breaks elsewhere (usually at the edge of the seal)(Theller,1988).

3.01.01 Flow Properties

As a polymer mass is being heated and placed under compression stress by the sealing bars, the polymer experiences viscoelastic flow.

The flow properties of polymer melts are, to say the least, complex. This is only to be expected when one is trying to deform entangled long chain molecules of a distribution of molecular weights. During flow, stresses imposed on the molecules will cause them partly to uncoil and possibly also to roll over and over as they travel down the melt stream. When imposed stresses are released there will be a tendency to re-coil. Furthermore, when convergent flow occurs, as in many processing operations, significant tensile deformation occurs in addition to the shear deformations normally considered in simple analyses (Brydson,1989).

3.01.03 Melt Viscosity

The melt viscosity also is a factor concerning thermal seal properties of polymers.

The melt viscosity of a polymer at a given temperature is a measure of the rate at which chains can move relative to each other. This will be controlled by the ease of rotation about the backbone bonds, i.e. the chain flexibility, and on the degree of entanglement. Because of their low chain flexibility, polymers such as polytetrafluoroethylene, the aromatic polyimides, the aromatic polycaronates and to a less extent poly(vinyl chloride) and poly(methyl methacrylate) are highly viscous in their melting range as compared with polyethylene and polystyrene.

For a specific polymer the melt viscosity is considerably dependent on the (weight average) molecular weight. The higher the molecular weight the greater the entanglements and the greater the melt viscosity. Natural rubber and certain poly(methyl methacrylate) products, which have molecular weights of the order of 10^6, cannot be melt processed until the chains have been broken down into smaller units by masication processes. Chain branching also has an effect. In the case of polyethylene and the silicones the greater the branching, at constant weight average molecular weight, the lower the melt viscosity (Brydson,1989).

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3.01.04 Other Seal Problem Factors

Laminates, have some special problem traits, especially with polyethylene as a layer. For example, the interface region (between seal and unaffected film) of a seal, may exhibit some stress due to shrinkage of the heated film in the seal (Briston,1989).

> Orientation and shrinkage:
> As previously stated molecular orientation occurs during melt processing of polymers. On removal of the deforming stresses the molecules start to coil up again but the process may not go to equilibrium before the polymer cools to below its Tg [glass transition temperature]. This can lead to planes of weakness and easy fracture when subject to shock (impact) stresses (Briston,1989).
>
> A second persistent processing problem is that of shrinkage. In the case of amorphous polymers the shrinkage is very small and is about 0.005 cm/cm.
> With crystalline polymers the more orderly molecular packing leads to much greater shrinkage (Briston,1989).
>
> There is another difference between polyethylene films and regenerated cellulose film [other types of polymer films] which adds to sealing difficulties, however. The latter has an infusible substrate and only the coatings melt when heat sealing is carried out. Polyethylene film is completely fusible and so there is a danger of loss of strength because the sealing pressure causes flowing and thinning of the seal. In addition, the seal is still molten when the jaws are opened and the seal is left unsupported so that there is a danger of the seal being ruptured (Briston,1989).
>
> When heat sealing laminated materials, it is generally preferable to use heated jaw sealers since laminates do not suffer from the disadvantages of single, fully fusible films. It is possible, therefore, to make use of the high speeds of the continuous heating method of sealing and embossed jaws can be used to give extra strength.

> In general, a good heat seal, using heated jaw or impulse sealers depends on the temperature at the interface, contact or dwell time, pressure between the jaws and the nature of the film.
>
> In the particular case of low density polyethylene film used on form/fill/seal sachet making equipment, the speed of cooling and the strength of the molten polymer are important factors. In the early days of such equipment, the filling speed was the rate determining factor but with increasing filling speeds, the limit is now set by the speed of sealing (Briston,1989).

The seal process, with thermal and mechanical effects to different seal regions, warrant stress considerations as noted above.

> Stress concentrations:
>
> However, if both the adhesive and substrates [or sealed films] are flexible, as is usually the case in a peel test, the mechanical properties of both play an important part. These properties may, however, differ considerably from those of the bulk materials due to stress concentrations, especially at or near the interface [of the seal]. These stress concentrations can be due to various factors, e.g. flaws or differences in thermal expansion between adhesive and substrate. A flaw will develop when local stress exceeds local strength. In other words, the strength of a joint is dependent upon the strength of the weakest region.
>
> If stress concentrations are reduced, then higher values are achieved and this effect is perhaps most marked in peeling tests. Peeling tests are more important when flexible substrates such as films are involved, whereas shear and direct tension tests are more important with rigid substrates, such as molded plastic parts. It is clearly desirable that published bond strengths should be accompanied by details of all the experimental conditions (Jenkins,1972).

3.02 Heat Seal Optimization Studies

The studies and procedures described in the literature dealing with optimization of heat seal parameters, are quite varied in scope and objectives.

3.02.01 Heat Seals

For example Miltz (1980) studied the effects of polymer structure on the heat seal properties and seal strength of low density polyethylene (LDPE), by varying the heat seal parameters of temperature, dwell time, and pressure, employing an impulse heat sealer.

Miltz (1980) evaluated three temperature/pressure levels, and four dwell times for optimization of heat seal parameters. The resulting data was presented graphically on a two-dimensional, series of curves, where seal strength was plotted as a function of dwell time (sec). Each of the parameters of temperature, pressure, and dwell time, where found to have upper and lower limits. The lower boundary of the temperature parameter, is the minimum amount of energy needed to begin melting of the polymer. The upper boundary of the temperature range describes the onset temperature for thermal degradation of the film.

The lower pressure boundary is the minimum pressure necessary to achieve intimate contact of the heat seal layers. The upper pressure limit, combined with temperature and time, can result in the thinning out of the seal. The upper and lower pressure levels considered by Miltz were 20 and 30 pounds per square inch, respectively.

The dwell times used by Miltz were 500, 750, 1000 and 1250 milliseconds(ms), respectively. Miltz evaluated the effect of dwell time on heat seal strength, at three sets of temperature/pressure conditions, and concluded that on an impulse heat sealer, at these specific conditions of test, the effect of dwell time on seal strength, increases to a maximum. That is, the seal strength passes through a maximum as the dwell time is increased from 500 ms to 1250 ms.

In a later study, Harder (1982) evaluated the effects of temperature and dwell time on the optimization of polyethylene heat seal strength.

This study was carried out on a commercial Form/Fill/Seal Machine, where the voltage was controlled by a step down from 220 volts. The pressure is exerted by a rubber strip compressing the film against the sealing wire, and the dwell time is controlled by a cam shaft that mechanically controls a seal activator button.

The studies reported by Harder employed two sets of experimental conditions in the test design. The first set of test conditions were the conditions used commercially. The second set of conditions, were selected, based on literature values reported for heat seal optimization of polyethylene, which employed a lower temperature and longer dwell time than was used commercially.

In his studies, Harder evaluated the impulse heat seals of a polyethylene milk pouch formed on a form/fill/seal machine. Under commercial conditions, five percent of the production run failed, and resulted in "leakers" (pouches in which a liquid product leaked).

Harder's studies included evaluation of the effect of the heat seal parameters on seal strength, as well as developing methods for optimizing seal strength. Theoretical considerations were combined with the mechanics of the commercial machine, and user requirements such as production considerations or line speed.

By varying machine parameters, Harder successfully increased the seal strength of the polyethylene pouches.

In a recent study, Berger (1989) examined the effect of sealing conditions and die configurations on polymer heat seals, and described methods of viewing and analyzing the data. These studies were carried out on a wide range of copolymers and laminates.

The testing utilized a laboratory sealer with multiple sealing dies.

In the study, Berger varied dwell time, seal pressure and seal temperature. Each parameter had three levels arranged into a high/ low/ mid-point, response surface experiment. This study evaluated six films, and five seal die designs, and the resulting seal strength and seal distortion values were presented graphically.

The heat seal pressures used by Berger were 200, 500, and 800 pounds, with flat dies of 1/4" x 5 1/2". This corresponds to pressures of 146, 364, and 582 pounds per square inch respectively. The other die configurations exhibited different surface areas, and thus, different pressure values.

The dwell times uses were 100, 500, and 900 milliseconds (ms), respectively.

The temperatures evaluated were selected for each individual packaging material, to ensure measurable seal strength data was obtained at the extreme sealing conditions. This was a necessary requirement for a response surface experiment (Berger,1989).

The test generated an average of nine replicates at each condition, and was designed to determine, if possible, the maximum seal strength trends in relation to die configuration. The author concluded that, "a response surface style of experiment can allow controlled tests of material and seal die designs (Berger,1989)."

From these studies, Berger further concluded that the response surface style experiment is a valid design for examining controlled tests of materials. The author also concluded that the effects of heat seal parameters are: temperature -- major positive effect, dwell time -- slightly negative effect; and pressure -- very little effect.

Theller (1989) also described the results of studies involving the heatsealability of flexible web materials in hot-bar sealing application.

The principle objective of Theller's study was to determine which factors in the heat sealing process significantly affect seal strength, and should be included when testing for heatsealability (Theller,1988). The parameters examined were seal die configuration, sealing die temperature, sealing die pressure on the film, and sealing die dwell time during sealing.

The equipment used was a laboratory heatsealer from Precision Instruments West.

The study was designed as a pilot test to observe trends and curve shapes of the various parameters, where seal strength is plotted as a function of the respective parameters.

In practice, the test parameter was increased from a minimum value to a maximum value, in incremental steps. This sometimes resulted in a great number of points (up to 50 graph points) with the high end coinciding with a plateau, or maximum seal strength.

In this study, no specific set of temperatures or preset points, such as onset temperature of melt temperature were used. The test conditions were increased until failure resulted.

The dwell times used were also chosen by seal strength responses. That is, the dwell time was increased at 100 ms increments, until no increase in seal strength was observed for an increase in dwell time.

The pressure was expected to be insignificant beyond a minimum point, with values of 40 psi and 400 psi being employed.

The die configurations were quite varied, from a flat die to

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Since this was a pilot style test, the number of replicates was 2-3, depending upon the difference between the first two replicates.

For a seal to form, according the Theller, "the seal interface must reach the activation temperature level for diffusion of chain ends," and "microbrownian movement of chain ends must occur." The minimum dwell time required to attain such a condition, in terms of application to commercial equipment, is the most desirable. The data shows that dwell times of about 260-410 ms allow chain-end diffusion at the interface.

A minimum pressure is needed to flatten out the films "microroughness" so as to increase the number of diffusion sites leading to an increase in seal strength. It is clear from Theller's results that for both Low Density Polyethylene and High Density Polyethylene/Ethylene-Vinyl Acetate - Paperboard (LDPE/EVA-PB and HDPE/EVA-PB), an increase from 40 psi to 400 psi offers only a slight improvement in seal strength.

Theller (1989) concluded that the "temperature of the heat seal layer at the interface, where the seal is formed, is the most important factor affecting its strength." This is true for both films tested. The significance of heat seal temperature appeared to be much greater than the dwell time or pressure. However, a statistical analysis was not provided.

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The theoretical calculations for the heat transfer analysis, were derived and figured to estimate the temperature at a film interface between two heated sealing bars. The physical test was run with temperature sensitive waxes placed in the interface of the films, to verify the theoretical calculations. The pre-melt transitions and relaxations, were investigated for effect on the heat seals, and derived from literature (Boyer,1977). One point examined were the "molecular level transition-relaxation associated with the thermal disruption of segment-segment contacts," or "non-flow or transition type phenomena (Boyer,1977)." This is noted in the literature as Tll (Boyer,1977). Another point examined was the "pre-melting relaxation occurring at temperature T-alpha-c (noted here as Tac) (Boyer,1985).

For LDPE, Theller presented the following conclusions: for dwell time, the effect on heatsealing is slight; for pressure, the effect is very slight; and for temperature, the effect is very significant. The parameter ranges used were: pressure -- 40 and 400 psi, dwell time -- 100 and 6000 ms, and temperature -- wide range.

The American Society for Testing Materials (ASTM) Task Group 2.33.03 (1988) is presently developing a standard test method for evaluation of two aspects of heatsealability, namely: surface sealability, and web sealability. The surface sealability deals only with the surfaces of the materials, and web sealability deals with the entire film (including heat

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transfer). The variables examined were temperature, dwell time, and die configuration.

The experimental design employed in this study, evaluated one variable at a time. For example, the pressure is held constant while determining an optimum sealing temperature. An optimum dwell time is then determined.

According to the ASTM Task Group Procedure, the pressure was maintained constant at 60 psi, and the seal strength was determined as a function of heat seal temperature, with a constant dwell time of 500 ms. Once the optimum seal strength temperature was observed, the dwell time was examined from 100-6000 ms at 100 ms increments, until a further increase in dwell time resulted in no accompanying increase in seal strength.

The procedure as proposed by the ASTM Task Group measures the relative sealability of films. It does not however, address maximizing parameters -- which is considered in the present study. With respect to the heat sealing parameters: (i) pressure is considered a constant and set at 60 psi; (ii) dwell time is considered of minor significance and ranged from 100 ms to 6000 ms; and (iii) temperature is increased from below onset of melt, through a maximum seal strength value.

3.02.02 Hot Tack

> The term "Hot Tack" is used as an expression for the strength of heat seals immediately after the sealing operation, in contrast to the term

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> "seal strength" which applied to the strength of the seal after it has cooled.
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> Information on the hot tack properties of materials is of special interest to the manufacturers of raw materials and laminates for the packaging industry. This includes manufacturers of basic polymers, plastic laminates, plastic coated paper and carton. The hot tack properties are of particular interest in form/fill operations where production disturbance can occur if the hot seals are still weak during the actual production operation. In addition, the influence of contamination from the product to be packed (powder or liquid) on hot tack is of interest (DTC,3).

A procedure for determination of the hot seal strength or hot tack strength of a heat seal using the Rucker Hot Tack Attachment on the Theller Model EB Precision Heatsealer, has been described by the James River Corporation (1988) and is summarized below.

As stated by the James River Corporation publication, "this procedure provided a determination of the hot seal strength of heat sealable flexible web materials," and will rank a series of films in order of hot tack strength. The values obtained are relative and may vary between different heatsealers. However, the order (rank) of the films will be the same.

On the hot tack equipment the sealing bars are automatically actuated, but the removal of the molten seal and the tensile testing are performed manually.

This is a very simple test methodology. The pressure and dwell time are held constant, and the temperature is raised incrementally, until the hot tack strength passes through a max

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maximum and back to zero (no seal at the lowest weight).

The temperature testing begins at 20-30 degrees Celsius below the optimum expected temperature level, that is the melt temperature. The temperature is then increased incrementally until the seal strength passes through a maximum.

The seal tensile testing is carried out with a lever that has a seal sample holder at one end, and a series of variable test weights at the opposite end (on the other side of the fulcrum). After the sealing dies have been activated, the test weights are released. When released, the lever opens the seal and the test weight is applied to the molten seal.

The maximum hot tack is defined as the highest weight at which a successful seal is obtained. The sensitivity of the test is ±25g.

In an alternate study, the test methods described by Frito-Lay (1984) for evaluating hot tack, measures the hot tack seal integrity of polymer film heat seals in terms of dwell time, temperature, pressure, and applied shearing force.

In the Frito-Lay study, a Sentinel Sealer from Packaging Industries Incorporated, with a hot tack attachment from Versa-Tool was utilized.

In this study, the dwell time (500 ms) and pressure (30 psi) were held at constant values and the temperature was increased until failure occurs. The highest weight to pass is then tested five times. If one of the five fails, then the next lowest weight is tried five times.

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This test procedure defines hot tack as: the ability of the heat seal to resist separation when stressed immediately after the seal is made.

This test procedure defines the top seal bar or the top film strip (Frito-Lay, 1988).

A test method for evaluating hot seal strength was also described by the Design and Test Consult (DTC) company. The DTC test procedure can vary the parameters of temperature, dwell time, pressure, sealing time, and seal peeling range. Usually only the temperature is increased in a test, but all variables could be tested.

The equipment used was the DTC Hot Tack Tester, Model 52-C. This is a totally automated sealing and testing instrument, with fully automatic parameter controls

While any combination of variables can be evaluated, usually only the temperature is varied in tests for hot tack strength. The Recommended test conditions for the DTC procedure are in Appendix E. It should be noted that the DTC company observed a normal type distribution when plotting hot tack as a function of sealing bar temperature. That is, the hot tack strength, increases to a maximum, followed by a decrease in hot tack strength as a function of increasing sealing bar temperature (DTC,1988).

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4.0 EXPERIMENTAL DESIGN

The experimental design was developed to evaluate the effect of temperature, dwell time, and pressure on the polymer seal characteristics during sealing. The goal was to use the response surface to determine values of temperature, dwell time, and pressure to achieve the maximum seal strength.

The initial study is referred to as the pilot test. The study that will generate the maximum seal strengths for future correlation, is referred to as the experiment test. These two tests were carried out on both cold seals and hot tack seals. Hot tack is the measurement of a seal that is still molten (has not been allowed to cool to ambient conditions) and a cold seal is the measure of a seal that has been allowed to cool to ambient conditions.

The objective of the cold seal study was to statistically determine the maximum seal strength and the optimum heat seal variables at this maximum. The variables for the heat seal are dwell time, pressure, and temperature. From the pilot test conducted on a randomly selected film in the series of test films (4.5% Vinyl Acetate), trends that were reported in the literature, were confirmed. Also, the selection of the heat seal variable levels (the specific points) for the full experiment, were verified in the previously completed pilot test.

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The objective of the hot tack study was to examine present practices for measuring hot tack, and obtain maximum hot tack values for the individual film samples.

The specific procedures used to determine seal strength were based upon the following:

ASTM D 882-83 "Standard test methods for tensile properties of thin plastic sheeting,"

ASTM F 88-85 "Standard test method for seal strength of flexible barrier materials,"

ASTM F 904-84 "Standard test method for comparison of bond strength or ply adhesion of similar laminates made from flexible materials,"

ASTM D 3078-84 "Leaks in heat-sealed flexible packages,"

ASTM task group 2.33.03 concerning the status report from "Test for heatsealability of flexible web materials"

For the hot tack testing, in addition to the ASTM test method, procedures adopted by the James River Corporation and the Design and Consult AB (DTC--Stockholm, Sweden) were followed.

> If measuring devices and those who used them were perfect, it would be possible to make a direct determination of the variability of the true values of measurement as well as variation in the quality measured. Actually, the measured values reflect errors of measurement as well as variation in the quality measured (Grant,1988).

A repeatability and reproducibility study was conducted for the purpose of determining how much variability is introduced through the laboratory measurement instruments, and the operator. The tests were carried out to determine the variations of:

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- Pressure (die loading)
- Temperature
- Dwell time
- The tensile testing machine
- The material's tensile strength
- The measured strength of a cold seal
- The measured strength of a very defective cold seal
- The operators testing technique, and the hot tack test reproducibility.

The results obtained were then considered in the development and analysis of the overall experimental design. The tests were ordered and grouped to minimize the possibility of an incorrect significant test result.

To evaluate the effect of the heat seal variables, a 5-by-3-by-3 (five temperatures, three dwell times, and three pressures) factorial design was run and a surface of the results was generated (not included). To display the response, one variable is held constant and the remaining two variables evaluated over a range of values. A three dimensional response surface is then produced with the two independent variables forming two of the coordinates of the surface response graph, and the resulting seal strength forming the third coordinate. The response surface profiles were examined to determine conditions yielding maximum seal strength values.

For convenience in the analysis, coded values were used for the three independent variables. From literature and preliminary examination of the seal effects, points were chosen which represent low, medium, and high values for the

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respective variables. In the case of temperature, the range had five values for a more detailed evaluation of the temperature effect.

The pressure levels are noted as 1, 2, and 3, which represent values of 10 psi, 100 psi, and 500 psi, respectively. The literature notes a constant effect on seal strength from 40 psi (Theller,1989) to 60 psi (ASTM Task Group 2.33.02,1988). The dwell time levels are noted as 1, 2, and 3, which represent values of 200 ms, 500 ms, and 1800 ms, respectively. The literature notes a constant effect on seal strength from 200 ms to 400 ms. The heat transfer studies examine effects of dwell time on the interface temperature.

The temperature levels are designated as 1, 2, 3, 4, and 5, which represent temperature values, calculated by a series of formulas that algebraically take into account the melt profile data of the respective film samples obtained by differential scanning calorimetry (DSC) analysis. In practice, the points are estimated as noted below. The formulas cover the representative melt range. Temperature "1" is one-third the difference between onset temperature and melt temperature. Temperature "2" is the melt temperature. Temperature "3" is the point at which the melt endotherm, obtained by DSC, is completed. This will be referred to as the burn temperature. Temperature "4" is 10% beyond the burn temperature. Temperature "5" is 20% beyond the burn temperature.

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4.01 Pilot Test Studies

As previously stated, this experiment represents a preliminary evaluation of the variables for optimization of heat seal parameters, and is defined as a pilot test. The goal of a pilot test is to roughly examine data and collect general, but statistically significant trends and shapes of curves, and to generate ranges to be used in the following experiment test. A preliminary pilot test was run on one randomly selected film sample, and the film was exposed to a broad range of the three variables. The pilot test was devised to examine the effect of temperature, dwell time, and pressure on heat seals and hot tack.

The effect of temperature, dwell time, and pressure, both individually and in combination, can be established from the resultant response data. Several of the variables were found to effect the seals at lower seal strength levels, but not at the maximum seal strengths. Since this study is concerned with the maximum seal strength, the effects will be discussed and analyzed only at the maximum seal strength levels. The effects of the variables, at maximum seal strength, were observed on a graph of average seal strength with standard error bars. Once this data was obtained and analyzed, the methods and exact ranges of the experiment test were revised.

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4.02 Experiment Test

Following analysis of the preliminary data, the final experimental procedures were revised, and the actual test points and ranges were selected.

After reviewing the literature and the result of the pilot test, heat seal variables were categorized according to their effect on seals, at the seal strength maximum, as: (i) constant, (ii) some effect, and (iii) major effect.

As with the pilot test, the experimental design was organized into response surface plots, and a table of significance levels.

4.03 Definitions

Some specifics and definitions are of importance to these tests and are presented below.

Heat Seal. A heat seal is a seal that has been formed by heated application, and allowed to cool to ambient conditions. Thus, this is sometimes referred to as a "cold seal." In agreement with ASTM standards on tensile tests, and ASTM seal strength tests, the break factor and tensile strength to break, will be noted as the seal strength. This is displayed on a digital read out on the tensile tester.

Detection of seal failure, thus, is completely automatic and defined as complete break of the film.

Hot Tack. Hot Tack is the ability of a material to hold,

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while the interface is still molten. It is sometimes referred as the initial amount of "stickiness." The definition of seal failure is a rip or peel of more than 1/8 inch. Presently there are no ASTM standards for hot tack, but there are a number of industry tests. The test is manual and has added errors that are not present in the cold seal test. The recorded test weights are sensitive to 10 g; this was modified down from 25 g for industry tests.

Thus, failure is noted by visual inspection of the seal after testing and cooling.

4.04 Determination of Maximum

Heat Seal. The objective of a heat seal study must be clearly defined when examining output or results. This experimental test involved optimization of seal parameters, for maximum seal strength of an individual film.

To obtain the maximum seal strength value, at the optimum conditions, this study involves selecting the highest average seal strength value for any of the test conditions. The highest average seal strength value is accurate because of the flatness of the response surface near the optimum conditions.

Hot Tack. The hot tack testing will clearly show a maximum seal strength. The hot tack strength is defined as the maximum weight applied at which a seal does not fail.

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5.0 MATERIALS AND METHODS

5.01 Materials

The polyethylene film, and a series of ethylene-vinyl acetate copolymer film samples used in the present study, were provided by Quantam Chemicals - USI Division (Rolling Meadows, Il.). The composition of the film samples, expressed as percent (weight/weight) vinyl acetate were as follows: 0% VA, 4.5%, 7.0%, 9.0%, 19%, and 29%.

5.02 Polymer Characterization

5.02.01 Composition

The relative composition of the representative test films was provided by the supplier, and was determined by an infrared spectrophotometric method of analysis. Below 6% vinyl acetate, the absorption band observed was at 1020 (cm^-1), and above 6% vinyl acetate, the absorption band observed was at 609 (cm^-1). A relative comparison of absorption band intensities was the basis for analysis.

5.02.02 Tensile Testing (no seal)

The tensile strength of the respective polymer samples was determined on an Instron 4201 Universal Tensile Testing Apparatus, with 90 psi Pneumatic Grips, and a 1KN Load Cell.

The tensile testing, for tensile strength, and break factor, were recorded as outlined in ASTM D 882-83 "Standard test methods for tensile properties of thin plastic sheeting," and the instruction manuals recommended procedures.

The tests were run at a cross head speed of 12 inches per minute, the specified cross head speed for testing of seals (normally films without seals are tested at 20 inches per minute). This allows for standardized conditions between both tests, and a more accurate correlation between the tensile strength of the film, and of the seal. The tensile strength was used in the statistical analysis, to allow direct correlation of tests.

5.02.03 Thermal Analysis

The thermal profile and melt temperatures (Tm) of the test samples were determined by Differential Scanning Calorimetry (DSC) analysis. Analysis was carried out on a DuPont Instruments 910 Differential Scanning Calorimeter, and DuPont Instruments 9900 Computer/ Thermal Analyzer. The procedure employed was based on the manufacturers recommended procedure. The polymer samples were heated from 30 degrees (C) to 200 degrees (C), at a programmed rate of 5 degrees (C) per minute.

5.02.06 Molecular Measurements

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respective polymer samples were determined by Gel Permeation Chromatography (GPC). The test employed a Waters-Millipore Gel Permeation Chromatography Model 150-C ALC/GPC with Styragel HT Linear Gel Permeation Columns 7.8 x 300 mm. A bank of three columns was used, with column temperature of 135 degrees (C), and a solvent of 1,2,4-trichlorobenzene spectrophotometric grade (Aldrich Chemical Company Inc., Milwaukee, WI.), at a flow rate of 1 ml/min.

5.02.07 Density Determination

The density of the perspective polymer samples was determined by a density gradient column (DGC) technique. A Cole-Palmer Instrument Company Density Gradient Column (DGC), with benzyl alcohol and methanol, to provide a density gradient ranging from 0.88 g/cm^3 to 0.98 g/cm^3, was used. The density value determinations were carried out according to ASTM D 1505-68(79) "Standard test method for density of plastic by Density Gradient technique."

5.02.04 Thickness

The thickness measurements of the respective polymer samples were determined with a Model 549 Micrometer, Testing Machines Incorporated (Amityville, NY.). The sampling scheme for the thickness tests was carried out as recommended in ASTM D 1898-68 (85), "Standard Practices for Sampling Plastics." The lab equipment is regularly calibrated.

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5.02.05 Melt Flow Index

The melt flow index (MFI) of the prospective test films was determined on a Ray Ran Model 2A Digital-Auto Melt Flow Index Unit. The testing was carried out with methods and conditions outlined in ASTM standard D1238-79 "Standard test method for flow rates of thermal plastics by extrusion plastometry," with recommendations for testing from the instruction manual. The test weight used was 2.16 Kg. The test temperature for the low density polyethylene and copolymers containing up to 19% vinyl acetate was 190 degrees (C). The test temperature for 29% vinyl acetate was 150 degrees (C).

5.03 Seal Experiments

The seal experiments were recorded in grams per seal width, since industry data specifies recording in these units (U.S. industries methods in the Appendices)

5.03.01 Pre-Research Tests

Repeatability and Reproducibility. Prior to conducting the pilot test studies, repeatability and reproducibility (R and R) of operator, equipment and material was studied. The tests were performed on the two extremes of vinyl acetate composition (0% VA and 29% VA) of the test films, and included: seal die loading, sealing bar temperature, dwell time, materials tensile test, and hot tack. The R and R test data were analyzed to determine if the studies were "in" or "out" of control.

Heat Transfer Analysis. The film used was a polyethylene co-polymer. For PE, a teflon sheet was placed between the film and the sealing bars. The order of layers was: upper sealing bar, teflon sheet, upper film, interface, lower film, teflon sheet, and lower sealing bar.

Heat transfer analysis during sealing by theoretical calculation and physical tests, with evaluation of pre-melting transitions and relaxations, was investigated by the following procedures.

1. Heat Transfer Determination by Heat Sensitive Waxes (Omega,1988).

Conditions: The pressure and dwell time were as per the heat seal tests, p=200 psi and dt=500 ms. The temperature was set at -10x (10 degrees less than maximum seal strength temperature), -5x, x, +5x, +10x; 'x' is the melt temperature of the specific wax.

The heat seal experimental methods were used in this analysis.

Procedure: Roughly 1mm^2 of wax was deposited in the region of polymer film that was to be heat sealed. The film was then sealed, and the heat sensitive wax observed to establish if the melt temperature was attained during the sealing process.

2. Theoretical Calculations.

The calculations were done with the "one dimensional transient heat conduction equation," and examined at 10 milliseconds (ms), 100 ms, and 200 ms.

$$\delta t/\delta\theta=\alpha(\delta^2/\delta x^2)+q'/cp \qquad \text{(eq. 2)}$$

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These equations [only the corresponding rectangular coordinate equation is shown] have been solved analytically for solid slabs, cylinders, and spheres. The solutions are in the form of infinite series, and usually the results are plotted as curves involving four ratios. Since each ratio is dimensionless, any consistent units may be employed in any ratio. The significance of the symbols is as follows: t = temperature at a given point in the body at the time theta measured from the start of the heating or cooling operations; p = uniform density of the body; c = specific heat of the body; and x = distance in the direction of heat conduction from the surface of a semi-infinite body (such as the surface of the earth) to the point under consideration.

3. Transitions and Relaxations.

Pre-burn melt relaxation (associated with amorphous burning - Tll) and pre-melt transition (associated with crystalline melting - Tac) were calculated from formulas developed by Boyer (1985). The temperatures are noted in the data above. The temperatures were calculated from DSC melt temperature data using Boyer's (1977) equations. These temperatures were compared to the seal temperature data, to determine their effect. The derivations are as follows:

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$$T_g(K) = \left(\frac{2}{3}\right) T_m(K)$$

(eq.3)

$$T_{l_l}(K) = (1.2)\, T_g(K)$$

(eq. 4)

$$T_{a_c}(K) = (30.47)\, T_g(K)$$

(eq. 5)

5.03.02 Heat Seal Pilot Study

5.03.02.01 Applicable ASTM Standards:

D882: Standard Test Methods for Tensile Properties of Thin Plastic Sheeting.

F88: Standard Test Methods for Seal Strength for Flexible Barrier Materials.

D1898-68: Standard Practice for Sampling of Plastics.

ASTM task group 2.33.03 - Heat Sealability of Flexible Web Materials (a proposed standard).

5.03.02.02 Equipment Used

The heat seal testing was conducted on a Precision Instruments West Laboratory Heat Sealer, Theller Model EB. This equipment was Provided by Hershey Foods (Hershey, Pa).

5.03.02.03 Experimental Design

The experimental design began with three general variables of temperature (T), dwell time (dt), and pressure (p). The pilot studies were carried out on a polymer in the

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test series, selected at random (by rolling a die). Both pilot tests used the 4.5% vinyl acetate copolymer.

To observe the activity across a broad range of the respective parameters, and to note specific seal changes, a 5-by-3-by-3 (T,dt,p) factorial test design was developed. This test would yield a response surface graph. Thus, a broad three point test would show the profile and the maximum seal strength.

The data obtained included the seal strengths at the respective conditions of the test, and the failure mode exhibited. The procedure was as follows:

1. Sample Cutting:

   First a film sample is removed from the roll stock, being very careful not to stress the film by pulling too hard. Also, care should be taken in handling the seal area. Both of these factors may be a problem with the more tacky films (the films that have higher percent vinyl acetate).

   Second, the samples are cut with a template, and stacked in order for later testing. The template is a paper board overlay that reduces tearing of the film during cutting. The template cuts four samples per column. Begin sample cutting by unrolling the film and discarding at least one full revolution of the surface film.

   The columns and the rows on the uncut film are numbered. Cut four samples (four rows) in each, and use as many columns as required for the test samples. Keep the samples in order of first cut to last cut. Later thickness sampling measurements will rely on these samples being in order.

2. Sealing Conditions:

   Pressure: The pressure, in pounds per square inch, range to be examined is represented by the values 1, 2, and 3, in the experimental design section. To cover a range of variable pressures, noting that a plateau would result, both the pilot and experiment test studies used pressures of 10, 100, and 500 psi, respectively.

Dwell Time: The dwell time, in milli-seconds (ms), range to be examined is represented by the values noted 1, 2, and 3, in the experimental design section. In the literature, boundary dwell time numbers were observed. To cover a range of variable times, noting that a constant would result, both the pilot and experiment test studies used dwell times of 200, 500, and 1800 ms, respectively.

Temperature: Each of the temperature values were algebraically selected and verified in the pilot test data. The exact points for each film had this origin in Differential Scanning Calorimeter analysis. The values beyond the melt temperature, are to assure the peak and then decrease in the curve. The points are:

1. [(1/3*(Tm-onset))+onset)]: This was near the first temperature at which a true fusion bond occurred.

2. Tm: Melt Temperature is the temperature at which the maximum seal strength should occur.

3. End: This may be beyond the maximum strength (a weakening affect may be observed) of the seal, but a maximum here would be reasonable.

4. [110%*End: This is a point that most definitely should show burn through, and an extremely weak seal.

These four points may be sufficient to show a peak. If not, then use points five and six.

5. [120%*end]: This is a point well beyond where burn through should occur, and thus, a lesser seal strength would be observed.

6. [140%*end]: This is a point well beyond where burn through should occur, and thus, a lesser seal strength would be observed.

For the combination of parameters proposed, the seal strength should show a curve of an increase, a constant value over a temperature range, followed by a decrease in seal strength as a function of increasing temperature.

The test was repeated three times for each combination of heat seal parameters. These three replicates were satisfactory for showing rough curve shape.

3. Sealing:

The test film is carefully folded, and placed in the Teflon sleeve of the heat seal equipment. Care should be exercised to limit contacting the film seal area. Seal the film, and note the conditions with permanent marker on the film. The film is then stored at standard TAPPI (Technical Association of Pulp and Paper Industries) conditions of 73 degrees F and 50% RH, until sealing.

4. Thickness Sampling:

While cutting the sample for tensile testing, take thickness measurements were taken. The sampling procedure was to measure every third consecutive piece of cut film. This method will monitor the sequential thickness throughout the roll of film. A standard micrometer was used for the measurements.

5. Cutting the film to Standard:

First trim the end loop, take a thickness measurement, and then cut to the one inch wide samples noted in the standards. The sample cutting will be done on the standard sample cutter.

6. Tensile Testing/Tensile Strength:

Wait for more than sixty minutes before taking tensile measurements. This will allow the measurement test to be truly a cold seal test.

According to standards regarding seal strength, the test is carried out at a cross-head speed of 12 inches per minute (12 ipm). The type of break exhibited is noted, and the maximum load (tensile strength and break factor) in pounds per seal width, and pounds, is determined. The test records the tensile strength to account for any differences in film thickness.

7. Maximum Seal Strength/Results:

This data will observe trends and the significance of the parameters examined. The results of the pilot test will be analyzed by a statistical computer program, and the outcome used to select parameter values for the experiment test.

5.03.03 Heat Seal Experiment

5.03.03.01 Applicable ASTM Standards

D882: Standard Test Methods for Tensile Properties of Thin Plastic Sheeting.

F88: Standard Test Methods for Seal Strength of Flexible Barrier Materials.

D1898: Standard Practice for Sampling of Plastics.

ASTM task group 2.33.03 - Heat Sealability of flexible web materials (a proposed standard).

5.03.03.02 Experimental Design.

The experiment test used the two parameters proven to be variable in the Pilot Test, namely: temperature (T), and dwell time (dt). This study was carried out on all of the film samples, and provides a range of vinyl acetate concentration levels.

To observe the activity across a broad range of the respective parameters, and to note specific seal changes, a 3-by-3 (T,dt) test design was run. Thus, a broad three point test would show the profile and the maximum seal strength. The data obtained included the seal strengths at the respective conditions of the test, and the failure mode.

The procedure was as follows:

1. Sealing Conditions:

   Pressure: From the data observed in the literature and the previous pilot test, the pressure, is set to a constant value. The pressure was set at 200 psi, because above which, increased pressure does not significantly affect a seal.

Dwell Time: The dwell time was reported to be almost insignificant and the seal strength was constant beyond 200 ms. However, to be sure, studies were carried out with dwell times of 500 ms and 1800 ms, respectively. These are industry used points, and 1800 ms is near the practical maximum. The two dwell time values selected were chosen to prove this testing range to be within the constant region of the surface response profile.

Temperature: Each of the test points were picked to be the same percentage around the peak (for example two degrees below a melt temperature of 40 degrees might be sufficient, but not for a melt temperature of 300 degrees). The exact points for each film was based on Differential Scanning Calorimeter analysis. The temperature values were similar to those evaluated in the pilot test and are summarized below:

1. [(1/3*(Tm-onset))+onset)]: This was near the first temperature at which a true fusion bond occurred.

2. Tm: The Melt Temperature is the temperature of the interface at which the maximum seal strength should occur.

3. End: This may be beyond the maximum strength (a weakening affect may be observed) of the seal but that is an acceptable outcome. The plateau will still be observed and defined.

4. [110%*End]: This is a point that most definitely should show burn through and an extremely weak seal.

These four points may be sufficient to show peak or plateau. If not, then use points five and six.

5. [120%*end]: This is a point well beyond where burn through should occur, and thus, a lesser seal strength would be observed.

6. [140%*end]: This is a point well beyond where burn through should occur, and thus, a lesser seal strength would be observed.

For the combination of parameters proposed (see results section), the seal strength should show a normal (for this test) type distribution curve of an increase, a constant value over a temperature range, followed by a decrease in seal strength as temperature is increased. The test is repeated five times for each combination of heat seal parameters.

2. Maximum Seal Strength/ Tensile Strength:

To complete a test series, the seal strength values are observed and analyzed to establish that a maximum seal strength value has been attained.

Once a maximum is observed (both tensile strength and break factor), the highest value of seal strength from the individual film data is noted as the maximum seal strength. The standard of deviation will also be noted at this maximum for correlations. The test records the tensile strength.

5.03.04 Hot Tack Pilot Study

5.03.04.01 Applicable ASTM Standards

D882: Standard Test Methods for Tensile Properties of Thin Plastic Sheeting.

F88: Standard Test Methods for Seal Strength for Flexible Barrier Materials.

D1898: Standard Practice for Sampling of Plastics.

ASTM task group 2.33.02: This group is reviewing industry procedures for Hot Tack (a proposed standard).

5.03.04.02 Equipment Used.

The heat seal testing was conducted on a Precision Instruments West Laboratory Heat Sealer, Theller Model EB. The Hot Tack Testing was carried out on the same laboratory heatsealer, but with the Rucker Hot Tack Attachment. Basically, this is a standard laboratory heatsealer, with a side attachment. The apparatus slides next to the sealing bar. Just after forming a seal, the apparatus is slid out, and the weighted bar is dropped to test the seal. The falling weight, stresses the seal.

5.03.04.03 Experimental Design.

The experimental design began with three general variables of temperature, dwell time, and pressure. Preliminary studies were carried out on a polymer in the test series selected at random. The 4.5% vinyl acetate copolymer was selected for this study.

To observe relevant data concerning the optimization, surface response graphs were constructed to obtain maximum hot seal strength values. As per the industry test, the dwell time and pressure were held constant, and the temperature varied, to include the following temperature values:

1. [(1/3*(Tm-Onset))+onset)]: This was the first temperature at which a true fusion bond occurred.
2. Tm: Melt Temperature. The Tm is the temperature of the interface at which the maximum seal strength should occur.
3. End: This may be beyond the maximum strength (a weakening affect may be observed) of the seal but that is an acceptable outcome, The plateau will still be observed and defined.

5.03.04.04 Methods

The James River Corporation Hot Tack Test Method (James River Corp.,1988) was initially followed for the Hot Tack Pilot testing. However, after a series of runs, in the Experiment test, the modifications of the test method were incorporated in the test procedures.

5.03.05 Hot Tack Experiment

Hot Tack seal strength data was obtained by three related test methods as discussed below. However, for statistical analysis to establish bond strength/polymer property correlations, data obtained by "Method C" was employed.

Method A: See James River Corporation Hot Tack Test Method in Hot Tack Pilot Study (See Appendix D).

Method B: (A modified James River Corporation Hot Tack Test Method) This test method incorporates an adhesive tape that is applied to the back of the test film. This tape acts as a carrier web to reduce the influence of a polymer films inherent elastic properties. The seal, not the material, is thus tested.

Method C: The modified James River Corporation Hot Tack Test Method with an additional weight sensitivity down to 10 grams instead of 25 grams. This is important, since with test method B, some films exhibited a strength of zero. This test method is noted below:

1. Sample Preparation:
    - A. Cut 2" x 5" Samples
    - B. Adhere an adhesive tape (a filament polymer tape) to one surface of the film.
2. Equipment Settings - Constants:
    - A. Set p = 40 psi
    - B. Set dt = 1000 ms
3. Set temperature. This temperature should be 20-30 degrees C below the expected seal strength maximum. The expected maximum peak will be at the Melt Temperature.
4. Put 50 gram weight, raise temperature until it adheres.
5. Hot Tack Failure:
    - A. Definition: "...when the fresh seal separates by more than 3/16" when stressed as measured from the junction of the seal to the outer mark of the top seal bar on the top film strip." Failure is also denoted if a break in the film/seal/interface occurs.
    - B. Record: The type of failure is recorded for analysis of film. The types of failure will be denoted as: 0 = no seal, P = peel failure, and, B = break failure.
6. Methodology:

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A. put the brace under the weight bar
B. put the sample on top of the teflon sleeve
C. put this in the lower HT jaw
D. put the film (material only) in the upper HT jaw
E. put a rod in the loop of material, and wrap the teflon sleeve around the entire seal
F. slide the material, by using the rod, under the sealing jaws
G. pull out the rod
H. make the seal
I. slide out from under the sealing bars, count 2 sec from the time the sealing bars open, to the time the weight is released.
J. release the weight bar, this is weight plus gravity, to make the seal fail.
K. before removing film, notice if failure occurs.

7. Do two replications for the maximum and one temperature value higher. This extra test will verify the maximum. Also, for questionable seals (very loose definition), retest.

8. For these tests, the maximum weight that led to a successful seal, and the accompanying sealing bar temperature will be recorded.
9. Tensile Testing Values: The test records the tensile strength at break, to account for variations in thickness of the sealant film.

5.04 Correlations

The computer software program Statgraphics Version 3.0, analyzed the test data, calculated the statistics, and then presented the data of: sample correlation coefficient (based on sample size), and the "p-value." These values are derived from the average test results. The table (Table 18) includes both the correlation coefficient and the p-value.

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6.0 RESULTS

The results are presented in three sections: 1. Polymer Characterization, 2. Seal Strength, and 3. Statistical Correlation. It is noted that the graphs could not be altered, to be on a true scale.

6.01 Polymer Characterization

Polymer characterization included both the bulk and molecular properties of the test polymers.

6.01.01 Tensile Testing (No seal)

The tensile strength to break and the break factor, for the respective test films, were determined on the Instron tensile tester. The results are summarized in Table 1.

6.01.02 Thermal Analysis

Differential scanning calorimetry (DSC) thermal analysis was performed to determine the melt profile of the respective test films. Both the melt onset temperature and the bulk melt temperature were recorded, and the results summarized in Table 2. From this data the heat of fusion (Hf) for the respective test samples was obtained, and these values were used to estimate the percent crystallinity of the polymer samples. The results are summarized in Table 3.

$$PercentCrystallinity-\frac{ActualH_f(Cal/g)}{Theoretical100\%H_f(Cal/g)}$$

(eq.6)

The percent crystallinity was obtained from equation (6) (Troedel,1984). The theoretical value of Hf was obtained from the literature. For polyethylene, the values for the theoretical 100% crystalline heat of fusion was 68.4 cal/g (Troedel,1984). For the respective vinyl acetate/ethylene copolymers, it was assumed that the theoretical value for the heat of fusion was equivalent to the PE value. The polymer supplier (Quantam) recommended using the PE heat of fusion value (the theortical value of 68.4 cal/g), for the respective vinyl acetate/ ethylene copolymer samples to estimate the percent crystallinity of the respective EVA copolymer samples.

From the DSC determination of the melt temperature (Tm), the glass transition temperature (Tg) and the relaxation or transition temperatures were calculated from equation 3. Pre-burn melt relaxation (associated with amorphous burning - Tll) and pre-melt transition (associated with crystalline melting - Tac) were calculated from formulas developed by Boyer (1985). This data is presented in Table 4.

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6.01.03 Density Determination

The density gradient column (DGC) was used to measure the density of the respective polymer samples. The data is summarized in Table 5.

The glass calibration beads used were: 0.8869 g/cm^3, 0.9316 g/cm^3, and 0.9716 g/cm^3. The equilibrium level of the polymer, in the column, was measured in column units. The column unit is converted to density by the point determination (y-intercept formula) from the linear regression. This test method is noted in ASTM D 1505-68(79).

6.01.04 Molecular Mass Measurements

Gel Permeation Chromatography (GPC) was used to determine the molecular mass and molecular mass distribution of the polymer samples. The data is summarized in Table 6, and the complete data is recorded in Appendix H. The GPC figures the averages automatically.

6.01.05 Thickness

The data was incorporated into all tensile tests. The thickness data is summarized in Table 7, and complete data is located in Appendix I.

6.01.06 Melt Flow Index (MFI OR MI)

The melt flow index (MFI) is a measure of the viscosity of a polymeric material, as per the conditions of the test.

The data is summarized in Table 8, and complete data is located in Appendix J.

6.02 Seal Experiments

The seal strength experimental data covers all aspects of the experimental design and the measured variables.

6.02.01 Pre-Research

From the repeatability and reproducibility tests, it was observed that the operator, heat seal equipment and materials are all statistically within control, exhibiting small standard of deviations under a variety of seal conditions. The data show no false trends or errors due to operator, machine, or materials. The test results are tabulated in Appendix K.

Parameters observed are: Pressure (seal die loading, measured by internal calibration), Temperature (by internal calibration, and by heat sensitive waxes), Dwell Time (by internal digital display read out), Seal Strength of extreme values of the test films (0% VA and 29%VA), Material Strength without a seal, Material Strength with defective sample cutting and without a seal (worst case scenario), and Hot Tack test repeatability of results (See Appendix K).

The data for calculations, are summarized in Table 9. The physical (wax) test data is summarized in Table 10, and the theoretical calculations are summarized in Table 11.

6.02.02 Heat Seal Pilot Study

The pilot data was examined to determine the effect of variables across the seal range, as noted in the methods. Only the effects around the maximum seal strength, were examined to select parameters for the detailed heat seal experiment. The results of the Pilot Study are summarized in Table 12.

For better illustration representative single variable graphs are included in Figures 1, 2, and 3, which show maximum effect of the variables. In these figures the middle value for pressure and dwell time were used (p=50 psi and dt=500 ms). The total data is presented in Appendix L.

Observations for consideration in the design of the heat seal experiment are: seal strength is constant for dwell times beyond 200 ms (or a minimum pressure), seal strength is constant with pressure values above 10 psi, and the maximum seals were viewed above the melt temperature (Tm) of the polymer film. The term constant is defined as a situation where the seal strength will not increase with an increase in temperature or pressure.

6.02.03 Heat Seal Experiment

For the heat seal experiment, the seal temperature was varied across the set range, the dwell time was varied within a region that was assured to show constant effects on seal strength, and the pressure was held constant. The results are summarized in Table 13.

The actual sealing test parameters are noted in table 14. Tabulation of all test data is presented in Appendix M. The maximum seal strengths are summarized in Table 15.

For better illustration, the results are presented graphically in Figures 4, 5, 6, 7, 8, and 9 (where p=50 psi and dt=500 ms). Where seal strength is plotted as a function of a single temperature, and variable dwell times; and as a function of a single dwell time, and variable temperatures, respectively. As shown, seal strength increased to a maximum with an increase in temperature, showing either a maximum followed by a decrease in seal strength with continued increase in temperature, or a plateau (where the seal strength remained constant with an increase in temperature).

6.02.04 Hot Tack Pilot Study

The results of the pilot study were evaluated for effect of variables across the seal range, as noted in the methods. Only the maximum seal strength effects were considered in the design of the heat seal experiment. The results of the Hot Tack Pilot Test are summarized in Table 16.

Graphs of: 'temperature versus dwell time,' 'temperature versus pressure,' and 'pressure versus dwell time,' are presented in Figures 10, 11, and 12. The constant variable was held at the melt temperature, and at the mid-point value for pressure and dwell time. The total data is presented in

Appendix N.

Observations for consideration in the design of the heat seal experiment are as follows: (i) dwell time is constant beyond 200 ms; (ii) pressure is constant beyond 10 psi; (iii) and the maximum seals are obtained at the Tm of the polymer. The term constant is defined as a situation where seal strength does not increase with an increase in temperature or pressure.

6.02.05 Hot Tack Experiment

The maximum hot tack seal strengths are tabulated in Table 17 and represented graphically in Figure 13. The complete data is located in Appendix O.

The curve represents the industry accepted hot tack seal strength (the curve) and the statistical range (between the square and the cross). This represents a range of seals, for example the hot tack of 4.5% VA is above 25 grams and below 30 grams.

The graphs included here, are a slice of the surface response. To fit in two dimensions, the mid-point values of two of the variables were held constant.

6.03 Correlations

The correlation data is summarized in Table 18. The physical and molecular properties of the respective test films, and the associated seal strength data obtained are summarized in table 19. The correlation plots of the significant correlation variables (viscosity average molecular mass, melt temperature, and percent composition), are included in Figures 14, 15, 16, 17, 18, and 19.

Table 1: Break Factor of Test Films (grams)

Film Sample (%VA)	Average	Standard Deviation	Replicates	Thickness
0	2277	402	10	1.419
4.5	2748	272	10	1.406
7.0	2799	423	10	1.663
9.0	3135	629	10	1.428
19	3824	467	10	1.691
29	4398	1150	10	1.678

Table 2: DSC Melt Profile Data

(A) Onset Temperature

Film Sample (%VA)	Onset Temperature (degrees C)		
	Run 1	Run 2	Average
0	103.31	103.47	103.4
4.5	93.71	95.71	94.7
7.0	85.31	95.25	90.3
9.0	89.39	89.21	89.3
19	67.77	70.88	69.3
29	59.61	54.5	57

(B) Melt Temperature (Tm)

Film Sample (%VA)	Melt Temperature (degrees C)		
	Run 1	Run 2	Average
0	113.21	113.77	113.5
4.5	105.2	104.64	104.9
7.0	100.85	103.09	102
9.0	100.85	101.08	101
19	85.54	83.97	84.8
29	74.08	65.49	69.8

Table 3: DSC Data of Heat of Fusion and Percent Crystallinity

Film Sample (%VA) Average	Heat of Fusion (cal/g)			Percent Crystallinity
	Run 1	Run 2	Average	
0	96.56	96.16	96.4	33.7
4.5	73.27	71.36	72.3	25.6
7.0	62.71	63.88	63.3	21.9
9.0	66.2	58.12	62.2	21.7
19	31.74	28.56	30.2	11.1
29	24.93	24.52	24.7	8.7

Table 4: DSC Derived Values of Polymer Transition and Relaxation Temperatures (a) (Degrees C)

Film Sample (%VA)	Tm (a)	Tg (b)	Tll (c)	Tac (d)
0	113	75.33	27.57	80.1
4.5	105	70	21.34	73.3
7.0	102	68	19	70.75
9.0	103	68.66	19.778	71.6
19	85	56.66	5.76	56.3
29	70	46.66	-5.92	43.55

(a) Tm = Melt Temperature
(b) Tg = Glass Transition Temperature
(c) Tll = Pre-Burn Relaxation (Amorphous)
(d) Tac = Pre-Melt Transition (Crystalline)

Table 5: Polymer Density Value by Density Gradient Column

Film Sample (%VA)	Bead Location (in column units)(a) Run 1	Run 2	Average	Density (Average) g/cm^3
0	41.7	41.85	41.8	0.925
4.5	41.2	41.2	41.2	0.925
7.0	40.25	40.5	41.4	0.927
9.0	37	37	37	0.932
19	31.1	30.6	30.9	0.941
29	30.5	31	30.8	0.941

(a) The calibrated beads of 0.8869, 0.9316, and 0.9716 g/cm^3, equilibrated at 65, 40, and 9 column units, respectively.

Table 6: Molecular Weight and Molecular Weight Distribution of test Polymer Films (in Atomic Mass Units - AMU's)

Film Sample (%VA)	Mn (a)	Mw (b)	Mv (c)	Mz (d)	MWD (e)
0	14682	155183	155139	301254	10.59
4.5	25600	172631	172571	469802	6.74
7.0	30285	179650	179618	484291	5.93
9.0	6934	171260	171242	520037	24.72
19	216531	166098	166057	436405	12.32
29	10893	120792	120764	238798	11.09

(a) Mn = Molecular Number Average
(b) Mw = Molecular Weight Average
(c) Mn = Molecular Viscosity Average
(d) Mz = Molecular Z Average
(e) MWD = Molecular Weight Distribution

Table 7: Thickness Measurements (in Mils, and 10 samples)

Film Sample (%VA)	Thickness Average	Standard Deviation
0	1.42	0.04
4.5	1.41	0.04
7.0	1.66	0.04
9.0	1.43	0.05
19	1.69	0.08
29	1.68	0.07

Table 8: Melt Flow Index

Film Sample (%VA)	Melt Flow Index (g/10 min) Average	Standard Deviation	Test Temperature (degrees C)
0	1.53	0.036	190
4.5	1.62	0.011	190
7	1.32	0.056	190
9	2.23	0.040	190
19	1.07	1.07	190
29	1.55	1.55	150

TABLE 9: Summary of Necessary Data For Heat Transfer Analysis

(a) Thickness of Relevant Materials (in mils)

Material	Thickness
Teflon Sleeve	2.88
0% VA Film	1.41
4.5% VA Film	1.66
7.0% VA Film	1.43
9.0% VA Film	1.69
19% VA Film	1.68
29% VA Film	1.68

(b) Temperature at Maximum Tensile Strength (in degrees C)

VA (%) (1)	Tss (2)	Tm (3)	First Non-Peel Seal (4)
0	113	113	113
4.5	111	105	105
7	107	102	102
9	101	101	101
19	115	85	96
29	121.7	70	87

(1) EVA is the percent vinyl acetate in the ethylene vinyl acetate film sample

(2) Tss is the temperature at which the maximum seal strength was achieved

(3) Tm is the melt temperature

(4) First Non-Peel Seal is the temperature at which the seal exhibited a break during failure.

Table 9 (cont'd)

(c) Transition and Relaxation Temperatures (in degrees C)

VA (%) (1)	Tll (2)	Tac (3)
0	27.6	80.1
4.5	21.3	73.3
7	19	70.75
9	18.2	69.9
19	5.76	56.3
29	-5.76	43.55

(1) VA is the percent vinyl acetate in the ethylene vinyl acetate film sample
(2) Tll is the temperature at which an amorphous molecule goes through a pre-burn transition
(3) Tac is the temperature at which a crystalline molecule goes through a pre-melt transition

TABLE 10: Physical Temperature Test Data (in degrees C)

Wax Melt Temp. (1)	Wax Melt Calibration Temperature (2)(Tm)	Sealing Bar Temp. to Melt (Tb) (3)	Difference (Tm-Tb) (%) (4)
65.6	66.3	65.6	0.7
79.4	80.6	82.2	1.6
93.3	93.4	93.3	0.1
107.2	108.4	112.8	4.4
121.1	121.8	118.3	3.5

(1) Wax Melt Temp. is the melt temperature of the wax, as noted by the supplier
(2) Wax Melt Calibration is the temperature at which the wax actually was observed to melt
(3) Sealing Bar Temp. to Melt is the sealing bar temperature (read-out on controls) that was necessary to melt the wax at the film interface
(4) Difference (Tm-Tb) is the percent difference between the Tm of the wax, and the sealing bar temperature to get the interface to melt.

Note: All other conditions were as per the Hot Tack Pilot Test.

TABLE 11: Theoretical Calculation Data: 4.5% VA only

Dwell Time (ms)	Sealing Bar Temperature (C)	Temperature at Interface (C)
10	105	73
100	105	98.9
200	105	104.5

Table 12: Heat Seal Pilot Test Data
(4.5% Vinyl Acetate Film)
(All tests are run with five replicates)
(Seal Average is in force per seal width)

A. Temperature = 88 Degrees Celsius (Coded 1)

Pressure (a)	Dwell Time(b)	Seal Average (g/in)	Standard Deviation (g/in)	Break (c)
2	1	7.718	20.40	1
2	2	36.77	6.360	1
2	3	88.08	4.086	1
3	1	66.74	6.810	1
3	2	86.71	20.88	1
3	3	107.0	4.086	1
1	1	113.0	5.900	1
1	2	98.97	11.80	1
1	3	77.18	16.70	1

B. Temperature = 94 Degrees Celsius (Coded 2)

Pressure (a)	Dwell Time(b)	Seal Average (g/in)	Standard Deviation (g/in)	Break (c)
2	1	26.33	26.01	2
2	2	57.66	30.55	2
2	3	5.448	3.53	2
3	1	22.25	2.69	2
3	2	79.00	28.10	2
3	3	4.086	3.6	2
1	1	34.96	29.01	2
1	2	13.62	3.54	2
1	3	25.88	3.80	2

Table 12 (cont'd)

C. Temperature = 100 Degrees Celsius (Coded 3)

Pressure (a)	Dwell Time(b)	Seal Average (g/in)	Standard Deviation (g/in)	Break (c)
1	1	668.71	29.50	1
1	2	1724.3	18.61	3
1	3	1786.0	86.26	3
2	1	1546.8	126.7	2
2	2	1734.7	96.70	3
2	3	1770.6	100.8	3
3	1	2088.9	82.17	3
3	2	1741.1	76.27	3
3	3	3.835	81.27	3

D. Temperature = 105 Degrees Celsius (Coded 4)

Pressure (a)	Dwell Time(b)	Seal Average (g/in)	Standard Deviation (g/in)	Break (c)
1	1	676.91	123.3	2
1	2	1041.0	28.15	2
1	3	1140.9	126.7	2
2	1	792.23	69.46	2
2	2	1167.7	88.08	2
2	3	1157.2	36.32	2
3	1	1045.1	74.46	2
3	2	926.20	44.95	2
3	3	1105.9	18.61	2

Table 12 (cont'd)

E. Temperature = 109 Degrees Celsius (Coded 5)

Pressure (a)	Dwell Time(b)	Seal Average (g/in)	Standard Deviation (g/in)	Break (c)
1	1	1582.2	257.0	2
1	2	1865.0	82.62	3
1	3	1691.6	65.83	3
2	1	1786.9	130.3	3
2	2	1799.7	78.10	3
2	3	1703.0	186.1	2
3	1	1872.3	127.6	3
3	2	1721.1	64.47	3
3	3	1714.8	224.7	2

(a) Pressure (pounds per square inch):

Code	Value
1	10 psi
2	50 psi
3	500 psi

(b) Dwell Time (milliseconds)

Code	Value
1	200 ms
2	500 ms
3	1800 ms

(c) Break denotes the type of break failure

Code	Value
1	Peel
2	Across Seal Tear
3	Break

Table 13. Heat Seal Experiment with all Films
(All tests are run with five replicates)

A. Film A - 0% VA

Sealing Conditions		Seal Strength Results	
Temp. (a)	Dwell Time (b)	Average (g/in)	Standard Deviation
1	1	1096.0	143.9
1	2	1338.4	43.13
2	1	1545.4	26.79
2	2	1606.3	31.78
3	1	1564.0	23.15
3	2	1516.8	53.12
4	1	1533.6	44.95
4	2	1493.7	88.98

B. Film B - 4.5%VA

Sealing Conditions		Seal Strength Results	
Temp. (a)	Dwell Time (b)	Average (g/in)	Standard Deviation
1	1	537.1	37.23
1	2	576.6	27.69
2	1	1668.9	61.74
2	2	1638.0	92.62
3	1	1801.5	80.36
3	2	1421.9	81.72
4	1	1580.4	314.2
4	2	1550.7	191.6

Table 13 (cont'd)

C. Film C - 7.0%VA

Sealing Conditions		Seal Strength Results	
Temp. (a)	Dwell Time (b)	Average (g/in)	Standard Deviation
1	1	562.96	386.8
1	2	558.87	33.14
2	1	1645.8	71.73
2	2	1667.5	116.7
3	1	1758.8	133.0
3	2	1822.8	169.3
4	1	1737.5	138.9
4	2	1813.3	126.2

D. Film D - 9.0%VA

Sealing Conditions		Seal Strength Results	
Temp. (a)	Dwell Time (b)	Average (g/in)	Standard Deviation
1	1	712.8	17.25
1	2	752.3	44.95
2	1	1833.7	202.0
2	2	1756.5	140.7
3	1	1709.8	58.11
3	2	1735.2	275.6
4	1	1692.5	97.61
4	2	1503.6	61.29

Table 13 (cont'd)

E. Film E - 19.0%VA

Sealing Conditions		Seal Strength Results	
Temp. (a)	Dwell Time (b)	Average (g/in)	Standard Deviation
1	1	136.2	15.44
1	2	156.6	19.98
2	1	603.4	39.04
2	2	694.2	60.84
3	1	1269.8	61.74
3	2	1321.6	82.62
4	1	1455.5	87.17
4	2	1526.0	157.0
5	1	1604.0	61.74
5	2	1419.2	92.62
6	1	1621.2	98.16
6	2	1347.9	129.8

Table 13 (cont'd)

F. Film F - 29.0%VA

Sealing Conditions		Seal Strength Results	
Temp. (a)	Dwell Time (b)	Average (g/in)	Standard Deviation
1	1	285.1	76.26
1	2	309.71	111.2
2	1	577.5	114.4
2	2	646.5	621.2
3	1	934.79	155.3
3	2	966.1	109.4
4	1	1024.2	159.8
4	2	1009.2	54.93
5	1	1284.8	149.8
5	2	1204.5	99.88
6	1	1284.8	149.8
6	2	1177.7	219.3

(a) Temperature (deg. C): See Table 14
(b) Dwell Time (ms): See Table 14
(c) Seal Strength is in grams/inch, see table 14 for g/in^2 in tensile strength.

Table 14. Heat Seal Experiment - Code For Actual Temperature, Dwell Time, and Pressure Conditions.

(A) Actual Temperature Conditions in Degrees Celsius

Film Sample (%VA)	Temperature 1	2	3	4	5	6
0	107	113	120	132	(a)	(a)
4.5	98	105	111	122	(a)	(a)
7	97	102	107	118	(a)	(a)
9	94	101	106	116	(a)	(a)
19	75	85	96	106	115	121
29	62	70	87	96	104	121

(a) These extra temperature conditions were not needed.

(B) Actual Dwell Time Conditions in Milliseconds.

Temp.	Film Sample (%VA) All
1	200
2	500
3	1800

Table 15. Maximum Heat Seal Strengths for each Film. (All tests were run with five replicates)

Film Sample (%VA)	Conditions Temp. (a)	Dwell Time (b)	Break Factor Average (c)	Standard Deviation (c)	Tensile Strength Average (d)	Standard Deviation (d)
0	2	2	1609.9	32.0	1134.7	22.6
4.5	3	1	1805.3	80.5	1148.0	22.8
7.0	3	2	1826.8	170.2	1098.8	102.4
9.0	2	1	1837.6	202.5	1286.7	141.9
19	6	1	1625	98.3	961.2	58.1
29	6	1	1287.5	149.7	611.8	94.9

(a) Temperature: see table 14.
(b) Dwell Time: see table 14.
(c) Break Factor in Grams
(d) Tensile Strength in Grams per Square Inch

Table 16. Hot Tack Pilot Test Data

A. Temperature = 100 Degrees Celsius (Code 3)

Pressure (psi)(a)	Dwell Time (ms)(b)	Average Seal Strength (g/in)
3	3	0
3	2	0
3	1	0
2	3	0
2	2	0
2	1	0
1	3	0
1	2	0
1	1	0

Table 16 (cont'd)

B. Temperature = 105 Degrees Celsius (Code 4)

Pressure (psi)(a)	Dwell Time (ms)(b)	Average Seal Strength (g/in)
3	3	100
3	2	50
3	1	75
2	3	50
2	2	50
2	1	50
1	3	50
1	2	50
1	1	50

C. Temperature = 109 Degrees Celsius (Code 5)

Pressure (psi)(a)	Dwell Time (ms)(b)	Average Seal Strength (g/in)
3	3	25
3	2	25
3	1	25
2	3	25
2	2	25
2	1	25
1	3	25
1	2	25
1	1	25

(a) Pressure in Pounds per Square Inch

Code	Value
1	10
2	50
3	500

(b) Dwell Time in Milliseconds

Code	Value
1	200
2	500
3	1800

(c) Seal Strength is noted in grams/inch

Table 17. Hot Tack Experiment Maximum Seal Values

(A) Break Factor and Sealing Bar Temperature
(grams @ Degrees Celsius)

Film Sample (%VA)	Method A (a)	Method B (b)	Method C (c)
0	25 @107	25 @118	25 @118
4.5	50 @107	25 @104	25 @104
7.0	50 @102	NA @107	11.2 @107
9.0	25 @102-107	NA @107	16.9 @102
19	25 @ 85-96	50 @ 91	50 @ 91
29	NA @ 71-77	75 @ 77	80 @ 77

(B) Tensile Strength and Sealing Bar Temperature
(grams/in @ Degrees Celsius)

Film Sample (%VA)	Method A (a)	Method B (b)	Method C (c)
0	17.6 @107	7.6 @118	17.6 @118
4.5	29.8 @107	14.9 @107	14.9 @104
7.0	35.6 @102	NA @107	8.0 @107
9.0	10.6 @102-107	NA @107	10.2 @102
19	12.3 @ 85-96	35.0 @ 91	35.0 @ 91
29	NA @ 71-77	44.4 @ 77	47.3 @ 77

(a) Method A: James River Corporation Test Method
(b) Method B: Modified James River Corporation Test Method (With Tape)
(c) Method C: Modified Method B (10g weight added)

Table 18. Correlation Coefficient Values with Hot Tack and Heat Seal Data

Characteristic Parameter	Heat Seal	Hot Tack
Heat Seal	NA	-0.92(.0095)
Hot Tack	-0.92(.0095)	NA
VA (a)	*-0.86(.0267)	* 0.86(.0266)
TENSILE (b)	-0.76(.0809)	0.73(.1002)
MFI (c)	0.43(.3907)	0.25(.6345)
DENSITY (d)	-0.71(.1169)	0.79(.0641)
DSC%C (e)	0.75(.0874)	0.73(.0974)
ONSET (f)	* 0.86(.0298)	*-0.85(.0342)
Tm (g)	* 0.88(.0218)	*-0.87(.0254)
Tg (h)	* 0.88(.0218)	*-0.87(.0254)
Mw (i)	0.54(.2664)	-0.36(.4825)
Mn (j)	0.18(.7371)	-0.46(.3540)
Mv (k)	* 0.85(.0330)	*-0.90(.0154)
Mz (l)	0.75(.0850)	-0.75(.0845)
MWD (m)	0.31(.5491)	-0.06(.9110)

Note: A negative confidence level denotes an inverse relationship.

*: Denotes a statistical significance at level 0.05.

(a) VA is the percent vinyl acetate in the test samples
(b) TENSILE is the tensile strength of the test samples
(c) MFI: the melt flow index values of the test samples
(d) Density is the DGC generated density of the test samples
(e) DSC%C is the DSC generated percent crystallinity values of the test samples
(f) ONSET is the DSC generated onset temperature of the test samples
(g) Tm is the DSC generated values of melt temperature of the test samples
(h) Tg is the DSC generated values of glass-transition temperature of the test samples
(i) Mw is the weight average molecular weight of the test samples
(j) Mn is the number average molecular weight of the test samples
(k) Mv is the viscosity average molecular weight of the test samples
(l) Mz is the Z-average, or high molecular weight average molecular weight of the test sample
(m) MWD is the molecular weight distribution of the test samples

Table 19. Summary of All Properties of the Ethylene-Vinyl Acetate Co-Polymer Films

Property	0	4.5	7.0	9.0	19	29
Maximum Heat Seal Strength (g/in)	1132	1143	1096	1284	959	610
Maximum Hot Tack Strength (g/in)	17.62	17.78	6.78	11.83	29.57	48.03
Break Factor (g/seal width)	2277	2748	2799	3135	3824	4398
Melt Temperature (deg C)	113.5	104.9	102	101	84.8	69.8
Percent Crystallinity	33.7	25.6	21.9	21.7	11.1	8.7
Density (g/cm^3)	0.924	0.925	0.926	0.932	0.940	0.941
Number Average Molecular Mass (Mn x 10^4)	1.468	2.56	3.028	0.69	1.351	1.089
Weight Average Molecular Mass (Mw x 10^5)	1.552	1.726	1.796	1.7126	1.660	1.2079
Viscosity Average Molecular Mass (Mv x 10^5)	1.551	1.725	1.796	1.712	1.660	1.2076
Z Average Molecular Mass (Mz x 10^5)	3.012	4.698	4.843	5.2	4.364	2.387
Dispersion Index (Dm=Mw/Mn)	10.59	6.74	5.93	24.72	12.32	11.09
Melt Flow Index (g/10 min)	1.53	1.61	1.324	2.72	1.067	1.55

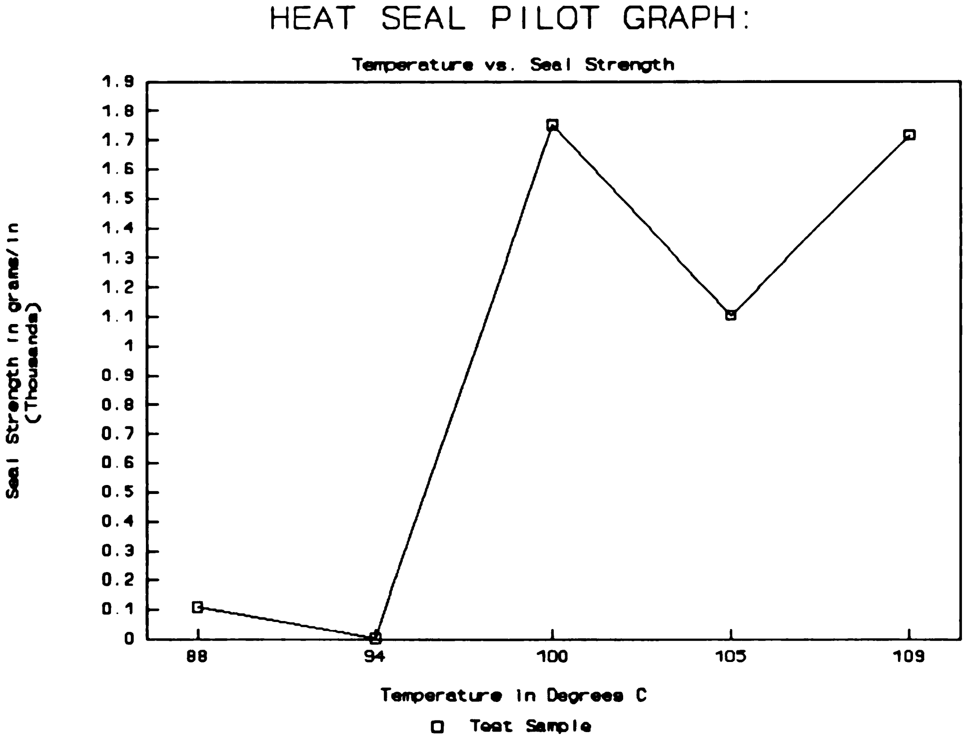

Figure 1: Heat Seal Pilot: Temperature versus Seal Strength

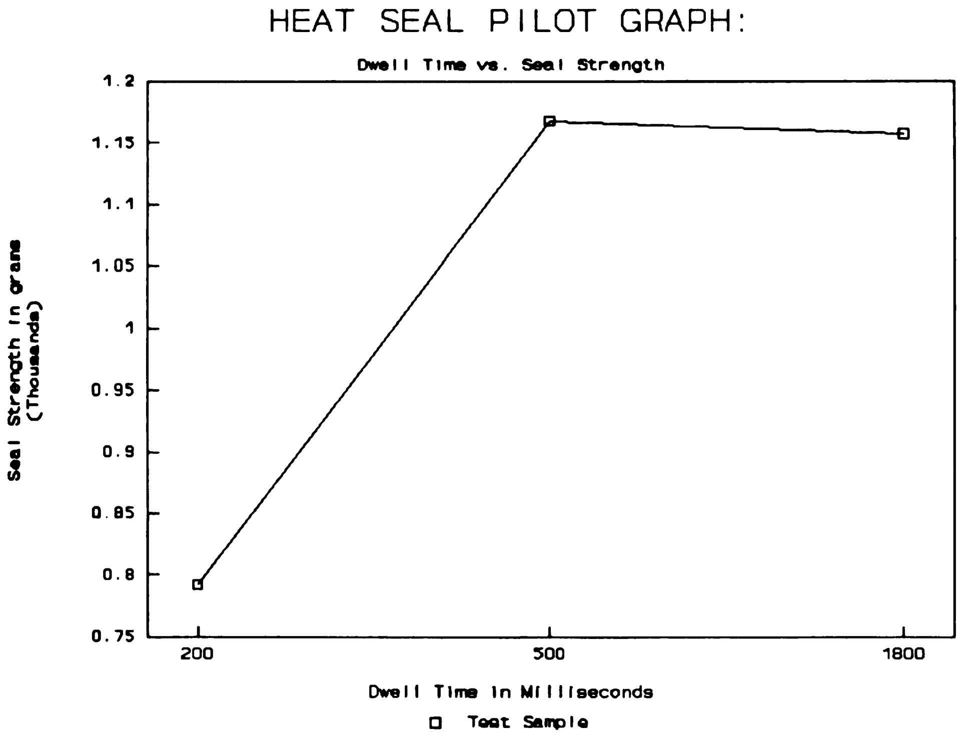

Figure 2: Heat Seal Pilot: Dwell Time versus Seal Strength

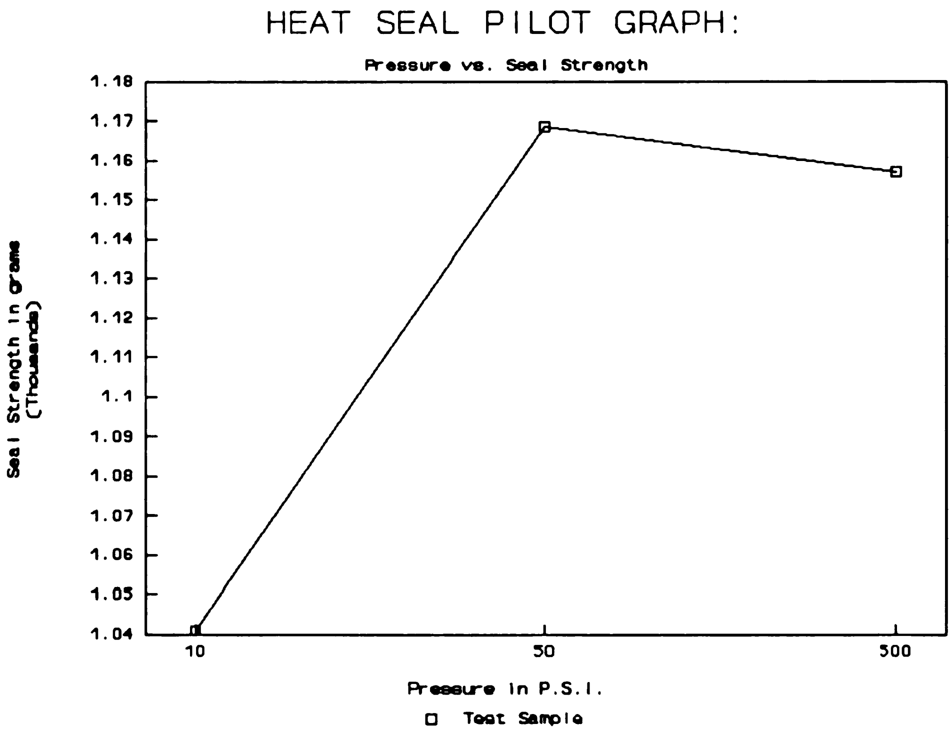

Figure 3: Heat Seal Pilot: Pressure versus Seal Strength

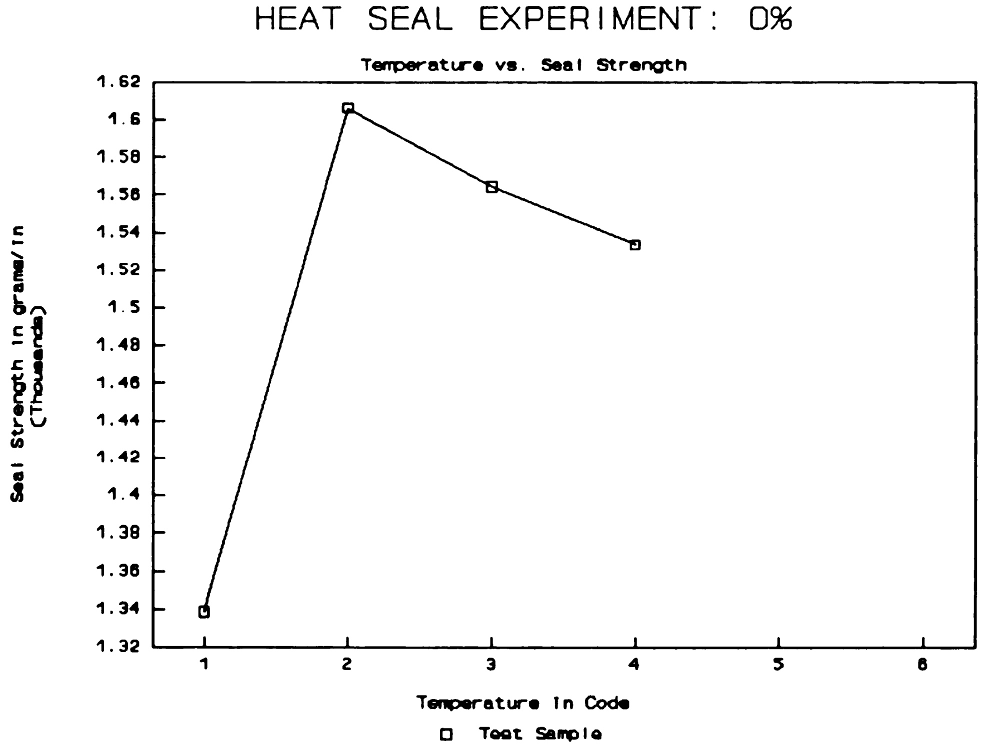

Figure 4: Heat Seal Experiment 0% VA: Temperature versus Seal Strength

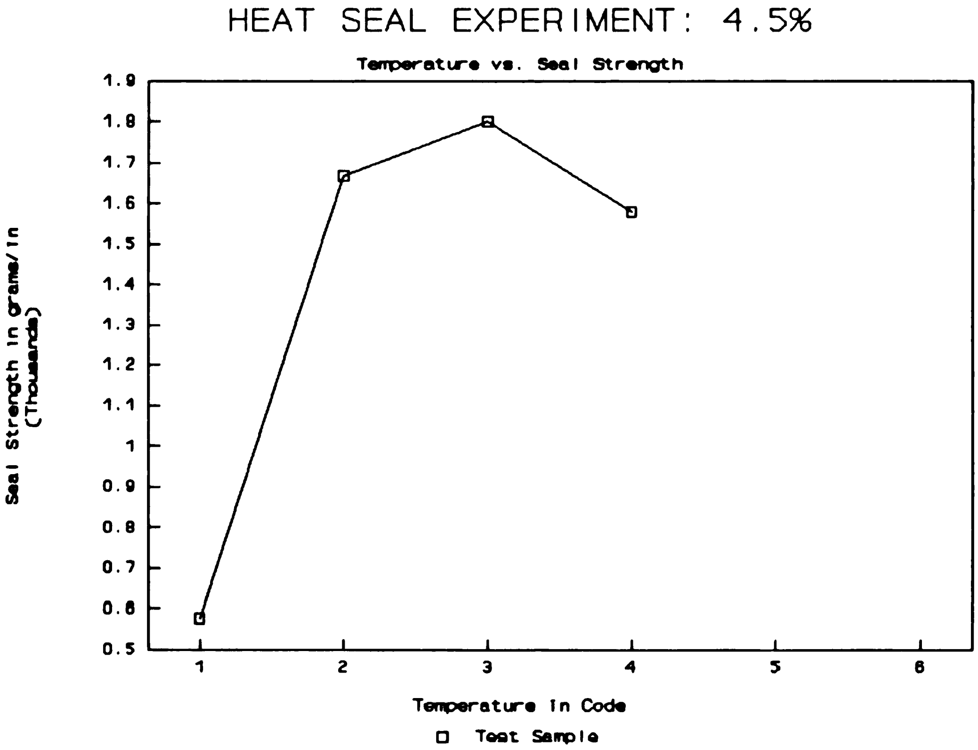

Figure 5: Heat Seal Experiment 4.5% VA: Temperature versus Seal Strength

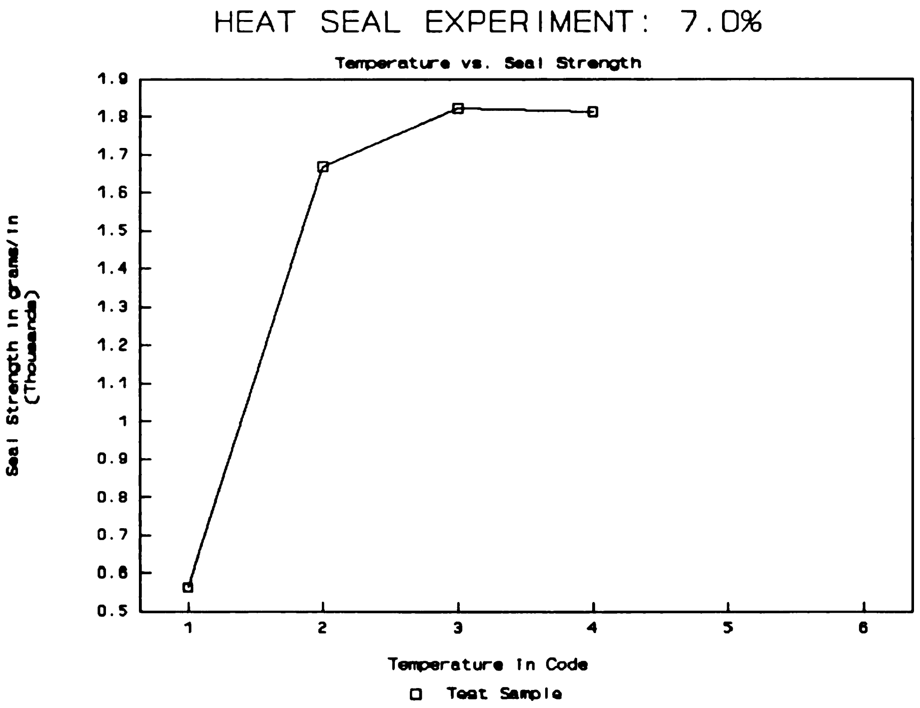

Figure 6: Heat Seal Experiment 7.0% VA: Temperature versus Seal Strength

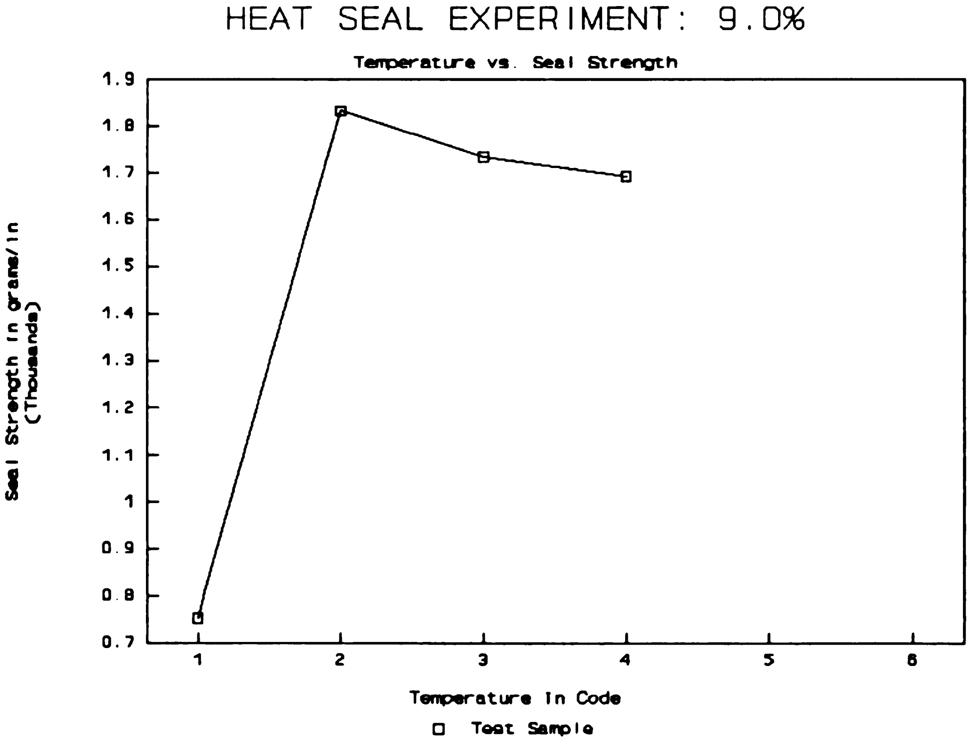

Figure 7: Heat Seal Experiment 9.0% VA: Temperature versus Seal Strength

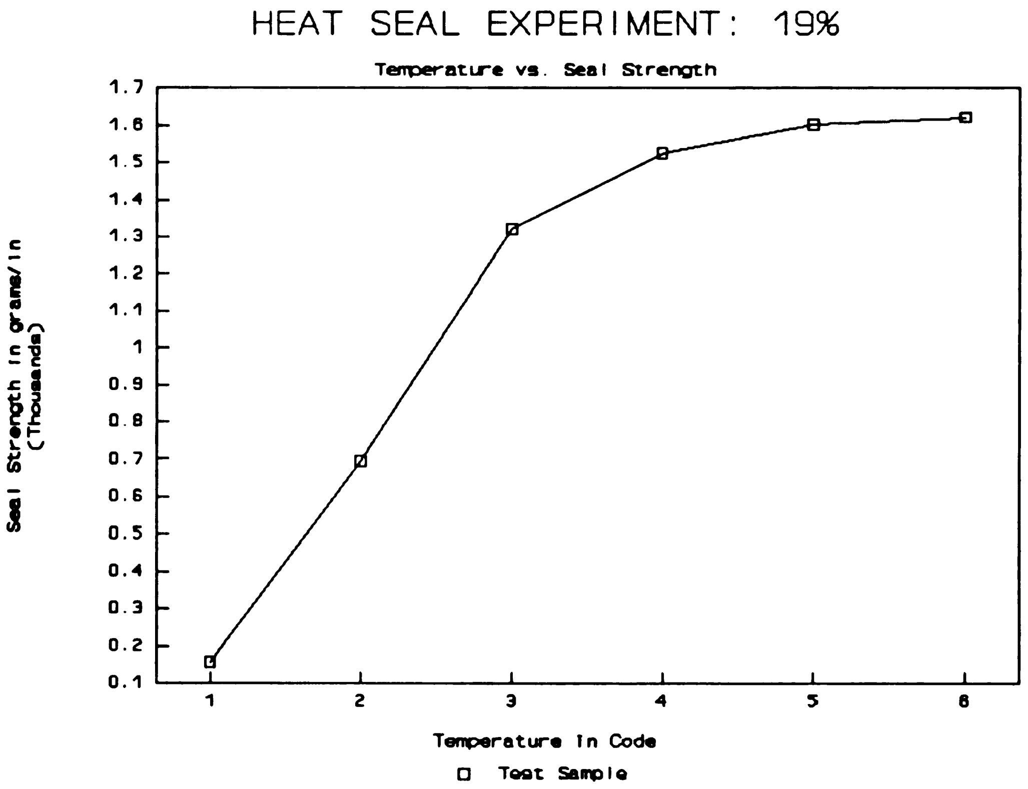

Figure 8: Heat Seal Experiment 19% VA: Temp. v. Seal Strength

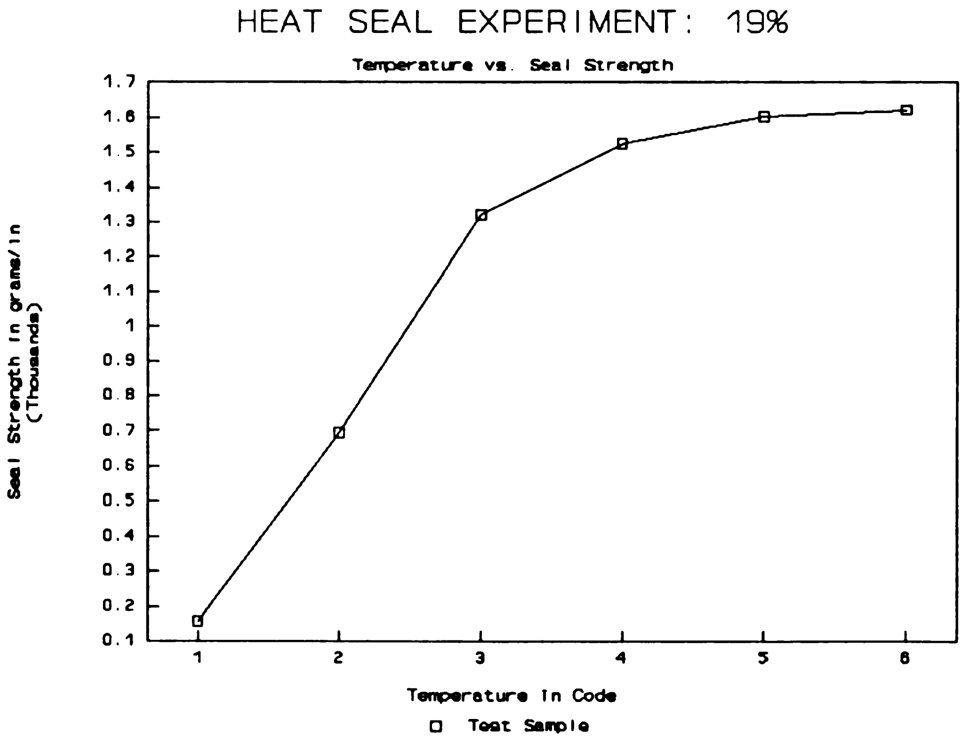

Figure 8: Heat Seal Experiment 19% VA: Temp. v. Seal Strength

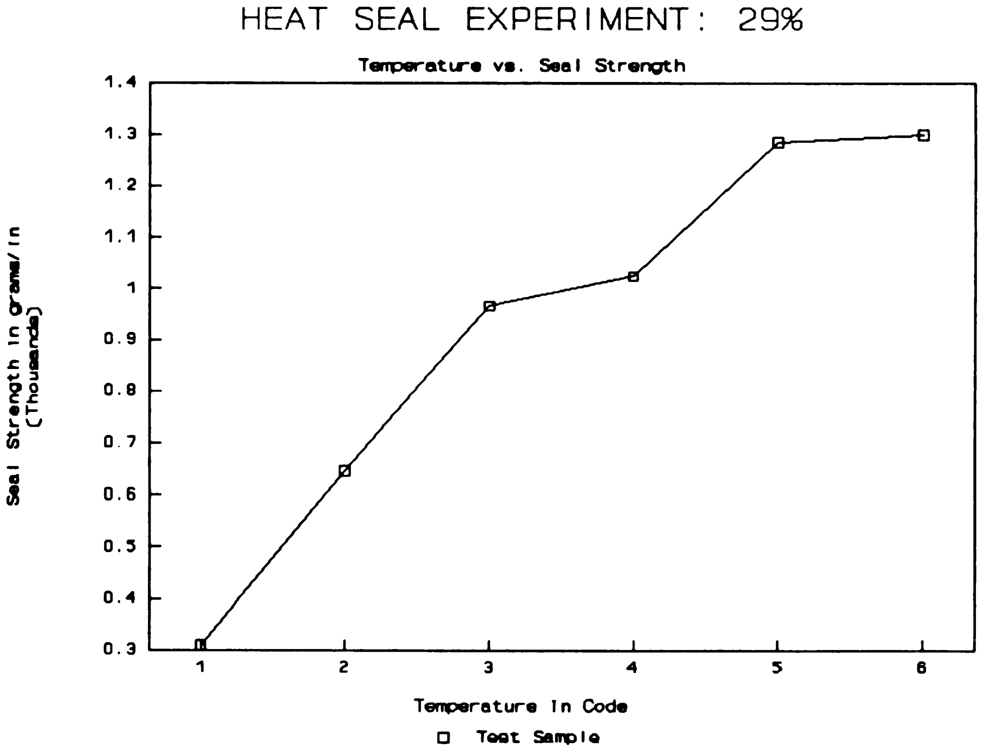

Figure 9: Heat Seal Experiment 29% VA: Temperature versus Seal Strength

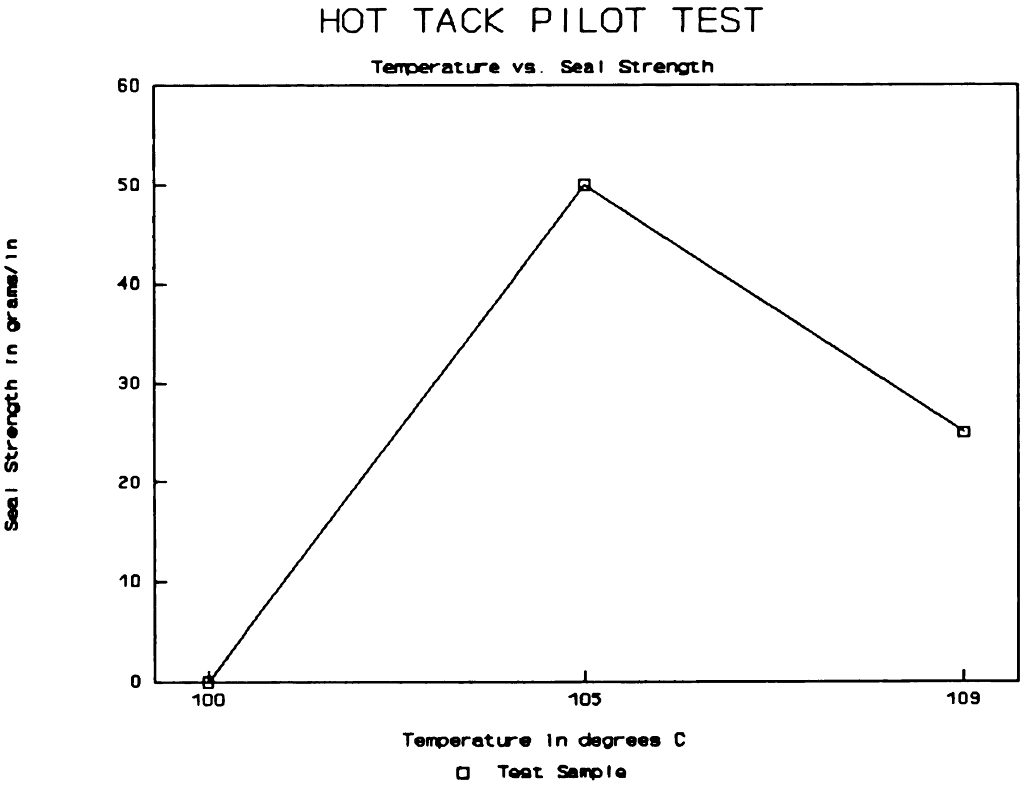

Figure 10: Hot Tack Pilot: Temperature versus Hot Tack Strength

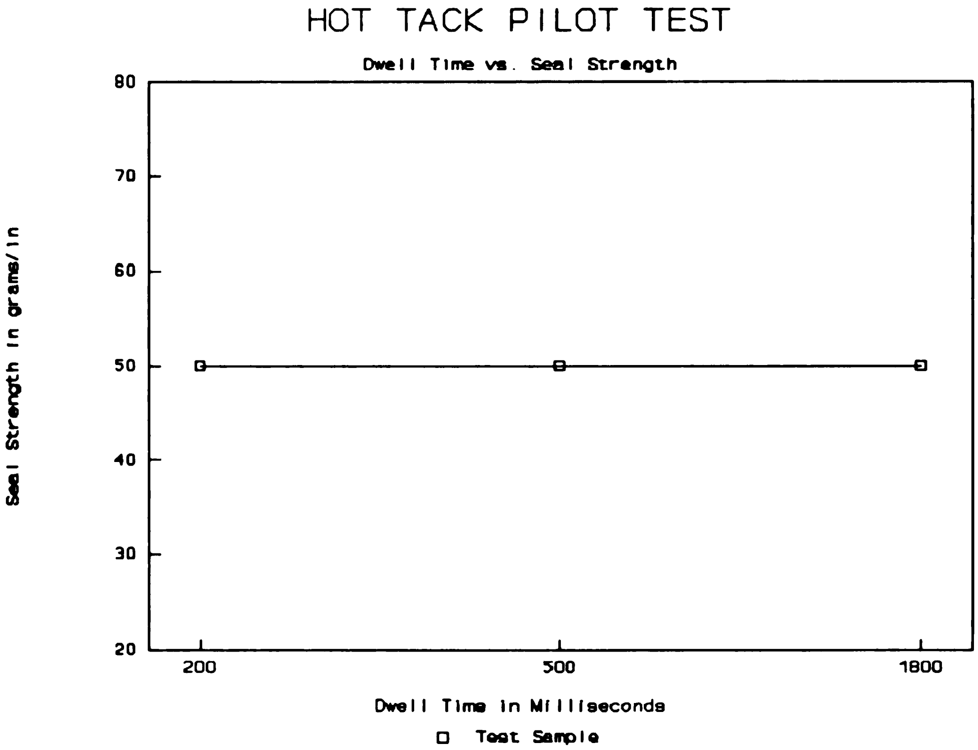

Figure 11: Hot Tack Pilot: Dwell Time versus Hot Tack Strength

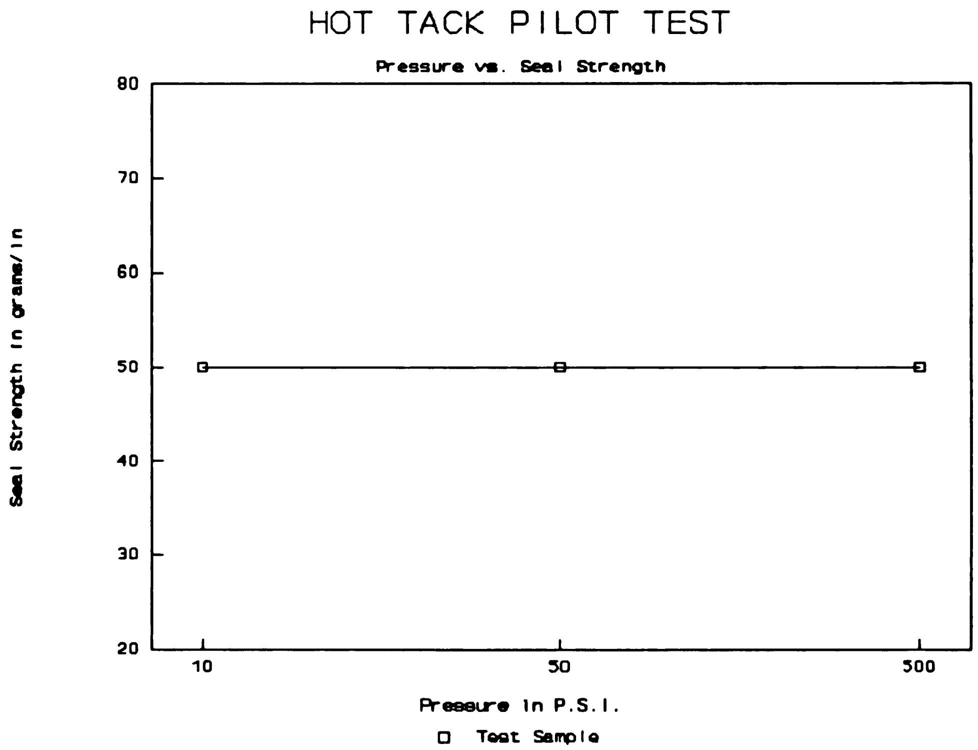

Figure 12: Hot Tack Pilot: Pressure versus Hot Tack Strength

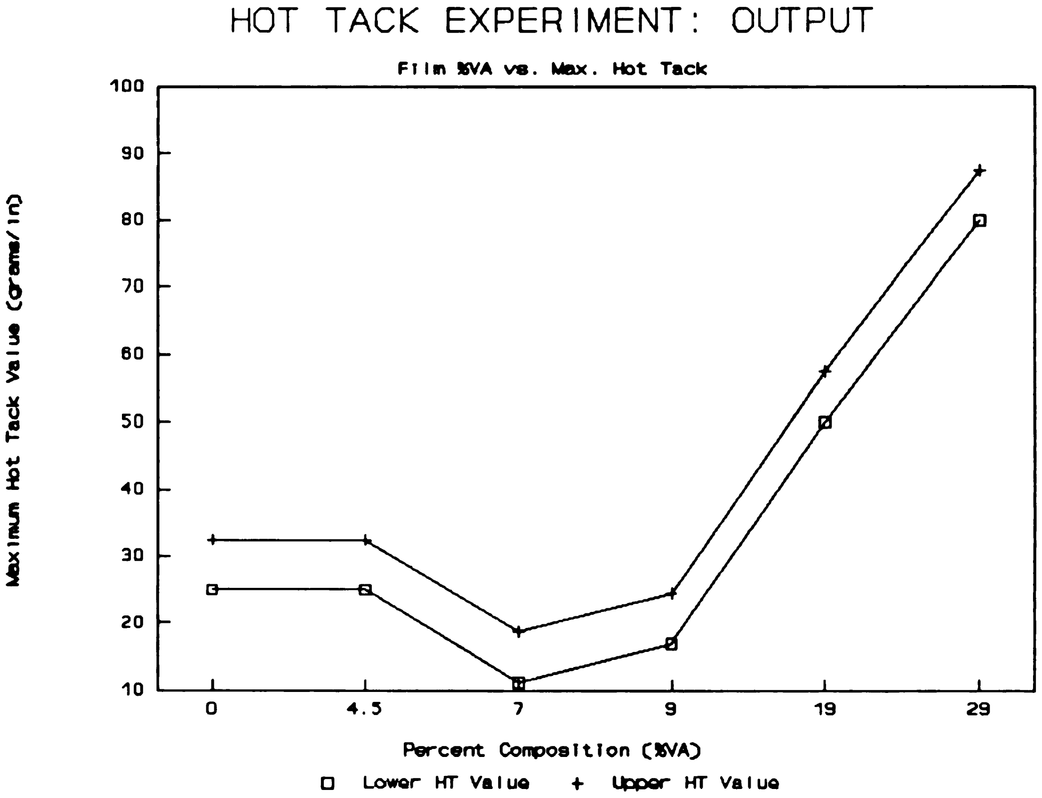

Figure 13: Hot Tack Experiment: Percent Composition versus Hot Tack Maximum

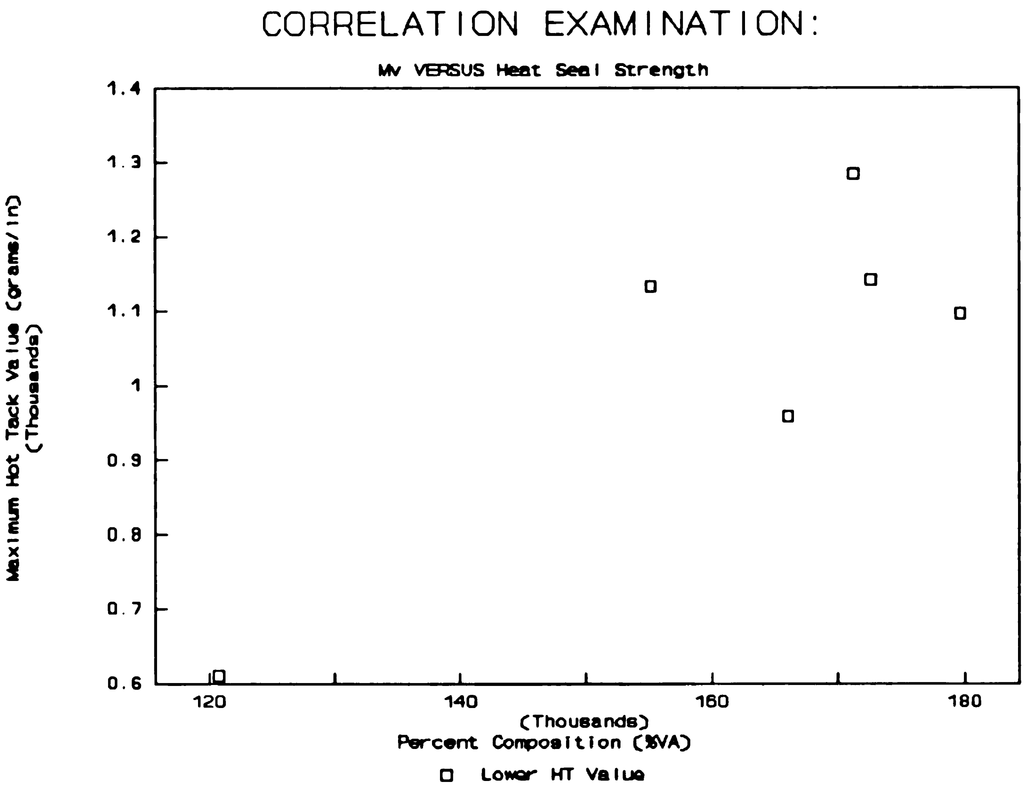

Figure 14: Correlation Examination: Mv vs. Heat Seal

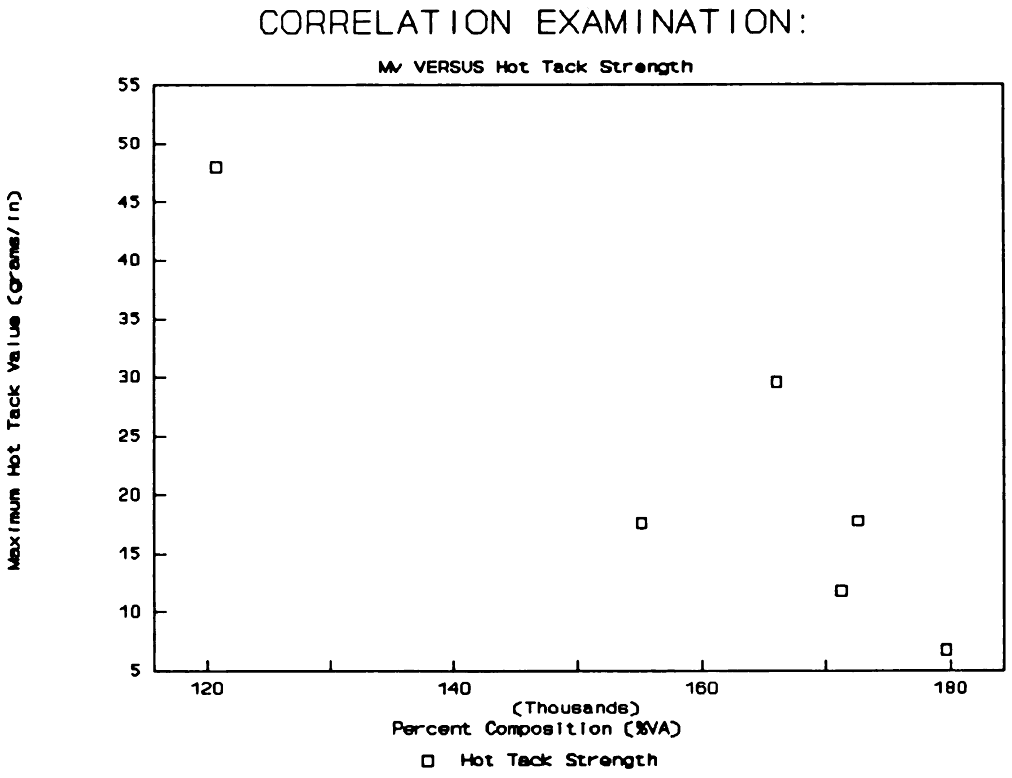

Figure 15: Correlation Examination: Mv vs. Hot Tack

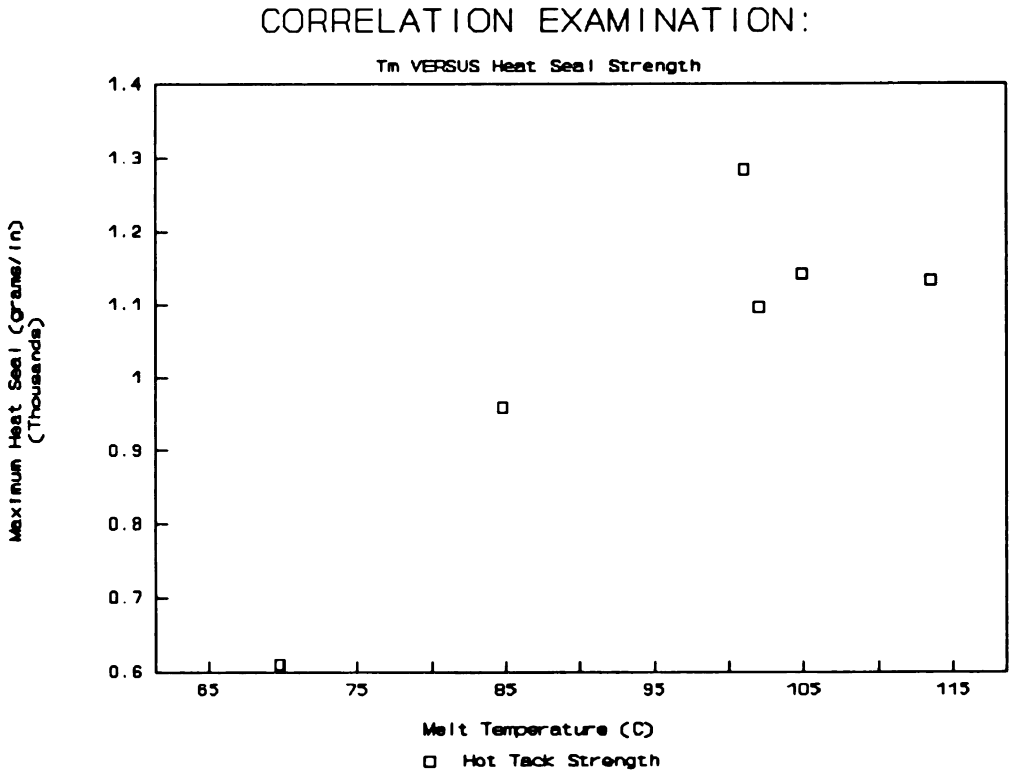

Figure 16: Correlation Examination: Tm vs. Heat Seal

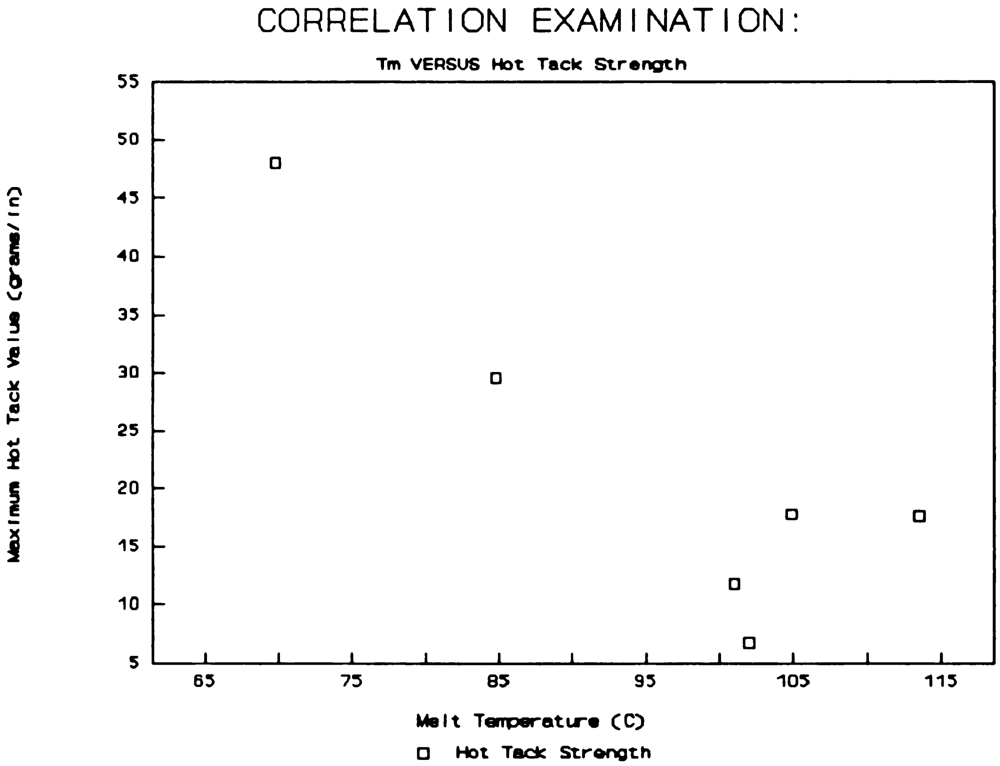

Figure 17: Correlation Examination: Tm vs. Hot Tack

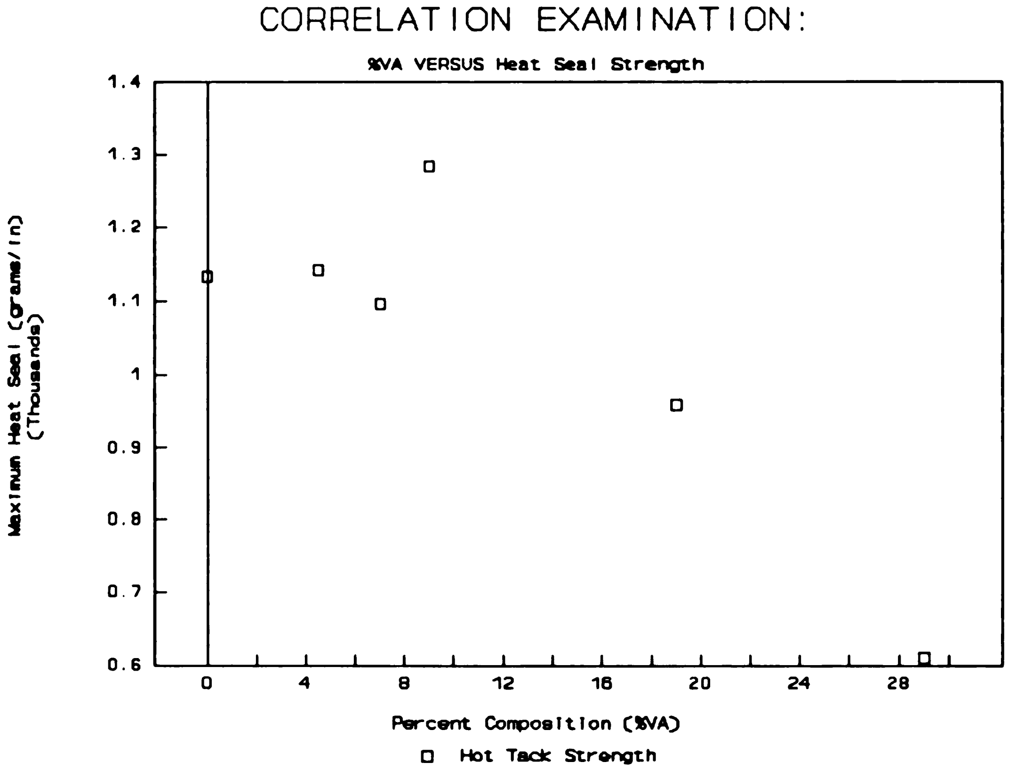

Figure 18: Correlation Examination: %VA vs. Heat Seal

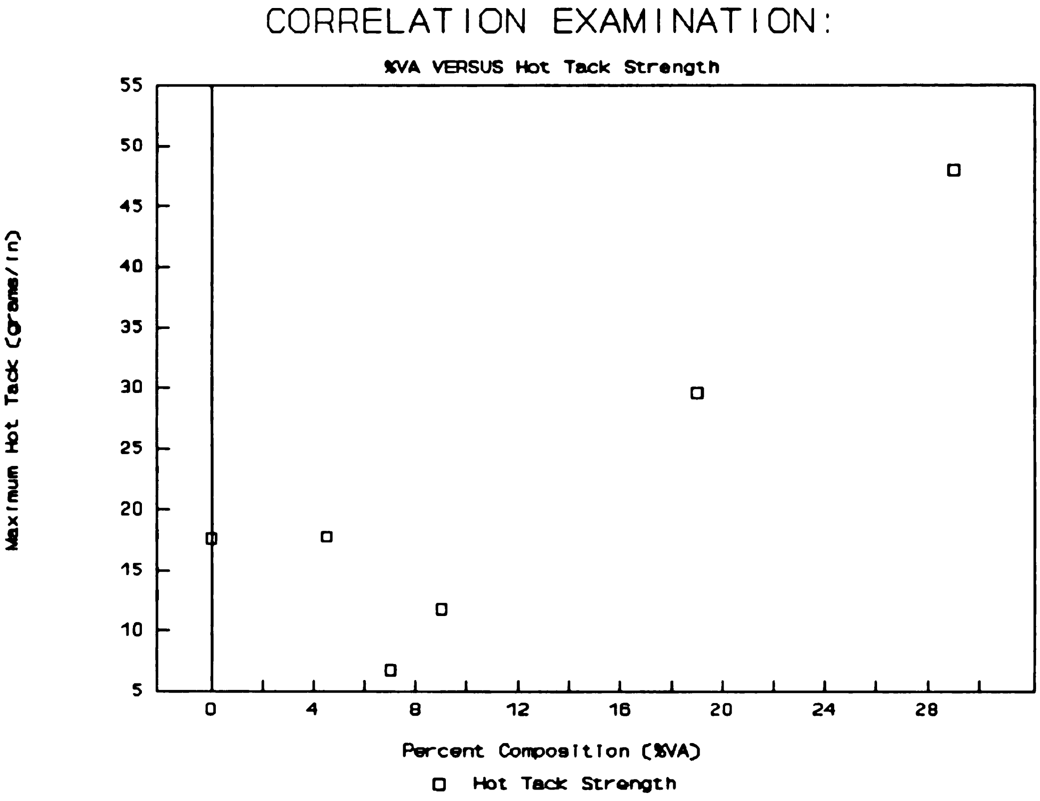

Figure 19: Correlation Examination: %VA vs. Hot Tack

7.0 DISCUSSION

7.01 Polymer Characterization

7.01.01 Composition (Percent Vinyl Acetate)

The supplier provided the percent vinyl acetate composition data for the respective test samples. Analysis was based on an Infrared Spectrophotometric procedure.

7.01.02 Melt Flow Index (MFI or MI)

There are two procedures described in the literature (ASTM D-1238) for determining melt flow index of polymer resins. Procedure A is a "manual cutoff operation based on time and is used for materials having a melt flow index that falls between 0.15 and 50 g/10 minute." Procedure B is for polymers with a very high melt index, and is an automatically timed experiment. For the resin samples evaluated in the present study Procedure A was employed (ASTM D-1238,Sec.3.2,1979).

For ASTM statistical analysis of the MFI test unit, and correlation to the supplier's measurements, ten replications were performed on the polyethylene resin. All the data was within ASTM specifications for equipment variance, 7%. For the vinyl acetate copolymer samples, 3 replicates were performed.

The ASTM standard procedure recommends different temperatures for different polymers. For example, 190 degrees C is standard for polyethylene, and 150 degrees C is standard for pure polyvinyl acetate. The defined procedures for polyethylene and polyvinyl acetate do not, however, note specific percent compositions. For all samples except the 29% VA copolymer, the lower temperature test was not valid according to ASTM D-1238. The extrudite was not equal to or greater than one gram. Also, the 29% vinyl acetate experienced thermal degradation at 190 degrees C. Therefore, this sample was tested at 150 degrees C.

The 9 percent vinyl acetate copolymer exhibited a very high melt flow index with respect to the other resins. This was acceptable, and can be correlated with the molecular weight distribution data.

7.01.03 Tensile Strength (No seal)

This data is discussed in the heat seal and hot tack sections. The strength variations, are additive effects of both polyethylene and ethylene-vinyl acetate copolymer characteristics. To obtain correlation between the tensile materials test, and the seal tests, the "tensile strength (in grams per inch squared)" and <u>not</u> the "break factor (in grams)" was used. This is reasonable for correlations since the film did exhibit significant thickness differences.

7.01.04 Density Gradient Column (DGC)

The density data is not unexpected, considering that as the polymer increases in VA content, the percent crystallinity decreases, but the molecular weight of the modifying co-monomer is greater. The increasing VA content thus results in the observed increase in polymer density.

7.01.05 Differential Scanning Calorimetry (DSC)

The two points on the melt profile graph that are derived from this experiment are the onset and the melt temperature. The heat of fusion is computer generated from the DSC output. Regarding the estimation of percent crystallinity from the heat of fusion, it was assumed that a value of 68.4 calories per gram for the heat of fusion of a 100% crystalline polyethylene (Troedel,1984) sample was valid for the copolymer samples as well. This was also recommended by the film supplier.

The percent crystallinity appeared quite low for the low percent EVA compositions (8% to 33%). The manufacturer was consulted, and the data were accepted as being within a reasonable range.

7.01.06 Gel Permeation Chromatography (GPC)

Any relationship between GPC data and other polymer properties characterized are described in the correlation section of the Discussion. All data was within acceptable ranges for the films tested.

7.01.07 Thickness

The thickness results did show a significant difference between films. The manufacturer has lab equipment capabilities to accurately control the width, but not the thickness of the film.

7.02 Seal Optimization

7.02.01 Pre-Research

For polymer films, the seal strength is an important property concerning package integrity and includes both the cold seal strength, and the hot seal strength. For a cold seal, failure is a total break of the seal system. For hot tack, failure is defined as a peel of more than 1/8" (even though in some cases a peel seal is not formed).

A cold seal is a polymer film seal that has been allowed to cool to ambient temperature. The cold seal strength is related to the ability of a seal to maintain its physical integrity during product distribution, and its aesthetic integrity for consumer preference.

A hot seal is a polymer film seal that is still molten after sealing, or cooled for a controlled period of time. The hot seal strength relates to the ability of a seal to maintain its integrity when stressed, while the seal is still molten.

This property is referred to as tack, or the stickiness of the seal, while the film is still molten.

The hot seal, or hot tack strength at differing lengths of delay time after sealing, is of direct importance while filling a product into a pouch on a form/fill/seal machine (Szemplensiki,1984).

A form/fill/seal machine unrolls film directly from roll stock, forms a pouch, seals the pouch, and either while the sealing bars are still forming the seal or just after, product is placed in the pouch. The sealing bars simultaneously form the closing seal on the filled pouch, and form the bottom seal on the new pouch. The product can be put into the pouch with great force, even while the seal is still molten. Thus, the hot tack is an important polymer characteristic.

Since the basic conditions of these two experiments are different (one molten and one cold), the seal equipment parameters, and the resulting maximum strength may be very different for the same film.

Specifically, the cold seal strength is derived by the fusion of two individual layers of film. The film is heated to a temperature which allows molten polymer to flow at the interface, and thus, a single structure is formed from the two individual film layers. Some films (polyethylenes) are completely fusible, and thus, the seal interface dissolves. Other films will seal, but maintain their interfacial integrity. They maintain an interface, even though they are sealed together.

Selected parameters that effect cold seal strength may also affect hot seal strength. The parameters of interest are discussed below.

For example, the temperature of the sealing bars is a parameter that influences both hot seal and cold seal. Dwell time for contact of the sealing bars is also a parameter which can influence both hot seal and cold seal strength. The pressure that is exerted on the film by the sealing bars can also effect both hot seal and cold seal bond strength. Unique to the hot seal process, is the delay time between seal formation and testing. This effect is directly proportional to the cooling of the seal from molten to ambient conditions. In a cold seal this is five minutes or more depending upon the test (ASTM F88,1985). Delay time is the single parameter that differentiates between a hot seal and a cold seal. The seal testing rate, or the speed that the seal is pulled apart, is also a parameter of the hot seal test, and it is of specific importance on the Rucker Hot Tack Attachment. The cold seal is tested at a constant rate that is a function of the tensile testing equipment and can be selected by the operator. Due to the mechanics of the Rucker hot tack testing equipment, the rate of testing speed is variable and uncontrolled.

ASTM task group 2.33.03 notes that the exact calibration of the seal parameters (temperature, dwell time, and pressure) must be carried out with a separate piece of equipment. This ensures that the variables set are the variables applied.

7.02.01.01 Temperature Verification

To verify the interface temperature, a series of heat sensitive waxes was used. The manufacturer calibrated temperature sensitive waxes were placed at the interface of the film, the film sealed, and the wax melt temperature observed.

The waxes were very different in texture. Potentially, some waxes were affected by age and drying. This is noted by the 107 degree C wax (as per manufacturer) exhibiting a higher melt temperature, and the 121 degree C wax exhibiting a lower melt temperature than specified (see Table 10).

The difference in sealing bar and wax calibration melt temperature (at 500 ms dwell time), ranged from 0.1% to 3.4% difference. The average was 1.7% difference. Thus, at the heat seal conditions set, the interface temperature of the seal agreed well with the sealing bar temperature. Based on these findings it was concluded that the sealing bar temperature is a good estimate of the interface temperature for all films tested.

Theoretical calculations (Table 11) showed that effective seals were formed at dwell times of 200 milliseconds (ms) to 1800 ms. At a 200 ms dwell time, the interface temperature rises to within 0.5% of the sealing bar temperature. In conclusion, for the dwell times used, heat transfer is not considered a major factor in influencing the interface temperature between the sealing bar and heat sealant film.

7.02.01.02 Dwell Time and Pressure Verification

Regarding dwell time and pressure, this calibration is beyond the scope of the present study. The instrument calibration will be accepted as correct and accurate.

7.02.01.03 Transition and Relaxation Temperatures

The molecular relaxation and transition temperatures determined for the respective test resins, lie far below the maximum seal strength temperature, and below the lowest recorded seal strength temperature values. Thus, these transitions and relaxations, are below the minimum sealing temperature and have little, if any, effect on seals within the sealing range evaluated (see Table 9).

7.02.02 Heat Seal Pilot Test

The pilot test was designed to obtain data and provide a means of selecting parameter levels for the principle heat seal experiment. The pilot test was performed on only one randomly selected film, 4.5 % VA. The design procedures are discussed in detail in the Experimental Design section.

To determine whether a parameter is significant (i.e. whether it has an affect on seal strength), the surface response graphs, and statistical data were evaluated.

7.02.02.01 Pressure

In the pilot test, a statistically significant plateau (peak) was obtained above a pressure of 50 psi. Since a constant seal strength value was reached at a very low pressure, there is no benefit of controlling a machine's pressure below this minimum point. In the present ASTM task group studies, pressure is treated as a constant, beyond a minimum point (60 psi). For this study, the pressure was maintained constant, at the midpoint of the plateau developed in the pilot test. From the Literature Review, the recommended pressure is 200 psi (Frito-Lay,1988; Theller,1988;etc.) (Figure 3).

7.01.03.02 Dwell Time

It was found that there was no significant benefit of controlling a production machine's dwell time at the lower values. ASTM Task Group 2.33.02 does not vary the dwell time below 500 ms. From the pilot test, a statistically significant plateau was observed between a dwell time of 200 ms to 1800 ms (see Figure 2). Unlike pressure, there is a significant benefit of sealing at the minimum dwell time to reduce manufacturing cycle time. The lab heat sealer is capable of a minimum dwell time of 90 ms. For commercial equipment, the minimum dwell time is 250 ms (James River Corp.,1989).

Since the present study is concerned specifically with the optimum conditions (and the optimum is within the plateau), the dwell times evaluated will be within the plateau region (see Experimental Design for detail of the plateau region). The recommended test dwell times and the dwell times used (James River Corp.,1988), are 500 ms and 1800 ms, respectively.

7.02.03.03 Temperature

It was found that there was a significant benefit from controlling a production machines temperature as temperature has a direct effect on seal strength (Figure 1). ASTM Task Group 2.33.02, specifically monitors the seal temperature effect on the strength of the seal.

According to the pilot test, the seal is affected by temperature from onset, to beyond the end temperature. The end temperature, is the point where the polymer is completely melted and begins to undergo thermal degradation. A satisfactory seal occurred only beyond melt temperature (Tm).

The molecular transition and relaxation temperatures, Tll and Tac (defined in the Methods Section) did not show a significant effect on the seals.

The temperature also was the major factor in effecting different modes of seal failure. The failure modes appeared to change at major transition temperatures (onset and Tm) (Theller,1988).

As previously discussed, with a dwell time above 200 ms, the heat transfer through the film was such that the seal bar temperature and the interface temperature were essentially equivalent.

The range of temperatures selected for study were chosen to show a normal type distribution curve to include an increase, plateau, and decrease in seal strength, as a function of temperature. If the surface response curve had shown the seal strength to increase and then remain at a constant value, beyond the Tm and end temperature settings, that would have been an acceptable result. From literature and DSC data, it is noted that the film undergoes thermal degradation beyond the end temperature (as noted in the Methods), and cannot form a significantly stronger seal.

Since each film has its own distinct melt profile, the individual heat seal temperatures evaluated varied according to the test films composition and thermal profile.

7.02.03.04 Replicates

From the literature (Theller,1988), it was noted that there should be a major increase in seal strength as the temperature variable was increased. Several of the tests described (Theller,1988;Frito-Lay,1989) used a small number of replicates to view the general shape (graphic trends) of the results. The large upward curve (of actual test data)resulted in the averages being representative of the general shape.

Five replicates for the pilot study.

7.02.03 Heat Seal Experiment

The objective of this study was to determine, statistically, the maximum seal strength, and the optimum heat seal variables at the seal value maximum. The variables for the heat seal experiment included the dwell time, pressure and temperature. In the pilot test studies, carried out on the 4.5% vinyl acetate film, trends that were reported in the literature were confirmed. Also, the selection of the heat seal variables (the exact points) were verified from the previously completed pilot tests.

7.02.03.01 Experimental Design

This experiment was reduced from a complete, and originally proposed 5-by-3-by-3 (temperature, dwell time, and pressure) surface response design, to a 4-by-2-by-1 surface response (see experimental design section). A response profile curve could still be obtained with four temperatures. The dwell time and pressure were found to be insignificant variables, and accepted as constant parameters for commercial applications. The validity of this reduction in response surface variables was verified by data in the pilot test, and in the literature. A maximum seal strength for each of the films, is the data that is sought by this study.

After reviewing the literature, and the results of the pilot test, it was noted that each variable would result in an increase of seal strength to a maximum, beyond which an insignificant increase or a decrease in seal strength would be observed. The variables found to affect a heat seal were: dwell time, pressure, and temperature.

7.02.03.02 Pressure

It was concluded from the literature and the results of the pilot study, that the effect of pressure would be insignificant above some minimum volume.

In a preliminary study, the effect of pressure was evaluated for all test films. For the polyethylene samples, an 11% increase in seal strength was observed for a 1000% increase in die pressure. For the 29% vinyl acetate copolymer, an 18% increase in seal strength was observed for a 1000% increase in die pressure (See Appendix A). Thus, the pressure was held at 200 psi.

7.02.03.03 Dwell Time

It was also concluded from the literature, and from the results of the pilot study, that the dwell time would be insignificant, beyond a minimum value. This parameter would be insignificant, in that a large increase in the dwell time would result in a minimal (70% increase in dwell time results in an average 3% increase in seal strength) increase in the seal strength.

The James River Corporation test method quotes applicable industry dwell times from 250 ms to 1000 ms (James River

Corp.,1988). The lowest time that the laboratory heat sealer will run is 90 ms. The lowest dwell time that was observed in the literature was 100 ms. Thus, dwell times of 200 ms and 500 ms were used.

7.02.03.04 Temperature

From Differential Scanning Calorimetry (DSC) thermal analysis, three characteristic temperature values were obtained: onset temperature, melt temperature, and end temperature. Onset temperature is the initiation of bulk polymer melt. Melt temperature is the point where the bulk of the polymer has melted (this represents a maximum on the DSC melt endotherm). End temperature is the point at which the entire polymer has melted (Troebler,1984).

A standard algebraic method was applied to select heat seal temperatures for testing. This is important since each film exhibited melt profiles and melt temperature values, characteristic of the specific film composition. To select temperature values that were of relative equivalence for each film, a ratio method was developed. The method observed the melt profile curve of each film, and selected temperatures that were equivalent, relative to the melt temperature. For example, "temperature one" is 80% of the melt temperature (for polyethylene this is 107 degrees C, and for the 29% vinyl acetate sample this is 75 degrees C). The method designed ensured a wide range of heat seal temperature values for all the films, yet the selected temperatures would be correlated by a specific relationship.

The maximum seal strength is observed on a 'conditions versus seal strength' graph.

The maximum seal strength is obtained above the melt temperature of the polymer sample, and is represented by the maximum value on the 'conditions versus seal strength' curve, or by a plateau of the curve (in a correctly run study, one or the other will occur). The experimental design was modified, where needed, to observe a maximum value or plateau.

7.02.03.05 Replicates

Statistically this study is justified using any number of replicates, picked to fit our desired level of significance (Burgess,1990). This was an original experimental design and ASTM procedures were followed where applicable. This study used five replicates per point.

7.02.03.06 Results

The method of measuring the tensile strength of the seals, as well as determining the type of seal failure followed the procedures outlined in ASTM F88 (Seal Strength) and ASTM D882 (Tensile Strength of Flexible Materials). For this study, both the break factor and the seal strength at break were recorded.

For the optimum seal strength (maximum), only the highest seal strength value was selected, regardless of dwell time, pressure, or the presence of a plateau. This method was considered the most statistically acceptable.

An alternate method was considered, but was rejected, since it was in conflict with the intent of the study. It was proposed that the plateau data be pooled for an average of the constant. The average seal strength determined would thus be less than the maximum value obtained at optimum conditions and would meet the objectives of the present study.

7.02.03.07 Variables

The dwell time was found to be a statistically significant parameter (a correlation coefficient of 0.05--95 percent correlation with the straight line), which was in contrast to the result obtained from the pilot test, and the literature references cited (Frito-Lay,1988;Theller,1989). To explain these results it is proposed that the dwell time, while statistically significant, may not be of practical significance with respect to commercial applications, and is of little significance when compared to the temperature effect. For the maximum seal strength, this study will use the highest seal strength, regardless of dwell time.

In comparison to the affects of temperature, while statistically significant, only minimal increases in seal strength were observed over a broad dwell time range. In the heat seal pilot study, a 600% increase in dwell time resulted in only a 13% increase in seal strength. These dwell time variations were considered commercially unable to be controlled to this accuracy, on standard industry equipment.

As expected, the temperature was the most significant variable effecting the strength of the heat seal. With the 19% and 29% vinyl acetate copolymers, however, the 'condition versus seal strength' plots did not show a plateau or peak within the temperature range of the test. However, evaluating these two films at higher temperatures (110% and 120% above the respective polymers' Tm) resulted in the typical plot.

These results may be due in part to the influence of the co-monomer (i.e. vinyl acetate) on the percent crystallinity and melt endotherm of the high vinyl acetate copolymer samples, where the polymer melt profile was distributed over a broad temperature range, and the melt initiation temperature was lower. These factors would be expected to influence both the seal initiation temperature and the seal range in a heat seal application.

All films exhibited enough significant information to determine maximum seal strengths.

It should be noted that the 19% vinyl acetate and the 29% vinyl acetate copolymer films were quite tacky and required an appreciable amount of handling to perform the heat seal strength studies. This ranged from a high stress when unrolling the film from the roll, to a significant amount of handling before and after sealing. Some stress was also exerted when mounting the film in the tensile testing jaws. This may have had an adverse affect on the maximum seal strength of these two films as well.

7.02.04 Hot Tack Pilot Test

The pilot study was run to evaluate the test method, and to suggest test modifications (discussed in the experiment section), as well as to determine the maximum hot seal strength (hot tack), the optimum heat seal combinations at this maximum, and the general trends around the maximum points. The type of bond failure was noted only in regard to finding the maximum seal strength. The optimum seal parameters are relevant only in regard to locating the maximum seal strength. The experimental design is discussed in detail in the experimental design section.

Hot tack is defined as the force to separate a polymer film seal 1/8 inch, or produce a visible hole, when the seal is stressed immediately after sealing.

The sealing parameters that effect the hot tack are: sealing bar temperature, dwell time of the sealing bars, pressure of the sealing bars, delay time between seal and testing, and the seal testing rate. Temperature, dwell time, and pressure are all variables in a cold heat seal test as well. The seal testing rate is a constant for all films in a cold seal test. In a hot tack test the seal testing rate is variable. The delay time is also variable for hot tack, since seals cool rapidly over the time between forming of the seal, and testing of the seal. Since a delay time of five to sixty minutes is acceptable, this is not a variable for a cold seal.

The James River Corporation Hot Tack Test Method (James River Corp.,1988) was followed in this experiment, with the exception that the dwell times and pressures were varied. A critical review of the procedure is discussed in the hot tack methods section.

The broad three point test (response surface style test) shows the seal profile, melt profile, and the maximum seal strength. Although a peak or plateau beyond Tm is critical for the maximum seal strength, the pilot test was designed to observe the effect of temperature on seals formed, over a temperature range from the onset temperature to the end temperature.

The studies began with developing response surface plots to evaluate trends in the combination of variables. The study used three variables (temperature, dwell time, and pressure) and three values for each variable, to generate a surface response graph.

7.02.04.01 Seal Testing Rate

This parameter could not be regulated on the test equipment employed for the study. However, since all films and test weights were subject to the same treatment, a relative comparison of hot tack strength is obtained.

7.02.04.02 Delay Time

This parameter was kept constant, and controlled by the operator. The time required to slide the seal out from the sealing bars, and activate the weight system, was variable and dependant upon both film and operator. Therefore, to control this parameter and conduct a uniform test, a two second delay time was employed throughout.

7.02.04.03 Dwell Time

It was concluded from the results of previous authors, and from preliminary tests, that the dwell time would be insignificant beyond a minimum point. For a complete statistical analysis, and correlation to the heat seal pilot study, the dwell time was tested. The results of the dwell time were in agreement with the literature.

7.02.04.04 Pressure

Previous authors concluded that the pressure would be insignificant beyond a minimum applied force (to create intimate contact of the soft film) with respect to hot seal strength (Theller,1989;ASTM 2.33.03,1989). The pilot test supported this conclusion, as no significant increase in seal strength was observed.

A standard statistical method was designed for this research to select hot seal temperatures, and was discussed previously in the experimental design section. These temperatures are recorded in the heat seal pilot test section.

The maximum seal strength is obtained from a 'conditions versus seal strength' plot, and is found to be either at the peak or at the plateau region of the plot. For all test films, the maximum cold seal strength was found to be at or above the polymer melt temperature.

7.02.04.05 Replicates

Since this is an original experimental design, there is justification for using any number of replicates picked to fit our desired level of statistical significance (Burgess,1990). Where applicable however, ASTM standard procedures were followed.

As with the industry procedures (James River Corporation Hot Tack Method for example), and due to the difficulty of each test, only one replicate is used at the test variables, and two replicates were run at the failure maximum stress. This second replicate helps verify the failure as valid.

The findings are presented in the results section and are based on the James River Corporation Hot Tack Method (James River Corp.,1988). The method requires a plot of successes and failures on a 'sealing bar temperature' versus 'seal strength (in tensile strength units of grams per seal width)' graph. The Hot Tack strength for a seal is defined as the maximum successful seal.

The hot tack pilot study was designed to evaluate the results of the James River Corporation test method (James River Corp.,1988) obtained on a randomly selected sample (4.5% VA), and to observe the affect, of dwell time, pressure, and temperature on heat seal strength.

7.02.04.06 Conclusion

From the results presented in the previous sections, it was concluded that, based on the James River Corporation Hot Tack Test Method, the dwell time and pressure had no effect on Hot Tack seal strength, and the temperature was found to be the controlling variable. As discussed in the following section, this procedure was found to be inadequate for several of the test films. Thus, the procedures were modified as described.

7.02.05 Hot Tack Experiment

A major consideration regarding hot tack testing is the lack of a standard method, and literature references that correlate all current data. There are several test methods described in the literature (Theller,1989; Frito-Lay,1988). However, they are primarily discussed for commercial quality control or qualitative measurement needs. These tests work well for individual companies and specialized groups of films, but have limitations when one tries to correlate, or standardize. That is, limitations arise when running this experiment, and in trying to develop a standardized procedure which would be applicable to a large range of heat seal polymers. A basic understanding of the limitations, validity of test, and application of this test, must be established for industry wide acceptance and applicability.

The commercial tests currently employed rank the films from "worst" to "best" regarding hot tack strength (the definition of worst to best are defined by the individual researcher). This is acceptable, since the only concern is whether a film has a stronger or weaker hot tack relative to a reference heat seal film.

To develop a test that would be more generally applicable to all polymeric films, a series of variations of tests and adaptations of tests were employed.

For the Rucker Hot Tack Attachment, the manufacturer recommends the use of the Hot Seal Strength test method by the James River Corporation (James River Corp.,1988). The test method is described in detail in the Materials and Methods section. This test method is a quick, simple, and inexpensive procedure for evaluating the relative hot tack strength of films. The hot tack apparatus is easy to install on a commercially available laboratory heat sealer.

7.02.05.01 Current Test Problems

This procedure provides a determination of the hot seal strength of heat sealable flexible web materials (James River Corp.,1988). The test procedure is quite easy to carry out, it uses very little film, and gets practical results quickly.

The procedure however, has a number of uncontrolled variables that should be noted; for example, the samples are cut by hand, and incur the associated errors. A standard precision sample cutter for the sample dimension was not available.

The use of only one replicate greatly reduces the statistical significance of the generated results. As is done on ASTM tensile testing or ASTM seal testing, a standard number of replicates should be used to ensure statistical significance. The modified test methods used two replicates, and three at the maximum for an additional replicate for statistical analysis.

The test procedure does not address the problem of only testing the seal, and not the surrounding heat or mechanically stressed film regions. A laminate or multi-layer film may not incur these problems, since the carrier web would not allow "seal system failure" due to the sealing process affects. The current test procedure also does not address the testing of completely fusible films such as PE and its co-polymers. For non-laminated/ non-multilayer film, or for PE based films, a constructed carrier web layer of adhesive tape could be applied (DTC,1988). This is discussed in detail later in this section.

Further, the test procedure does not address the potential pre-heating of the film samples. When the rod is used to move the samples under the hot sealing bars (see methods), the film may begin to pre-heat. Thus the dwell time set on the equipment may not be totally representative. The amount of pre-heating may also vary with the speed and precision of the operator. It should also be noted, that several of the films required a longer period of time to be placed in testing position. For these test samples, a large piece of film, that could more easily be fed into the sealing jaws, was employed.

The nature of the operator and the equipment itself, had further limitations. For example, the weight system is not very sensitive. The standard test weights range from 25g to 500g. For some thick films, this may be precise enough for hot tack determination. However, for the PE/ EVA films (1.5 mil) evaluated in this study, the 25g weights did not offer enough sensitivity. A 10 g weight was utilized to successfully test the film samples.

The weights used with this hot tack attachment are applied to the sample manually, that is, the operator drops a weight. This method of applying the weight introduces variables of concern, as well.

As stated above, the drop of the weight can result in a wide range of applied force, for the same weight. In one example, the operator can vary the release height. In another example, the operator can accidentally give the weighted bar an initial velocity. This variation can result in a change in the final velocity of the weight, and effect the final force applied to the film. The fact that an operator must slide the hot tack apparatus out from under the sealing bars, and release the weight can result in a "less than molten" test. Even when conducted at high operator speed and precision, it may be a full second from the release of the sealing bars and the testing of the seal. During this time, the polymer cools. The extent of cooling will be dependant upon the surrounding environmental conditions and on the polymer. To better control this parameter, a standardized delay time of two seconds between the opening of the sealing bar jaws, and the release of the weight bar was selected. This time was chosen since two seconds could be achieved for all samples.

Additional problems which occurred in developing the hot tack test included: "Seal" problems associated with the physical handling of the test film, or with the definitions and applications of this test.

A handling problem arose with these films, as it did in the heat seal test. Several of the films are extremely tacky, and thus, needed much manipulation in preparation and loading on the apparatus. Also, increased handling was required when employing Method "B" and "C" with the adhesive tape. This handling is a problem because of impurities deposited on the film by human hands and dirt.

It is beyond the scope of this study to statistically determine the precise effects on seals, to which these test limitations contribute. However, by nature, the hot tack test measures large differences in film properties. The variances and standard error that arise from the above mentioned concerns are considered so small however, that they will not effect the validity of the test results.

7.02.05.02 Intended Test Goal

The Hot Tack test was initially developed to examine laminates and coextrusions, and their ability to hold immediately after sealing. PE, being completely fusible, has Hot Tack values, but not in accordance with the basic objectives of the test as it was initially designed. For example, if the Tm is reached, there is no longer a film interface. The current test methods indicate that the Hot Tack test was not originally designed for completely fusible films.

A heat seal (hot or cold) test is concerned with three zones. The first zone (Zone 1) is the actual seal. The second zone (Zone 2) is the region between the seal and the unaffected material. This region can be adversely affected, and weakened, by the thermal and physical processes involved in heatsealing. Basically this region is weakened because of the stress involved in compressing the two films into a seal, and due to adverse affects of the film melting. The third zone (Zone 3) is the polymer film that is unaffected by sealing temperature or sealing mechanical processes. This heat seal model has been discussed in detail in the Literature Review (Theller,1988).

A multilayer laminate has a carrier web that reduces the stress on the middle zone, and therefore, only the heat seal layer, or the actual seal, will be stressed. As the interface (Zone 2) breaks, the seal is still stressed since the carrier web, is still supporting the seal layer.

For a simple and elastic structure such as polyethylene film, the film itself (zone 3) is stressed just as much as the seal, since the film also absorbs some of the force. A problem did arise with the 29% vinyl acetate copolymer structure, as the sample was elongated beyond the testing range for the hot tack tester, before the seal failed.

7.02.05.03 Solutions

To compensate for this tensile characteristic of the test films, the DTC test procedure of using an adhesive tape to provide a carrier web, and thus, eliminate the problem of film elongation, was employed (DTC,1988).

As noted in the Literature Review section regarding polyethylene, the polymer is completely fusible, and the peelable interface disappears as the polymer melt temperature is reached. The adhesive tape procedure (Method "C") did, however, eliminate some of the inherent problems associated with hot tack testing of polyethylene.

Regardless, the polyethylene did continue to fail by break at the hot tack maximum strength. The original intent of the hot tack test -- a measure of the peel failure of the seal -- is not met with polyethylene non-laminated, or non-coated films.

From the limitations observed in applying Method "A" to measure hot tack seal strength, a modified procedure was developed which is referred to as Method "B." This procedure was designed to minimize the problems inherent with the initial procedure (Method "A").

From further limitations observed in applying Method "B" to measure hot tack seal strength, an additional modification of the test procedure was made, which is referred to as Method "C." This procedure (Method "C") was designed to minimize the problems inherent with Method "B" as described above.

In Method "C," a lighter weight was added to the series of standard test weights. Instead of going down to 25g, the test method added a weight that lowered the sensitivity to 7.5 grams.

In summary, the major differences between the initial test procedure and the modified test methods are as follows: (i) the use of a standard delay time; (ii) the use of adhesive film as a carrier web for a co-polymer of ethylene-vinyl acetate; and (iii) a more sensitive range of weights.

The standardized delay time was simply a two second wait from the opening of the sealing bar jaws, and the release of the weight bar. This time was chosen since two seconds could s

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be achieved for all samples.

The adhesive tape functioned like the carrier web on a multi-layer film sample. For most films, the result was that the temperature at hot tack maximum increased, and the hot tack maximum strength decreased. The temperature increase was due to the increased resistance to heat transfer of the tape layers. The hot tack maximum strength decreased since the material itself was not contributing to the overall tensile strength and the seal system alone was being evaluated.

With Method "B", for the test films with 7 and 9 percent vinyl acetate respectively, the hot tack maximum strength dropped below the sensitivity of this equipment. Therefore, the effect on hot tack was undefined for these two copolymers in Method "B."

In Method "B", for the 29% vinyl acetate copolymer structures, the adhesive tape method produced seal failure which was not determined previously, because of the films inherent force/deflection characteristics.

The logistics of placing the adhesive tape on the film was quite crude, however, and thus errors were introduced. For example, air bubbles under the film aided seal failure, where the film did not have the support of the carrier web.

7.02.05.04 Justification of Adapted Test Methods

The concept of testing the seal system is both reasonable and practical, since failure of a commercial heat sealant film will occur in the seal system (Zones 1 and 2) or with the material itself (Zone 3). Tests designed to determine the tensile strength of a polymeric material (Zone 3) are well defined in the literature (ASTM D 882), as are standard procedures for measuring seal strength (Zone 1)(ASTM F88). However, there is no standard test for the strength of the interface (Zone 2). The initial test was modified to define a test for the interface (Zone 2). Notable considerations regarding the two modified procedures (both Method "B" and Method "C") are discussed below:

A point can be made that the hot tack is measuring the tensile strength between two materials while the seal is still molten. This assumes that the seal is made up of two discrete films that will separate. As was found with polyethylene during hot tack testing, the surrounding film (Zone 2) will fail.

An argument can also be made that the hot tack is measuring the polymer while molten. This test equipment, even when run as rapidly as possible, still exhibited a significant time delay that resulted in the seal area being cooled below the molten polymer state.

7.02.05.05 Comparison with Other Tests

Lack of method correlation between the cold seal test (ASTM F88) and Hot Tack Method "C" is noted. For quantitative comparison of the two test procedures and their results, the same methods and treatments must be applied. However, for relative comparison of test procedures, the exact methods are not significant with respect to the rank order of the specific property being evaluated.

7.02.05.06 Conclusion of the Hot Tack Experiment

This procedure of employing an adhesive tape backing was designed to reduce or eliminate the effect of stress on the film itself, and increase the range of materials that can be tested on the Rucker Hot Tack Attachment. This test can be used on polymer films having a wide range of tensile (percent elongation) properties.

7.02.06 Conclusions Of Seal Testing

It was concluded from the results of these studies that by utilizing an adhesive tape laminate, that the hot tack for non-laminate films, will be defined as the force to separate a seal system (zone 1 and 2, not specifically the seal alone) 1/8", or to produce a visible hole, when stressed immediately after the seal is made. This procedure is outlined in Method "C." For laminated materials that may experience delamination, the old definition of hot tack may not suffice, since this failure may not be a peel. However, Method "C" will then be applied.

For non-polyethylene films that will not completely fuse, Method "B" can be applied, since it is universally applicable.

Also, with Method "B," all films can be compared on the same relative scale.

If correlation to tensile strength data is necessary, then both Method "B" and Method "C" should be run, or run the "cold seal strength to break" with the same adhesive tape.

7.03 Statistical Correlations

A primary objective of this study was to establish relationships between the physical and molecular properties of a polymer, and the maximum seal strength of both hot seals and cold seals. The goal is to identify correlations that will allow prediction of the peel/seal strength of a film from knowledge of the physical and molecular properties of the polymer resin.

7.03.01 Data Groupings

Once the data were collected, it was sorted into four(4) groups. The groups were basically: (i) polymer characterization excluding DSC data; (ii) polymer characterization to include DSC data,; (iii) heat seal results; (iv) and hot tack results. These groupings, and their statistical comparison allowed examination of not only characteristics versus seals, but also for examination of the correlation between standard polymer tests. For example, an examination of the correlation between MFI and DSC data was observed and is discussed in Appendix C.

Group one contained the following property values: (i) percent composition; (ii) melt flow index; (iii) density gradient column values of density, and percent crystallinity; (iv) differential scanning calorimeter data of onset, melt temperature, and percent crystallinity; and (v) the gel permeation chromatography data of weight average molecular mass values (not the other averages here). This group was mainly compiled to observe relationships within the polymer characterization data.

Group two, consists of: (i) group one; (ii) GPC data of number average molecular weight, viscosity average molecular weight, Z-molecular weight average, and molecular weight distribution; and (iii) DSC derived data of glass transition temperature (Tg), Tm-Tg (a measure of the range of the melt profile), and Tm-Onset (a measure of the range of the melt profile). This was the grouping of total polymer characterization. Group three was the heat seal results, and used as described below.

Group four was the hot tack results, and used as described below.

The relationships examined were as follows. Relationship between parameters within group one -- observed relationships of polymer characterization traits with other characterization traits. Group two versus group three -- observed polymer characterization relationships with heat seal data. Group two versus group four -- observed polymer characterization relationships with hot tack data. Group three with group four -- observed the relationship of hot tack data to heat seal data.

7.03.02 Statistical Analysis Details

The computer software program Stat-graphics, produced data of: sample correlation, sample size, and p-value. The p-value is the probability that this sample correlation coefficient is a false correlation; this is a measure of how likely it is that a random correlation estimate could be generated about a set of numbers which the true correlation is zero. As the p-value approaches zero, the chance for a false correlation decreases. The table notes the correlation coefficient (how close the points are to the line), and the p-value (how sure we are that this is a true correlation).

For the correlation coefficient, standard scientific practice is to consider values above 85 as significant; for p-values standard scientific practice is to consider values below 0.05 as significant (Gilliland,1989).

The "multi-co-linearity" of the date (i.e. correlation between tensile strength and percent composition) was not examined in this research. The statistical analysis only viewed liner relationships between sample characteristics and maximum seal strengths. The "multi-co-linearity" of the heat seal maximum, to the hot tack maximum are recorded for reference, in Table 18.

Each seal maximum is an average of several test runs (sample size of n=6 to n=10). Though the data was observed for abnormal or large standard deviations, the standard deviation was not used in the correlation data.

7.03.03 Discussion of Effects

7.03.03.01 Viscosity Average Molecular Mass

The viscosity average molecular mass was found to be a statistically significant parameter with respect to heat seal and hot tack strength, and this predictor would be expected to have a direct correlation to the seal strength of other EVA copolymer films.

For hot tack, an inverse relationship was observed. This may be attributed to the fact that for hot tack measurements, the seal is tested while still in a semi-molten state and the contribution of physical entanglement of the high molecular mass chains to the cohesive bond strength of the seal is reduced markedly, as compared to the cold seal, where such interactions have been optimized. Here, interaction may involve the lower molecular weight polymer molecules, where the relative rates of diffusion of polymer chain segments across the interface will favor the more mobile low molecular mass species.

The viscosity average molecular mass (Mv) is a measure of long chain polymer molecules. Polymer-chain physical entanglement, which results from the diffusion of polymer chain segments, is a primary factor contributing to seal strength. The long chain polymer molecules would therefore be expected to exhibit a high degree of entanglement and thus the observed increase in seal strength with an increase in Mv.

7.03.03.04 Temperature

The thermal transition and relaxation temperatures determined in the present study were also found to be statistically significant with respect to seal strength and hot tack. The melt temperature (Tm) and the glass-transition temperature (Tg) appear to have the highest correlation of the transition and relaxation temperatures determined. The glass transition temperature was estimated from the single variable expression:

$$T_g = (2/3)\,T_m \qquad \text{(eq. 3)}$$

Thus, the melt temperature should provide a predictor of seal strength for a series of EVA copolymer heat sealant films.

7.03.03.05 Composition

As shown in Table 18, a change in composition (%EVA) resulted in a concomitant change in the physical and molecular properties of the respective copolymers, to include transition and relaxation temperatures, and molecular mass average values. The correlation of composition to seal strength is thus dependent upon other parameters related to basic molecular and physical properties of the polymer film.

8.0 SUMMARY AND CONCLUSIONS

Heat seal parameters and their correlation with the physical and molecular properties of a series of ethylene-vinyl acetate copolymers of varying percent vinyl acetate was investigated. Selected physical and molecular properties of the test copolymers were determined, to include: percent composition, onset temperature, glass transition temperature, melt temperature, percent crystallinity, density, number average molecular mass, weight average molecular mass, viscosity average molecular mass, Z-average molecular mass, dispersion index, and melt flow index. The heat seal parameters, including seal temperature, dwell time, and pressure were evaluated to determine the conditions affording maximum seal strength and hot tack for the respective film compositions. In the present study, the pressure was held constant and the dwell time and temperature were varied.

The evaluation of heat seal parameters was based on a statistically designed experiment, and carried out on a laboratory Heat Sealer with Hot Tack attachment. For each seal, the seal strength was evaluated by tensile stress or weighted balance technique, respectively. All tests were statistically designed, and analyzed for significance and validity. During the selection of test methods, it was necessary to compile and standardize the hot tack procedure. Many varying industry tests (and no ASTM method) could not be directly used.

To establish correlations between the physical and molecular properties of the test copolymers and maximum heat seal strength and hot tack values, linear regression analysis from the Stat-graphics software program was performed. The film composition, melt temperature, and viscosity average molecular mass (Mv) were found to be statistically significant parameters with respect to heat seal strength and hot tack.

Based on the correlations, polymer properties such as melt temperature and viscosity average molecular mass could provide a basis for predicting relative heat seal and hot tack strengths for heat seal film structures.

9.0 FURTHER RESEARCH

For further research, it is recommended that the optimization tests be officially standardized (Heat Seal Optimization, Hot Tack Optimization, and Standardized Correlation Testing), and applied to other polymers, polymer blends, and to laminates. It is also recommended that the correlation studies be applied to other polymers and polymer blends, to verify the "predictors."

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APPENDICES

APPENDIX A: Pressure Analysis - Heat Seals

Analysis of Applied Seal Pressure on the Resulting Heat Seals

INTRODUCTION

It has been theorized in literature that pressure has little, if any, affect on heat seals beyond a minimum level. This pilot test will quickly observe this phenomenon on the film that will be used for future heat seal testing.

METHODS AND MATERIALS

Heat Sealer: Theller Model EB Laboratory Heat Sealer

Tensile Tester: Instron 4202

Materials: PE (1.42 mil), and PE/EVA 29% VA (1.58 mil)

Methods

The temperature and dwell time is held constant at conditions within the seal regions. The pressure will be raised from 10 psi, to 100 psi, and then to 500 psi. The average, and standard of deviation will be recorded for a sample size of three. All tests as per ASTM F88 "Standard Test Methods for Seal Strength for Flexible Barrier Materials."

RESULTS

Table 1-1: Heat Seal Strength Statistical Data: 0% VA

Data 0%

	p= 10	p=100	p=500
avg. =	3.08	3.23	3.476
std. =	1.47	0.18	0.094
n =	3	3	3

Table 1-2: Heat Seal Strength Statistical Data: 29% VA

Data 29%

	p= 10	p=100	p=500
avg. =	2.332	2.47	2.83
std. =	0.33	0.36	0.42
n =	3	3	3

DISCUSSION

The goal of this test was to preview the effects of pressure on seal strength. The results will be verified in the full test. The data shows no significant difference between the three pressure variables, even though the averages appear to show an increase. We, thus, theorize that since the two films are at the same extremes of the test samples, that the middle films will act in a like manner.

A small increase in the averages, but with the standard of deviations, there is no significant gain in seal strength.

It is noted that the standard of deviations get better for 0% as the pressure increases, and the standard of deviations for the 29 % get worse as the pressure increases. This may be due to the 0% film having more intimate film contact as the pressure increases. The 29% film thinning out upon increase in pressure, may be the cause of the increase in standard of deviation.

Considering the film results independently, and only examining the averages: for 0% VA a 5000% increase in pressure resulted in an 11% increase in seal strength, for 29% VA a 5000% increase in pressure resulted in an 18% increase in seal strength.

APPENDIX B: Analysis of Density and Crystallinity Effects due to Variable Percent Composition of EVA

Analysis of Density and Crystallinity Effects due to Variable Percent Composition of EVA

OBJECTIVE

An interesting phenomenon occurred concerning the relationship between crystallinity, density, and composition, in the series of PE/EVA test films. The multiple effects are examined here.

LITERATURE REVIEW

> "An increasing density increases crystallinity [concerning Low Density Polyethylene] (Duncan,1988)."
>
> "Vinyl acetate controls crystallinity and flexibility. The major effect of vinyl acetate addition [to another polymer] is to reduce crystallinity (Duncan,1988)."

MATERIALS AND METHODS

This is a specific examination of the polymer characterization regarding the measured results of density, crystallinity, and percent composition.

The results of density and crystallinity experiments, by density gradient column and differential scanning calorimetry, were plotted on a graph versus percent composition, as given by the manufacturer.

RESULTS

Figure one records the density versus percent composition. Notice the upward trend.

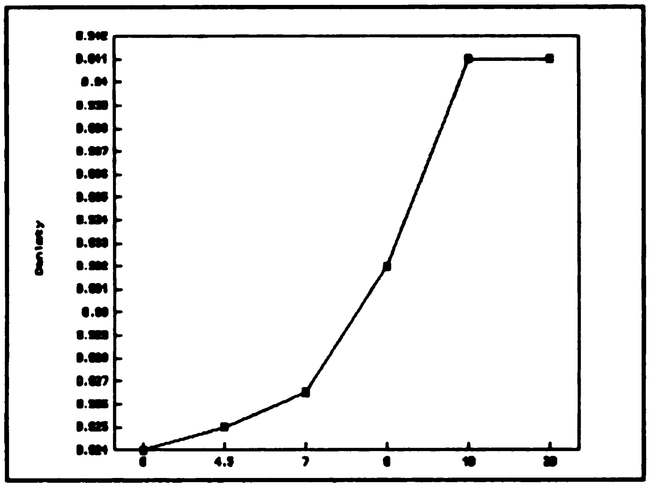

Figure 1: Density versus % EVA

Figure two records percent crystallinity versus percent EVA. Notice the downward trend

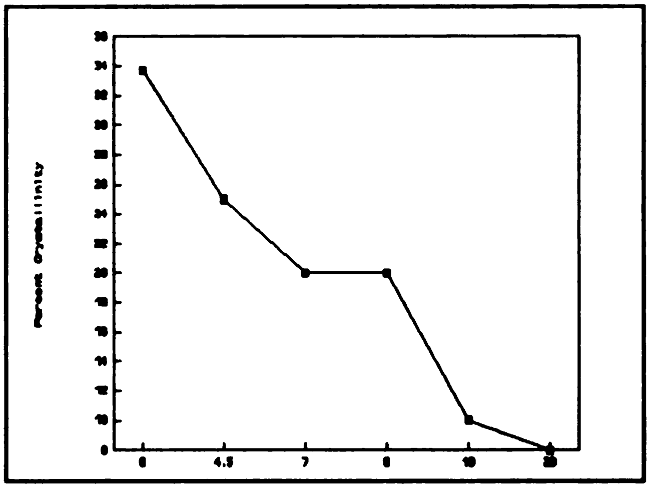

Figure 2: % Crystallinity versus %EVA

DISCUSSION

As the vinyl acetate composition increases, the polyethylene gets more random. The vinyl acetate is very dense (very little free volume) and thus the density rises with the increase in vinyl acetate. Due to the vinyl acetate, less than 50% VA equals a 100% amorphous polymer, the percent crystallinity decreases as the density rises.

This is contrary to the belief that as a polymer gets more random and more disordered, the crystallinity decreases, and the density decreases. Specifically, in this series of co-polymers, the VA controls the density and the crystallinity in this series of copolymers.

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APPENDIX C: Comparison of Melt Flow Index and Gel Permeation Chromatography estimates of Molecular Weight of Polymers

Comparison of Melt Flow Index and Gel Permeation Chromatography estimates of Molecular Weight of Polymers

INTRODUCTION

A statistically significant test method correlating data from the Melt Flow Index (MFI), and precise data results of Molecular Weight Average, is highly desirable and valuable to those using the MFI for indirect measurements of molecular weight average.

The objective was to measure a series of commercially average PE/EVA co-polymers by Melt Flow Index and Gel Permeation Chromatography (GPC), for comparison of indirect molecular weight average versus actual molecular weight average. The GPC is a very accurate measurement tool for molecular weight of polymers. Also, the relationships between actual data was noted.

LITERATURE REVIEW

The Melt Flow Index has been used as quality control and quantitative measuring device in industry, mainly because of its convenience and its availability (Sweeting,1963).

Very simply, the Melt Flow Index pre-heats a polymer -

resin, film or powder - to a standard temperature, then a standard weight forces the flowing polymer through a standard die. The result is noted in grams per 10 minutes as a function of the standard test conditions. This flow rate is referred to as the melt index.

The lack of comparison of melt index (MI) and melt viscosity for various reasons (rates of shear for different polyethylenes may be quite different, and deformation under a shear stress is dependant on time) is well noted in the literature (Sweeting, 1963).

The melt index results have been correlated to molecular weight average (Mw) (Ganapothy,1981). It is well supported that as MI decreases, the Mw increases (Ganapothy,1981). The relationship is in inverse proportion (MSU,1988).

$MFI=1/M$ viscosity

Also, a direct numerical relationship has been cited between the Mw and the melt viscosity (Sweeting,1963), as shown in Sweeting's equations below.

$Mv=Mw^{3.4}$

$no=3.01\times10^{-12}*Mw^{3.4}*e^{-2.35Nc}$

no= isothermal viscosity of melt under Newtonian conditions of flow

Mw=weight average molecular weight,

Nc= number of methyl groups (=short chain branches)

Thus, the Mv and Mw can be directly correlated to the MFI. This is also shown in an example from MSU:

ie: mfi=.2...mv= 300,000 p

mfi=20...mv= 3,000 p

This is an empirical comparison.

Even though these correlations are commonly accepted, the manufacturer only claims that the test is a measure of flow rate (Ray-Ran,1980). This follows ASTM's definition of the test as a quality control method (Recker,1988). The literature expresses the melt flow index as a test method to distinguish between grades of polymer film, or instance incoming polymer resin (MSU,1988). The numbers calculated from the Melt Flow Index will be compared and correlated versus the numbers calculated from Gel Permeation Chromatography (GPC). The GPC accurately determines the weight average molecular weight (Mw), the number average molecular weight (Mn), the viscosity average molecular weight (Mv), and the high molecular weight average molecular weight (Mz). From these, the molecular weight distribution is calculated.

This research will focus on the comparisons of weight average molecular weight to conclude the validity for some of the assumptions made regarding the Melt Flow Index.

STATISTICAL ANALYSIS

Only linear examinations can be used for this data. Not enough films were examined to produce significant polynomial examinations.

Linearity - MFI vs. Mw: The basic data of the melt flow index should correlate to Mw if the above mentioned assumptions are true.

Quality Control Differences - MFI vs. series of PE/EVA films: The measured MFI rate was viewed versus: percent composition, density, melt profile (onset, and melt temperature), and the tensile strength of the films. These were analyzed by significance levels.

RESULTS AND DISCUSSION

The data visually illustrate the correlation or lack of correlations of the MFI derived numbers and the actual derived numbers. Conditions were chosen as in ASTM, and varying temperature flow rates were adjusted for a common flow rate.

Thus, the melt flow index estimates the weight average molecular weight, but does not offer insight into quality control questions regarding changing polymer characteristics.

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APPENDIX D: JAMES RIVER CORPORATION HOT TACK TEST METHOD

JAMES RIVER HOT SEAL STRENGTH TEST METHOD
using the Rucker Hot Tack Attachment

A. SCOPE

This procedure provides a determination of the hot seal strength of heatsealable flexible web materials.

B. APPARATUS

1. Theller Precision Heatsealer, or other hot bar laboratory heatsealer.
2. Rucker hot-tack apparatus, with 50, 100 and 200 gram weights.
3. Template: 2" x 4".
4. Razor blades.
5. Charts of weight in grams versus temperature in °F. (See Figure 1 attached.)

C. PROCEDURE

1. Bolt the Rucker apparatus to the heatsealer frame.
2. Set heatsealer bar and anvil temperatures to a value 20°-30°F below the anticipated optimum.
3. Use the template and blade to cut 20-30 MD 2" x 4" samples.
4. Set desired dwell time and pressure.
5. Position one of the 2" x 4" samples in the jaws of the Rucker unit, making certain it is perpendicular to the sealer jaws and that the sealing surface is inside.
6. Place a 50 gram weight on the peg nearest the sealer jaws, then lift the lever arm on the Rucker unit so that the sample forms a closed loop pointing toward the sealer jaws.
7. Hold the lever in that position and push the sliding unit forward. Use a rod to thread the sample between the open sealer jaws.
8. Actuate the heatsealer and simultaneously release the lever arm.
9. At the exact moment the jaws open, pull the sliding unit out of the jaws to allow the 50 gram weight to move the lever.
10. If the sample is so poorly sealed that the weight pulls it open more than 1/8", raise the temperature 10°F, let it stabilize, and repeat steps 6-9 with a fresh sample.
11. After each seal attempt, record the result on a work sheet prepared as in Figure 1. Use "X" if the sample passes and "F" if the sample fails.

12. Continue to increase the temperature in 10° increments until the sample finally adheres with the 50 gram weight on the #1 peg. Then, maintain that same temperature, but move the 50 gram weight to the center peg and repeat the test with a fresh sample.
13. As can be seen on Figure 1, as the sealing temperature is increased, the hot seal strength increases to a maximum and then drops to a minimum. The force applied to the sample is adjusted by positioning one or more of the weights to the various pegs on the lever arm. Use Table A as a guide in weights to use and pegs on which they should be placed.
14. When the hot seal strength has dropped back to a minimum, or if the sample begins to deteriorate, the test is complete.

D. <u>REPORTING</u>

A graph should be drawn from the work sheet as illustrated as a "X-Y" graph of the output.

The graph must show the sample identity, jaw pressure, jaw type, dwell time, and whether the sample was preheated to the jaw temperature prior to application of the specified pressure.

TABLE I
WEIGHT COMBINATIONS

Desired Separation Force (gms.)	Pin Near Sealer (Factor 0.5)	Center Pin (Factor 1.0)	Pin Away From Sealer (Factor 1.5)
25	50	--	--
50	--	50	--
75	--	--	50
100	--	100	--
125	50	100	--
150	--	--	100
175	--	100	50
200	--	200	--
225	50	200	--
250	100	200	--
275	--	200	50
300	--	--	200
325	50	--	200
350	--	200	100
375	50	200	100
400	--	100	200
425	50	100	200

Source: Packaging Research & Development Laboratory, Crown Zellerbach Corp., San Leandro, California

FIGURE 1
WORKSHEET

```
300                   Y Axis: Force (grams)
275                   X Axis: Temperature in degrees F
250                   Material: HDPE/EVA
225
200
175
150                       F F
125                     F X F
100                   F X X X F
75                  F X       X F
50                  X X         X X F
25                F                 X X X X X F
0
-------------------------------------------------------
  170  180  190  200 210 220 230 240 250 260 270
```

APPENDIX E: DESIGN AND TEST CONSULT (DTC) AB HOT TACK TEST METHOD

D T C Hot Tack Tester

model 52-C

OPERATION INSTRUCTIONS

D T C DESIGN & TEST CONSULT AB
Tranebergsv. 41
S-161 32 Stockholm-Bromma / Sweden
#9-011-
telephone: + 46 - 8 - 718 - 3345
telex: 13264 Testing S.
Mr. Habhold

Operation instructions

Contents

<u>Figures</u>

Operation Instructions and Technical Data

1.1. Introduction

The term "Hot Tack" is used as an expression for the strength of heat seals immediately after the sealing operation, in contrast to the term "seal strength" which applies to the strength of the seal after is has cooled.

Information on the hot tack properties of materials is of special interest to the manufacturers of raw materials and laminates for the packaging industry. This includes manufacturers of basic polymers, plastic laminates, plastic coated paper and carton. The hot tack properties are of particular interest in form fill operations where production disturbance can occur if the hot seals are still weak during the actual production operation. In addition, the influence of contamination from the product to be packed (powder or liquid) on hot tack is of interest.

1.2. Installation instructions

1. Lift machine out of crating by supporting the base plate. Do not attempt to lift machine by other part.

2. Place machine on a plane bench top. Suitable bench top height above the floor is approx 29 - 30".

3. Connect to electrical power 220 V, 50 Hz, or a step-up transformer, if power is 110 V, 60 Hz. Protective ground is mandatory.

4. Connect to compressed air supply with the hose of 10 mm inside ø (approx 3/8"). The pressure of the air supply shall be min. 6 bar, max. 10 bar.

5. Continue with procedures as per instruction manual. The apparatus is calibrated with the incoming pressure regulator inside the apparatus at 5 bar.

6. Open rear door of the apparatus housing and check following:

   a .
   after connection to your air supply, the pressure gauge on the incoming air regulator shall indicate 5 bar, which is the secondary pressure.

If necessary, adjustment can be accomplished by turning the knob of the regulator.

b .
oil level in the lubricator adjacent to the pressure regulator. If necessary, refill with clean, light-grade oil.

<u>2.1. General test principle</u> (Ref layout diagram fig. 1)

The DTC Hot Tack Tester enables one to simulate production sealing conditions and to control the following variables:

1. Sealing temperature
2. Sealing time
3. Sealing pressure
4. Delay time to load on seal
5. Seal peeling rate

The normal test procedure is to fix the variables 2 to 5 and to measure the hot tack at various sealing bar temperatures. Results are usually presented graphically and have general shape shown in fig. 2.

For general testing purposes, the following conditions have been found suitable but should be adjusted to meet specific demands.

Table 1. General Test Conditions	
Sealing time	0.5 seconds
Sealing pressure	0.5 N/mm^2 on the seal itself (this corresponds to 100 kPa on the test unit pressure gauge for test strips of width 15 mm)
Delay time	
Peel rate	0.1 seconds (refer 3.5)
Temperature	200 mm/second
	5 - 10°C intervals over the heat sealing rate

It is advisable to also measure the cold strength (which is usually several magnitudes higher than the hot tack) over the same temperature range before making definite recommendations for production machine settings as it is necessary to optimize both the hot tack and the cold seal strength.

3.1. Setting Instructions

Start Up and Pressure Adjustment

Connect to 220/240 V mains supply and switch on.

Turn on compressed air supply and check the pressure on the gauge of the incoming pressure regulator for 500 kpa (5 bar).

(The reason for this is that the apparatus has been calibrated with an incoming pressure of 500 kPa).

With the manual seal button pressed down and the sealing bars closed, adjust the sealing bar pressure (6) with the regulator (7) to the required pressure as follows.

The normal test strip width is 15 mm and the seal breadth is 5 mm giving a seal area of 75 mm^2. The unit is so adjusted that for general test pressure of 0.5 N/mm^2 on the seal itself, the pressure gauge is set on 100 kPa. Note: 100 kPa = 1 Bar, corresponding to 0.5 N/mm^2 on 15 mm wide specimen.

Temperature

The required sealing bar temperature is set on the temperature controllers with the digital set point buttons. As the thermo couple measuring the sealing bar temperature is situated some mm below the bar surface, there will be a slight difference between the two points, i.e., the surface temperature will always be slightly lower than the temperature in the control point. A further temperature difference will result if the surface is covered with PTFE-cloth. Thus, the set point value can be corrected for, if desired. The graph fig. gives the correction values for a set-up with a certain PTFE-cloth cover 0.07 mm thickness.

It is normal practice to start measurements at a low temperature, e.g., 90 - 90°C, and after each set of measurements (3 to 5 per setting) increase the temperature in steps of 5 - 10°C, up to 200°C, perhaps 300° if required.

Peel Speed Adjustment

The required peel speed is adjusted by turning the dial (8) to the required position after reference to the peel speed calibration curve/Figure 3, (the gene al test speed of 200 mm/sec is obtained by setting the dial at approx 725).

The speed control valve is turned counterclockwise for increased speed. The values for speed setting, read on the knob, start at 999 (corresponding to zero speed) and decrease down to 000 for maximum speed.

Seal time adjustment

The seal timer (5) is set to the required figure (0.5 seconds for general testing).

Delay Time Adjustment

The required delay time is set by coordinating the settings of timers 4 and 5, Fig. 1. This allows compensation for the slack in the sample to be made. The geometry of the apparatus is such that a constant slack of 30 mm is obtained in the bottom section of the test strip whereas the top section has virtually no slack.

Example 1

Conditions of test

Sealing time 0.5 sec.

Peeling speed 150mm/sec.

Delay time required: Zero

At 150 mm per second a time of 0.2 seconds is required in order to take up the slack before the seal is loaded, and therefore the sealing time is set at 0.5 sec and the delay time compensation unit is set at 0.3 sec. This will allow the tension mechanism to start before the sealing bars open and enable peeling of the seal to start simultaneously with the opening of the sealing bars.

Example 2

Conditions

Sealing time 0.5 sec

Peeling speed 150 mm/sec

Delay time required: 0.2 sec.

In this case both timers are set at 0.5 sec. as 0.2 sec delay will have elapsed before the peeling of the seal begins.

Example 3

Sealing time 0.3 seconds

Speed of peeling 300 mm/sec

Delay time required 0.2 sec

Time to take up slack = $\frac{30}{300}$ = 0.1 sec.

Therefore set sealing time at 0.3 sec and delay time compensation unit at 0.4 seconds

Total delay time = (0.4 - 0.3) + 0.1 = 0.2 sec

4. Test Procedure (Ref. Illustrations in Fig 4.1. to 4.5)

After setting the instrument as described above, measurement of hot tack are made as follows for flexible materials (see point 4.6 for rigid materials).

4.1. Test samples

These sample of width 25.4 mm and length approx 28 cm are cut in a sharp accurately set strip cutter and placed for instance in a clean upright container. 28 cm is convenient length for material sent in A 4 envelope.

Note: Under no circumstances must fingers touch this area to be sealed as slight amounts of human fat, sweat and other contaminants can seriously affect results.

4.2. Mounting

A test strip is mounted between the upper and lower eccentric clamps with the sample insertion left inside the apparatus refer to fig. 4.1. It is important that all of the samples be reasonably tightly stretched and mounted in same manner in order to get constant results. An important operation before inserting the specimen into the lower grips is the checking of zero load indication on the force indicator.

4.3. Folding and Insertion

The reset button is pushed to raise the peel cylinder with lower clamp. The specimen will now form a bow. If not, move center part of specimen to the right, so it will be placed on the right side of the folding pins (F).	FIG 4.2
Pull out specimen clamp (C) from the apparatus with handle (H), until stop. The clamp will be open now.	FIG 4.3
Push button "folder out" and the two folding pins will move out. Move folding blade (B) to the left until specimen is folded however not stretched, especially the upper part.	FIG 4.4
Move folding blade (B) to the right until micro switch is actuated and release handle. The folding pins will now remove automatically.	FIG 4.5
Move clamp handle (H) to the left into the apparatus until stop, and release handle immediately. During this movement the clamp has gripped the folded specimen and pulled it between the sealing bars. In the left end position a switch will be actuated which starts the testing procedure. The upper sealing bar will now come down and seal the folded specimen. In it's lower position, the sealing bar opens the clamp and releases the specimen, thus avoiding friction in the following peeling sequence. After present sealing time the bars open and the peeling actuator will pull the specimen downwards.	FIG 4.6
After sealing bar has been activated, clamp handle (H) must be moved slight back to the right - otherwise seal gap will be activated again.	

4.4. Measurement

After the completion of test cycle, the hot tack value is read on force indicator and noted. At least three measurements at each setting are required in order to get sufficient data to be able to construct a reliable hot tack curve.

The data so obtained are usually illustrated on curves which usually have the general shape shown on Fig. 2.

It is normal practice, if several materials are to be examined, to cut test strips of all samples and to measure the hot tack on all samples before changing the temperature for the next set of measurements.

4.5. Uncoated materials

It is not possible to get true hot tack values on uncoated materials such as unlaminated polyethylene film. Instead of "peeling", the seal as is intended for hot tack, one often gets a "break" in the seal. In order to achieve a peeling effect, it is necessary to first cover the film with non-thermoplastic tape, such as ordinary adhesive tape which is often based on polyester.

The procedure is to cover the plastic film with tape first and then carefully cut the test pieces in the cutting apparatus into 15 mm strips. The strips are then mounted in the normal way with the polyester nearest the sealing bars. One may always use the same tape quality in order to get reproducibility.

4.6. Rigid and semi-rigid materials (such as stiff paper, carton and thick plastic laminates)

Samples of rigid and semi-rigid materials cannot be mounted and tested as in point 4.1. - 4.4 as the non-flexibility of the materials results in a load on the force measuring cell before the sealing operation. In order to avoid this effect, the rigid material samples are connected to flexible thread loops which are in turn fixed to the sample hold r clamps. Alternatively, garters can be supplied with the apparatus to be used as shown on Fig.. The samples of rigid material, also of width 25.4 mm, are cut into lengths of 60 mm. Two pieces at a time are then taken with the surfaces to be sealed against one another, and two small slits are made, as shown in the diagram, with a pair of short, very sharp scissors. Ref. Fig.

The samples are then hooked onto the flexible threads and placed in the specimen clamp. This is accomplished by moving the clamp slightly to the left while simultaneously holding the strip between the jaws. As soon as the strips are held by the clamp, the test procedure can be continued as in normal operation. Of course, the folding device is not used. Care must be taken in adjusting the loops of flexible threads so as to obtain the same degree of slackness as with wholly flexible samples. This is to ensure that one obtains an accurate delay time as explained in point 3.5.

5. Electronic control and measuring system

The Hot Tack value is measured by an electrical force transducer to which the upper specimen grip is attached. A signal conditioner, placed under the electrical control panel, supplies the transducer with ± 5 V stabilized DC, and amplifies the signal coming from the transducer. A peak hold circuit catches the peak signal during the peeling of the specimen and displays it on a digital panel meter (DPM) directly in N. The force transducer is a sensitive device and must not be subjected to forces higher than 100 N in vertical direction and not to any side load.

A lock-out circuitry, controlled by two solid state switches, ensures that force is only measured during the peeling phase. The switches are placed along the peeling actuator and triggered by a rod parallel to the actuator. The first switch enables the measuring circuit when the piston has accelerated and the second switch disables further measurement when the piston has reached a position close to the bottom.

Below the DPM there are controls for the measuring mode, i.e., "follow", "peak", and "reset", as well as for the zeroing of the measuring system. Under the label adjacent to the DPM there are further controls for electronic adjustments and for options. See fig. 9. Zero setting of the force measuring system has to be done in "follow"-mode or with actuator in top position in "peak"-mode.

The force measuring system is also equipped with a shunt calibration device. By pressing button a resistance is connected over one bridge arm giving a known change in signal, i.e., a simulated load reading. The correct reading is individual for each transducer and noted on each apparatus. Thus, the condition of the force measuring system can be checked on request and eventual recalibration can be made should the shuntcal reading be out of tolerance (say ± 1%).

D T C
DESIGN & TEST CONSULT AB
Calibration sheet. Hot Tack Tester 52-C
Customer: Du Pont USA force calibration/ Shunt
Calibration value: 39,9
August 1986.

APPENDIX F: FRITO-LAY HOT TACK TEST METHOD

FRITO-LAY TEST METHOD

DESCRIPTION: HOT TACK TEST PROCEDURE
TM NUMBER:FL 61
DATE ISSUED:03/04/86
SUPERSEDES:08/24/84

REVISION: Revisions in D.2, 7, 10, and 11

A. SCOPE

This test method measures the hot tack seal integrity of polymer film heat seals in terms of sealer dwell time, temperature, pressure, and applied shearing force.

NOTE: Test Method FL 61.1 - Procedure For Determining Hot Tack Of Spot Applied Adhesive Blocks On Outer Film Surface - should be utilized for specific and unique cases as referenced in the individual structure specifications.

B. DEFINITIONS

1. Hot Tack: Is the ability of the heat seal to resist separation when stressed immediately after the seal is made.

2. Hot Tack Failure: Is when the fresh seal separates by more than 3/16" when stressed as measured from the junction of the seal to the outer mark of the top seal bar on the top film stripe.

C. APPARATUS

1. Sentinel Sealer: Packaging Industries, Inc., Hyannis, Massachusetts (see Method 60 for operational details).

2. Hot Tack Tester: (See Appendix A) is to be mounted on the front of the Sentinel Sealer by means of two bolts. (May be purchased from

Versa-Tool Manufacturing, 222 River Hill Road, Irving, Texas 75061, (214) 554-6851.)

3.
JDC Precision Specimen Cutter: Thwing-Albert Instrument Company, Philadelphia, Pennsylvania.

4.
Type K Calibrated Pyrometer: See Method 60 for calibration procedure.

D. PROCEDURE

1.
Set the Sentinel Sealer to the desired temperature, dwell time, and pressure (see Appendix B for guidelines). Temperature is set on an idle sealer, do not cycle the jaws before setting the temperature. Temperature is measured on the side of the teflon coated plate attached to the upper seal jaw, directly above where the specimen will be tested. Dwell time is set to 0.5 seconds and verified by use of a microswitch activated timer (see Method 60). Pressure is set to 30 psi gauge setting (see Method 60).

2.
Take test specimens from the sample film by cutting 1" x approximately 10" strips using the JDC Precision Specimen Cutter (10" length parallel to machine direction of specimen). Prepare about 30 specimens then mix them up to randomize the set of specimens. Take special care not to contaminate the sealant surface. Do not touch the area to be sealed.

3.
Use an appropriate force variable (weight) based on product fill weights and bag seal widths if nothing is known about the sample. Place the weight on the stirrup.

4.
Fold the test specimen end to end with the sealant side facing itself. Hook the stirrup on the loop end.

5.
Web the specimen into the hot tack tester so that it hooks the stirrup and goes over the back roller. Continue by webbing the loose ends, one

above the top front roller and one below the bottom front roller.

6.
Activate the sealer three times without the specimen in the jaws.

7.
Quickly extend the unsealed loose ends between the sealer jaws and out the back area of the seal jaws. However, do not bunch the specimen against the back of the sealer, and make sure the top and bottom ends of the specimen line up with each other.

8.
Close the sealer jaws and simultaneously release the stirrup.

9.
When the sealer jaws release the sealed strips, the weight will instantaneously pull the sealed layers across the shear rollers. Wait until all motion has ceased. Cut the specimen free and measure the width of delaminated seal area.

10.
Measure from the imprint of the upper seal bar on the top end to the junction between the top and bottom ends (where the seal still is intact). Record the weight and extend of delamination.

11.
Repeat steps 3 through 10 until a weight is found which causes failure (higher than 3/16" separation). Lower the weight until a weight is found where ten grams higher than the chosen weight causes failure. Repeat the test at this weight for a total of five times. If one failure is obtained at any point in the repeat tests, reduce the weight ten grams and attempt to gain a total of five passing tests at this weight. Note the example test sequence shown below.

Test #	Weight (g)	Results
1	400	Fail
2	200	Pass
3	300	Fail
4	250	Pass (1)
5	280	Fail
6	275	Fail
7	260	Pass
8	270	Pass
9	270	Pass
10	270	Fail
11	260	Pass
12	260	Fail
13	250	Pass (2)
14	250	Pass (3)
15	250	Pass (4)
16	250	Pass (5)

At 250°F. Hot Tack

E. REPORT

Report the highest weight (in grams) which passes five tests with <u>no</u> failures at the specified seal temperature using 0.5 seconds dwell time at 30 psi.

INSTRUCTIONS ON SETTING TESTER DEVICE TO HOT TACK SEALER

In order for device to seat properly, the front face of the tester casting must be milled flat.

After milling casting, compress sealing jaws to the closed position.

Measure a distance 2-1/4" down from the parting line of the sealing jaws.

Etch a horizontal line on the casting at this dimension.

Find the vertical center line of the jaws and etch a vertical line 2-1/2" to each side of center line and intersecting at the horizontal line previously drawn.

Center punch and drill and tap (2) 1/4-20 holes. These holes will mount locator plate provided with (2) screws and (2) washers.

Mount tester device to locator plate with screws provided.

Adjust vertical position such that the center line of front rollers will be in-line with parting line of sealer jaws when in the closed position.

The distance from the front face of the sealer jaws (in the closed position) to the center line of the front rollers should measure 11/32 (.343).

Note: Due to the variance in base castings, shimming or removal of material from locator plate will be necessary to attain the 11/32 dimension.

APPENDIX G: COMPLETE TENSILE STRENGTH DATA

Table G: Complete Tensile Data by Material

MATERIAL: 0% VA

TEST:	LOAD (lbs.)	EXT. (in)
1	5.09	9.232
2	4.768	9.515
3	5.181	9.608
4	5.283	10.03
5	5.084	9.414
6	6.992	9.322
7	3.146	10.16
8	4.988	9.366
9	4.521	9.538
10		
AVG=	5.006	9.576
Std. Dev=	0.932	0.299
n=	9	9
%ELONG	.479	

MATERIAL: 4.5% VA

TEST:	LOAD (lbs.)	EXT. (in)
1	5.616	5.051
2	5.922	6.411
3	6.26	6.045
4	5.305	4.688
5	6.824	7.292
6	6.4	5.868
7	5.348	4.8
8	5.262	8.621
9	6.803	8.341
10	6.668	7.957
AVG=	6.04	6.51
STD.DEV=	0.6	1.4
n=	10	10
%ELONG.		325.37

Table G (cont'd)

MATERIAL: 7.0% VA

TEST:	LOAD (lbs.)	EXT. (in)
1	5.197	6.597
2	4.612	6.245
3	6.604	6.461
4	5.734	7.771
5	6.131	8.664
6	6.003	8.664
7	5.938	8.251
8	6.808	9.032
9	6.223	8.091
10	8.274	9.034
AVG=	6.152	7.89
STD.DEV=	0.931	1.02
n=	10	10
%ELONG.		394.05

MATERIAL: 9.0% VA

TEST:	LOAD (lbs.)	EXT. (in)
1	8.279	11.94
2	7.748	12.62
3	5.809	9.998
4	10	11.37
5	7.05	10.31
6	5.09	8.114
7	6.69	11.41
8	6.158	11.27
9	5.638	10.78
10	6.443	12.78
AVG=	6.9	11.1
STD.DEV=	1.38	1.3
n=	10	10
%ELONG.		552.96

Table G (cont'd)

MATERIAL: 19% VA

TEST:	LOAD (lbs.)	EXT. (in)
1	8.204	9.882
2	6.991	8.015
3	7.382	7.941
4	8.29	5.967
5	9.584	8.771
6	8.451	8.537
7	8.344	9.316
8	9.074	9.378
9	7.259	7.467
10	10.48	7.327
AVG=	8.41	8.26
STD.DEV=	1.028	1011
n=	10	10
%ELONG.		413.005

MATERIAL: 29% VA

TEST:	LOAD (lbs.)	EXT. (in)
1	11.91	9.997
2	6.652	12.47
3	6.523	9.799
4	10.08	11.46
5	12.08	12.13
6	4.96	8.014
7	11.68	12.84
8	12.28	12.79
9	10.84	12.33
10		
AVG=	9.67	11.3
STD.DEV=	2.68	1.58
n=		9
%ELONG.		566

APPENDIX H: COMPLETE DATA OF MOLECULAR MEASUREMENTS

Table H: Summary of Molecular Measurements (a)

A. Mn (b)

FILM (% VA)	REP 1	REP 2	Average
0	15444.8	13919.2	14682
4.5	25255.2	25945.6	25600.4
7	30202.4	30367.6	30285
9	7177.56	6690.038	6933.799
19	14426.2	12636.6	13531.4
29	10920.4	10864.8	10892.6

B. Mw (c)

FILM (%VA)	REP 1	REP 2	Average
0	157733	152634	155183.5
4.5	173287	171974	172630.5
7	179940	179360	179650
9	173361	169159	171260
19	166933	165264	166098.5
29	118993	122590	120791.5

C. Mv (d)

FILM (% VA)	REP 1	REP 2	Average
0	157680	152597	155138.5
4.5	173223	171918	172570.5
7	179340	179896	179618
9	173325	169159	171242
19	165231	166882	166056.5
29	122562	118966	120764

Table H (cont'd)

D. Mz (e)

FILM (% VA)	REP 1	REP 2	Average
0	312334	290174	301254
4.5	468665	470939	469802
7	485524	483057	484290.5
9	524017	516057	520037
19	430390	442420	436405
29	250922	226673	238797.5

E. MWD (f)

FILM (% VA)	REP 1	REP 2	Average
0	10.2126	10.9657	10.58915
4.5	6.80944	6.66788	6.73866
7	5.90682	5.9576	5.93221
9	24.1531	25.2839	24.7185
19	11.5715	13.0782	12.32485
29	11.2257	10.9521	11.0889

(a) All measurements are in Atomic Mass Units
(b) Mn= Molecular Number Average
(c) Mw= Molecular Weight Average
(d) Mv= Molecular Viscosity average
(e) Mz= High Molecular Average
(f) MWD= Molecular Weight Distribution

APPENDIX I: COMPLETE DATA OF THICKNESS MEASUREMENTS

Table I: Thickness Measurements

THICKNESS TESTING:
EVERY THIRD WILL BE TESTED ON STANDARDIZED MICROMETER

Film: A - 0% VA

T	DT	P	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Seal Std.Dev	AVG.
1	1	c	1.5			1.5		0	1.5
1	2	c	1.35			1.5		0.075	1.425
2	1	c	1.45			1.35		0.05	1.4
2	2	c	1.45			1.4		0.025	1.425
3	1	c	1.35			1.45		0.05	1.4
3	2	c	1.3			1.4		0.05	1.35
4	1	c	1.35			1.45		0.05	1.4
4	2	c	1.4			1.5		0.05	1.45

THICKNESS AVG = 1.42
Std.Dev = 0.04

Film: B - 4.5% VA

T	DT	P	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Std.Dev	Seal AVG.
1	1	c	1.2			1.5		0.15	1.35
1	2	c	1.5			1.4		0.05	1.45
2	1	c	1.7			1.4		0.15	1.55
2	2	c	1.3			1.4		0.05	1.35
3	1	c	1.5			1.3		0.1	1.4
3	2	c	1.3			1.4		0.05	1.35
4	1	c	1.4			1.3		0.05	1.35
4	2	c	1.5			1.4		0.05	1.45

THICKNESS AVG = 1.41
Std.Dev = 0.04

Table I (cont'd)

Film: C - 7.0% VA

T	DT	P	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Std.Dev	Seal AVG.
1	1	c	1.9			1.7		0.1	1.8
1	2	c	1.5			1.5		0	1.5
2	1	c	1.5			1.6		0.05	1.55
2	2	c	1.5			1.4		0.05	1.45
3	1	c	1.9			2		0.05	1.95
3	2	c	1.7			1.6		0.05	1.65
4	1	c	1.5			1.6		0.05	1.55
4	2	c	2			1.7		0.15	1.85

THICKNESS AVG = 1.66
Std.Dev = 0.04

Film: D - 9.0% VA

T	DT	P	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Std.Dev	Seal AVG.
1	1	c	1.3			1.6		0.15	1.45
1	2	c	1.5			1.4		0.05	1.45
2	1	c	1.3			1.45		0.075	1.375
2	2	c	1.45			1.5		0.025	1.475
3	1	c	1.55			1.5		0.025	1.525
3	2	c	1.35			1.35		0	1.35
4	1	c	1.35			1.35		0	1.35
4	2	c	1.4			1.5		0.05	1.45

THICKNESS AVG = 1.43
Std.Dev = 0.05

Table I (cont'd)

Film: E - 19% VA

T	DT	P	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Std.Dev	Seal AVG.
1	1	c	1.5			2.1		0.3	1.8
1	2	c	2.5			2.1		0.2	2.3
2	1	c	1.55			1.7		0.075	1.63
2	2	c	1.6			1.4		0.1	1.5
3	1	c	1.9			1.7		0.1	1.8
3	2	c	1.65			1.4		0.125	1.53
4	1	c	1.7			1.5		0.1	1.6
4	2	c	1.35			1.4		0.025	1.38

THICKNESS AVG = 1.69
Std.Dev = 0.08

Film: F - 29% VA

T	DT	P	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Std.Dev	Seal AVG.
1	1	c	1.5			1.6		0.05	1.55
1	2	c	1.6			2		0.2	1.8
2	1	c	1.6			1.7		0.05	1.65
2	2	c	1.7			1.7		0	1.7
3	1	c	1.6			1.7		0.05	1.65
3	2	c	1.65			1.7		0.025	1.68
4	1	c	1.8			1.6		0.1	1.7
4	2	c	1.5			1.9		0.2	1.7

THICKNESS AVG = 1.69
Std.Dev = 0.07

APPENDIX J: COMPLETE DATA OF MELT FLOW INDEX TESTING

Table I: Complete Table of Melt Flow Index Data (a)

Sample (%)	Test	Temp(F)	Wt. (G)	Time (S)	Flow
0	M1	190	NA	NA	1.567
	M2	190	NA	NA	1.518
	M3	190	NA	NA	1.509
	M-AVG	190			1.531
	M-STD	190			0.026
0	A-1	190	1.4263	498.7	1.719
	A-2	190	1.4426	548.1	1.582
	A-3	190	1.454	590.2	1.481
	A-AVG	190			1.59
	A-STD	190			0.098
4.5	M-1	190	1		1.629
	M-2	190			1.603
	M-3	190			1.616
	M-AVG	190			1.616
	M-STD	190			0.011
4.5	A-1	190	1.503	539.6	1.675
	A-2	190	1.413	576	1.475
	A-3	190	1.44	553	1.568
	A-AVG	190			1.573
	A-STD	190			0.082
7.0	M-1	190	1.434	605	1.425
	M-2	190	1.362	635	1.290
	M-3	190	1.471	645.3	1.370
	M-AVG	190			1.362
	M-STD	190			0.056
	A-1	190			1.446
	A-2				1.320
	A-3				1.206
	A-AVG				1.324
	A-STD				0.098
9.0	M-1	190			2.245
	M-2	190			2.263
	M-3	190			2.170
	M-AVG	190			2.226
	M-STD	190			0.040
	A-1	190	1.531	351	2.622
	A-2	190	1.442	387	2.240
	A-3	190	1.466	400	2.203
	A-AVG	190			2.355
	A-STD	190			0.190

Sample (%)	Test	Temp(F)	Wt. (G)	Time (S)	Flow
19	M-1	190			1.109
	M-2	190			1.068
	M-3	190			1.024
	M-AVG	190			1.067
	M-STD	190			0.035
	A-1	190	1.552	852	1.095
	A-2	190	1.513	587	1.550
	A-3	190	1.524	727	1.261
	A-AVG	190			1.302
	A-STD	190			0.188
29	M-1	150			1.540
	M-2	150			1.562
	M-3	150			1.559
	M-AVG	150			1.554
	M-STD	150			0.010
	A-1	150	1.591	624.6	1.531
	A-2	150	1.849	700	1.588
	A-3	150	1.766	639.4	1.66
	A-AVG	150			1.593
	A-STD	150			0.053

(a) MFI Flow Rate = ((427*((C2)/1.804)*2.54)/D2))

APPENDIX K: COMPLETE DATA FROM REPEATABILITY AND RELIABILITY TESTING

Table K: Repeatability and Reliability Tests
(using a 4.5% VA test sample)

A: Heat Seal Data

1: Die Loading and Seal Pressure

Sample	Force (lbs.)	Pressure (PSI)
1	101.7	381
2	100.8	343
3	101	374
4	101.7	374
5	101.4	375
6	101.8	365
7	101.9	379
8	101	376
9	101.3	377
10	NA	NA
Avg.	101.4	371.5556
STD.DEV	0.377	10.94
Range	100.8	101.9381
"n"	9	9

2: Temperature (degrees F)

Sample	Top Bar Temperature	Bottom Bar Temperature	
1	258.5	258.3	
2	258	257.9	
3	257.2	257.3	
4	257	257.5	
5	256.9	257	
6	257.4	257	
7	257.4	257	
8	257.7	256.9	
9	257.6	257.3	
10	257.5	257.4	
Avg.	257.52	257.36	
STD.DEV	0.449	0.425	
Range	256.9	258.5	256.9 258.3
"n"	10	10	

Table K (cont'd)

3: Dwell Time (in ms)

Sample	Dwell Time (in ms)
1	2095
2	2090
3	2003
4	2095
5	2095
6	2095
7	2088
8	2094
9	NA
10	NA
Avg.	2106
STD.DEV	36.42
Range	2088 2203
"n"	8

APPENDIX L: COMPLETE HEAT SEAL PILOT TEST DATA

Table L: Heat Seal Pilot Test - Complete Data

#	T	P	DT	BRK			Seal	Seal	Seal	Seal	Seal
				1	2	3	1	2	3	Avg	Std.Dev.
	(a)	(b)	(c)		(d)		(e)			(f)	
1	0	-1	-1	1	1	1	1.41	1.992	1.149	1.517	0.352
2	0	-1	0	3	3	3	3.812	3.839	3.742	3.798	0.041
3	0	-1	1	3	3	3	3.678	3.989	4.134	3.934	0.190
4	0	0	-1	2	2	2	3.758	3.076	3.388	3.407	0.279
5	0	0	0	3	3	3	4.086	3.565	3.812	3.821	0.213
6	0	0	1	3	3	3	3.812	3.683	4.204	3.900	0.222
7	0	1	-1	3	3	3	3.774	3.742	4.140	3.885	0.181
8	0	1	0	3	3	3	3.828	4.134	4.220	4.061	0.168
9	0	1	1	3	3	3	3.726	4.075	3.705	3.835	0.170
10	1	-1	-1	3	2	3	2.685	3.898	3.871	3.485	0.566
1	1	-1	0	3	3	3	4.360	3.936	4.027	4.108	0.182
2	1	-1	1	3	3	3	3.533	3.764	3.882	3.726	0.145
3	1	0	-1	3	3	3	4.317	3.619	3.871	3.936	0.289
4	1	0	0	3	3	3	4.097	4.075	3.721	3.964	0.172
5	1	0	1	3	2	3	4.317	3.356	3.581	3.751	0.410
6	1	1	-1	3	3	3	4.137	4.462	3.774	4.124	0.281
7	1	1	0	3	3	3	3.823	3.946	3.603	3.791	0.142
8	1	1	1	3	2	3	3.731	3.195	4.404	3.777	0.495
9	-1	-1	-1	1	1	1	0.107	0.193	0.209	0.170	0.045
20	-1	-1	0	1	1	1	0.188	0.161	0.193	0.181	0.014
1	-1	-1	1	1	1	1	0.193	0.172	0.183	0.183	0.009
2	-1	0	-1	1	1	1	0.129	0.166	0.145	0.147	0.015
3	-1	0	0	1	1	1	0.128	0.236	0.209	0.191	0.046
4	-1	0	1	1	1	1	0.236	0.226	0.247	0.236	0.009
5	-1	1	-1	1	1	1	0.263	0.252	0.231	0.249	0.013
6	-1	1	0	1	1	1	0.183	0.242	0.023	0.149	0.092
7	-1	1	1	1	1	1	0.118	0.199	0.193	0.170	0.037
8	2	0	-1	2	2	2	1.842	1.181	1.450	1.491	0.271
9	2	0	0	2	2	2	2.282	2.373	2.223	2.293	0.062
30	(below)										

Table L (cont'd)

#	T	P	DT	BRK			Seal	Seal	Seal	Seal	Seal
				1	2	3	1	2	3	Avg	Std.Dev.
	(a)	(b)	(c)		(d)		(e)			(f)	
30	2	0	1	2	2	2	2.846	2.529	2.164	2.513	0.279
1	2	-1	-1	2	2	2	1.621	1.960	1.654	1.745	0.153
2	2	-1	0	2	2	2	2.298	2.717	2.701	2.572	0.194
3	2	-1	1	2	2	2	2.438	2.717	2.701	2.619	0.128
4	2	2	-1	2	2	2	2.266	2.121	2.518	2.302	0.164
5	2	1	0	2	2	2	2.624	2.545	2.439	2.536	0.076
6	2	1	1	2	2	2	2.898	2.491	2.421	2.603	0.210
7	3	0	-1	2	2	2	0.591	0.494	0.634	0.573	0.058
8	3	0	0	2	2	2	0.775	0.751	0.494	0.673	0.127
9	3	0	1	2	2	2	0.795	0.773	0.768	0.779	0.012
40	3	1	-1	2	2	2	0.650	0.601	0.532	0.594	0.049
1	3	1	0	2	2	2	0.548	0.859	0.451	0.619	0.174
2	3	1	1	2	2	2	0.811	0.789	0.800	0.800	0.009
3	3	-1	-1	2	2	2	0.532	0.677	0.709	0.639	0.077
4	3	-1	0	2	2	2	0.789	0.811	0.739	0.780	0.030
5	3	-1	1	2	2	2	0.838	0.768	0.907	0.838	0.057

(a) The temperature Code is noted in Appendix J-1

(b) The pressure codes are noted:

Code	Pressure
-1	10
0	50
1	500

(c) The dwell time codes are noted:

Code	Dwell Time
-1	200
0	500
1	1800

(d) The "BRK.1" refers to the type of break for seal one (etc. for each seal)

Code	Break
1	Peel
2	Seal Split
3	Interface Split

(e) The Seal Average is noted in lbs. and is accurate to three decimal places

(f) The Seal Std.Dev is the seal standard of deviation, and is accurate to three decimal places

APPENDIX M: HEAT SEAL OPTIMIZATION EXPERIMENT - COMPLETE TEST DATA

Table M: Heat Seal Optimization Experiment - Complete Data

A: Film = 0% VA

T (a)	DT (b)	P (c)	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Seal AVG. (d)	Std.Dev.
1	1	c	2.072	2.695	2.464	2.808	2.03	2.414	0.317
1	2	c	3.055	3.001	3.007	2.878	2.797	2.948	0.095
2	1	c	3.442	3.388	3.490	3.313	3.388	3.404	0.059
2	2	c	3.522	3.463	3.517	3.517	3.672	3.538	0.070
3	1	c	3.522	3.388	3.485	3.431	3.399	3.445	0.051
3	2	c	3.302	3.264	3.189	3.506	3.442	3.341	0.117
4	1	c	3.297	3.452	3.538	3.302	3.302	3.378	0.099
4	2	c	3.431	2.948	3.479	3.393	3.200	3.290	0.196

Max. Seal Strength = 3.5382 @ T=2,DT=2
Std.Dev @ Max. = 0.0703

B: Film = 4.5% VA

T (a)	DT (b)	P (c)	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Seal AVG. (d)	Std.Dev.
1	1	c	1.278	1.068	1.170	1.128	1.272	1.183	0.082
1	2	c	1.238	1.305	1.289	1.348	1.170	1.270	0.061
2	1	c	3.791	3.823	3.672	3.656	3.436	3.676	0.136
2	2	c	3.597	3.227	3.705	3.683	3.828	3.608	0.204
3	1	c	4.070	3.839	3.968	3.726	4.236	3.968	0.177
3	2	c	2.867	3.109	3.420	3.195	3.071	3.132	0.180
4	1	c	2.255	3.640	3.678	4.392	3.442	3.481	0.693
4	2	c	2.991	3.313	3.007	3.667	4.102	3.416	0.422

Max. Seal Strength = 3.9678 @T=3,DT=1
Std.Dev @ Max. = 0.1774

Table M (cont'd)

C: Film = 7.0% VA

T (a)	DT (b)	P (c)	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Seal AVG. (d)	Std.Dev.
1	1	c	1.165	1.203	1.208	1.219	1.407	1.240	0.085
1	2	c	1.133	1.358	1.230	1.197	1.235	1.231	0.073
2	1	c	3.474	3.715	3.871	3.442	3.624	3.625	0.158
2	2	c	3.544	3.479	4.177	3.527	3.640	3.673	0.257
3	1	c	3.973	3.436	3.646	4.242	4.075	3.874	0.293
3	2	c	4.032	3.576	4.376	3.613	4.478	4.015	0.374
4	1	c	3.887	3.560	4.387	3.748	3.554	3.827	0.306
4	2	c	4.531	3.780	3.817	3.984	3.860	3.994	0.277

Max Seal Strength = 4.015 @T=3,DT=2
MAX's std dev= 0.374

D: Film = 9.0% VA

T (a)	DT (b)	P (c)	Rep 1	Rep 2	Rep 3	Rep 4	Seal Rep 5 (d)	AVG.	Std.Dev.
1	1	c	1.525	1.589	1.541	1.632	1.562	1.570	0.038
1	2	c	1.766	1.750	1.670	1.498	1.600	1.657	0.099
2	1	c	3.683	3.989	4.650	3.452	4.419	4.039	0.445
2	2	c	3.721	3.506	3.764	3.925	4.429	3.869	0.310
3	1	c	3.823	3.705	3.629	3.989	3.683	3.766	0.128
3	2	c	3.560	3.640	4.993	3.270	3.646	3.822	0.601
4	1	c	3.544	4.032	3.925	3.662	3.479	3.728	0.215
4	2	c	3.270	3.323	3.205	3.565	3.195	3.312	0.135

Maximum Strength = 4.039 @T=2,DT=1
Std.Dev @ Max. = 0.4453

Table M (cont'd)

D: Film = 9.0% VA

T (a)	DT (b)	P (c)	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Seal AVG. (d)	Std.Dev.
1	1	c	0.322	0.279	0.354	0.279	0.263	0.300	0.034
1	2	c	0.284	0.332	0.408	0.381	0.316	0.345	0.044
2	1	c	1.192	1.417	1.272	1.407	1.358	1.329	0.086
2	2	c	1.530	1.648	1.289	1.664	1.514	1.529	0.134
3	1	c	2.577	2.829	2.921	2.942	2.717	2.797	0.136
3	2	c	2.878	2.985	2.819	2.663	3.209	2.911	0.182
4	1	c	3.420	3.340	3.168	3.238	2.862	3.206	0.192
4	2	c	2.765	3.737	3.238	3.667	3.404	3.362	0.348

Max. Seal Strength = 3.3622 @T=4,DT=2
Std.Dev @ Max. = 0.3485

Table M (cont'd)

F: Film = 29% VA

T (a)	DT (b)	P (c)	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Seal AVG. (d)	Std.Dev.
1	1	c	0.649	0.773	0.300	0.690	0.719	0.628	0.168
1	2	c	0.869	0.515	0.826	0.912	0.279	0.681	0.245
2	1	c	0.934	1.519	1.541	1.348	1.020	1.272	0.252
2	2	c	1.246	1.611	1.552	1.321	1.391	1.424	0.138
3	1	c	1.476	1.976	2.518	2.212	2.115	2.059	0.342
3	2	c	2.099	1.793	2.212	2.013	2.523	2.128	0.241
4	1	c	2.502	2.405	1.783	1.906	2.685	2.256	0.350
4	2	c	2.164	2.293	2.191	2.411	2.056	2.223	0.121

Maximum Strength = 2.256 @T=4,DT=1
Std.Dev @ Max. = 0.3501

(a) For Temperature Codes see Table I-2
(b) Dwell Time Codes:

Code	Dwell Time
1	500
2	1800

(c) The Pressure was held constant at 200 PSI
(d) The seal strength is noted in grams

APPENDIX N: HOT TACK PILOT TEST - COMPLETE DATA

Table N: Hot Tack Pilot Test - Complete Data

#	T	P	DT	Test Weight (grams)						Type of Failure	Max Seal Strength
	(f)	(psi)		(s)	25	50	75	100	125		
	(a)	(b)		(c)	(d)						(e)
1	-1	1	1	O	O					P	0
2	-1	1	0	O	O					P	0
3	-1	1	-1	O						P	0
4	-1	0	1	O						P	0
5	-1	0	0	O						P	0
6	-1	0	-1	O						P	0
7	-1	-1	1	O						P	0
8	-1	-1	0	O						P	0
9	-1	-1	-1	O						P	0
10	0	1	1	-	X	X	XX	OO		B	100
11	0	1	0	-	X	OO				B	50
12	0	1	-1	-	X	X	O			B	75
13	0	0	1	-	X	O				B	50
14	0	0	0	-	X	O				B	50
15	0	0	-1	-	X	O				B	50
16	0	-1	1	-	XX	O				B	50
17	0	-1	0	-	X	O				B	50
18	0	-1	-1	-	X	OO				B	50
19	1	1	1	X	O					B	25
20	1	1	0	X	O					B	25
21	1	1	-1	X	O					B	25
22	1	0	1	X	O					B	25
23	1	0	0	X	O					B	25
24	1	0	-1	X	O					B	25
25	1	-1	1	X	O					B	25
26	1	-1	0	X	O					B	25
27	1	-1	-1	X	O					B	25

(a) The Temperature Codes are as Follows: (degrees F)
Code=Temp.: -1=210 F, 0=220 F, and 1=230 F
(b) The Pressure Codes are as Follows: (in PSI)
Code=PSI: -1=10, 0=50, and 1=500
(c) The Dwell Time Codes are as Follows: (in ms)
Code=Dwell Time: -1=200, 0=500, and 1=1800
(d) Legend: O=SEAL PASSES; X=SEAL FAILS; B=BREAK FAILURE; P=PEEL FAILURE (MULTI O'S OR X'S =MULTI REPS)
(e) The Maximum Seal Strength is noted in grams

APPENDIX O: HOT TACK EXPERIMENT - COMPLETE DATA

TABLE O-1: Hot Tack Experiment With Method A - Complete Test Data

A: Film = 0% VA

Temp.	Tm	Hot Tack Seal Str.	Type of Fail	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					
215		0	P	OO				
225		25	P	XX	OO			
235	*	0	B	OO				
245								

B: Film = 4.5% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					
210		0	P	OO				
220	*	50	P	X	X	OO	O	
230		25	B	X	OO			
240		25	B	XX	O			
250		0	B	O				

C:Film = 7.0% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Fail	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					
205		0	P	O				
215	*	50	P	-	X	O		
225		25	B	X	O			
235		0	B	O				

Table O (cont'd)

D: Film = 9.0% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Fail	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					

205		0	B	O	O			
215	*	25	B	X	O			
225		25	B	X	O			
235		0	B	OO				

E: Film = 19% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Fail	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					
175		0	P	O	O			
185	*	25	P	X	O	O		
195		25	B	X	OO			
205		25	B	X	O			

F: Film = 29% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Fail	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					
150		50	P	X	X	O		
160	*	NA	NO BREAK					
170		NA	NO BREAK					
180		75	B	X	X	X	O	
190		75	B	-	-	X	O	
200		0	B	OO	O	O		

Table O (cont'd)

TABLE O-2: Hot Tack Experiment With Method B - Complete Test Data

A: Film = 0% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					
205		0	NO ADHESION					
215		0	NO ADHESION					
225		0	P	O				
235	*	0	P	O				
245		25	P	X	O			
255		0	P	OO				
265		0	B	O				

B: Film = 4.5% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					
200		0	NO ADHESION					
210		0	P	O	O			
220	*	25	B	X	O			
230		0	P	O				
240		0	B	O				
250		0	B	O				

C: Film = 7.0% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					
185		0	NO ADHESION					
195		0	NO ADHESION					
205		0	NO ADHESION					
215		0	P	O				
225		0	B	O				
235		0	B	O				

Table O (cont'd)

D: Film = 9.0% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					
185		0	NO ADHESION					
195		0	NO ADHESION					
205		0	NO ADHESION					
215	*	0	P	O				
225		0	B	O				
235		0	B	O				

E: Film = 19% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					
185	*	25	P	X	O			
195		50	B	-	X	O		
205		25	B	X	O			
215		0	B	O				

F: Film = 29% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)				
				25	50	75	100	125
(a)	(b)	(c)	(d)					
160	*	50	P	-	X	O	O	
170		75	B	-	-	XX	O	O
180		0	B	O	O	O		

Table O (cont'd)

TABLE O-3: Hot Tack Experiment With Method C - Complete Test Data

A: Film = 0% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)							
				25	30	35	40	50	75	100	125
(a)	(b)	(c)	(d)								
205		0	NO ADHESION								
215		0	NO ADHESION								
225		0	B	O							
235	*	0	P	O							
245		25	P	X	OO	O	-	O			
255		0	P	OO							
265		O	B	O							

B: Film = 4.5% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)					
				25	30	35	40	50	75
(a)	(b)	(c)	(d)						
200		0	NO ADHESION						
210		0	P	O					
220	*	25	B	X	O	O	-	O	
230		0	B	O					
240		0	B	O					
250		0	B	O					

Table O (cont'd)

C: Film = 7.0% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)					
				25	30	35	40	50	75
(a)	(b)	(c)	(d)						
185		0	NO ADHESION						
195		0	NO ADHESION						
205		0	NO ADHESION						
215	*	25	P	X	O	-	OO		
225		10	B	-	X	O	O		
235		0	B	O					

D: Film = 9.0% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)					
				25	30	35	40	50	75
(a)	(b)	(c)	(d)						
185		0	NO ADHESION						
195		0	NO ADHESION						
205		0	NO ADHESION						
215	*	15	P	-	X	X	O		
225		0	B	-	O	-	O		
235		0	B	O					

E: Film = 19.0% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)					
				25	30	35	40	50	75
(a)	(b)	(c)	(d)						
185	*	25	P	X	O				
195		50	B	-	X	O	-	O	O
205		25	B	X	O				
215		0	B	O					

Table O (Cont'd)

F: Film = 29.0% VA

TEMP.	Tm	Hot Tack Seal Str.	Type of Failure	Test Weight (grams)							
				25	30	35	40	50	75	100	125
(a)	(b)	(c)	(d)								
150		50	P	-	X	O	O				
170		80	B	-	-	XX	X	O	O	O	O
180		0	B	O	O	O					

(a) The Temperature is in degrees Celsius
(b) Tm = Melt Temperature, and is designated with an 'X'
(c) The hot tack is denoted in grams per one inch sample width
(d) Type of Failure = The type of failure during the test: Peel, Break or No Adhesion