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The need for the definition and acceptance of a standard test methodology and standard material requirements for MRE packaging material qualification has been noted. In order to define these standards a number of mechanical tests were performed on films provided by Cadillac Products and NSC and the corresponding material properties were obtained. These tests were performed under various conditions including temperature, backing material, rate, orientation, etc. Based on these results minimum requirements have been defined for future MRE packaging films. In addition, scratch testing of the packaging material proved to be one of the most reliable and useful test methods. As well, the use of scratch testing methodology potentially renders some previous tests unnecessary (such as puncture and tensile). Finally, the standard minimum requirements for the qualification of a film to be used in an MRE packaging application are quantitatively characterized.

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Identification and Development of Simple Acceptance Tests for MRE Film Pouch Materials

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Summary

The need for the definition and acceptance of a *standard test methodology* and *standard material requirements* for MRE packaging material qualification has been noted. In order to define these standards a number of mechanical tests were performed on films provided by Cadillac Products and NSC and the corresponding material properties were obtained. These tests were performed under various conditions including temperature, backing material, rate, orientation, etc. Based on these results minimum requirements have been defined for future MRE packaging films. In addition, scratch testing of the packaging material proved to be one of the most reliable and useful test methods. As well, the use of scratch testing methodology potentially renders some previous tests unnecessary (such as puncture and tensile). Finally, the *standard minimum requirements* for the qualification of a film to be used in an MRE packaging application are quantitatively characterized.

Introduction

-Background Information:

MREs, or Meals Ready to Eat, were introduced in 1981 to fill the need for an easily transported and prepared field ration. One of the key requirements for the MRE is a long shelf-life—while maintaining nutritional value and palatability. It is obvious that the MRE packaging is a key factor in determining the performance of the MRE itself. To that end, the materials used in MRE packaging are critical, both to the quality and the cost-effectiveness of the finished product.

Some material properties that are generally important in packaging materials include glass-transition temperature (T_g), melting temperature (T_m), the ductile-brittle transition temperature (T_{db}), heat-seal temperature, oxygen and moisture permeability, and cost. These properties can be easily quantified and addressed with little effort. However, there are other properties that are relevant to MRE packaging materials that are more difficult to describe, let alone quantify. These properties include the appearance and durability of the pouch films under different environmental and handling conditions, i.e., rough handling resistance, etc.

A previous CORANET I research project (STP 1008) studied the most common modes of failure for MRE pouches. This project identified that the most common defects found in disqualified MRE pouches were abrasions, delaminations, pinholes, and cuts. Among these defects, abrasion and delamination were found to be the most troublesome to ration producers. Abrasion and delamination related defects amount to over 80% of all defects in MRE pouches and are often times difficult to locate. Even when identified, there is often uncertainty as to whether or not a given defect is a critical defect[†]. The inability of ration producers to deal with the above problems has resulted in significant expense in both material and labor. Therefore, it is highly desirable to reduce pouch material defects—especially abrasion and delamination related defects—if at all possible.

[†]Critical defects are defects that could affect the quality and safety of the packaged food products.

Currently there are no simple test methods that the ration producers can conduct that may identify problem materials before the start of a production run. As well, there are no testing standards that producers may perform that correlate to end item rough handling test performed by the US Army's Natick Soldier Center (NSC). The establishment of a simple, precise, and uniform test method and acceptance criterion would ensure that the materials received by ration producers will achieve the expected and specified performance. Such testing procedures would also allow the validation and approval of materials before their use in production, thereby preventing the loss of significant man hours and cost otherwise may be lost due to a failed lot.

The current ASTM test standards for plastic films include the dart impact test, Elmendorf tear resistance test, and tensile test. Other tests, such as the J-integral, fracture, and delamination tests widely utilized in the paint and automotive industry, may also be relevant to the property requirements for packaging materials. A new ASTM standard (ASTM D7027-05) for scratch performance evaluation of plastics and coatings has recently been developed by Texas A&M University. This scratch test method has not been utilized for evaluation of pouch laminates in the past. In this report, we will focus on how the new ASTM scratch test method can help address quantitative performance evaluation of MRE pouch laminates.

One major advantage of the ASTM scratch testing is that it allows for the direct measurement of certain key material properties that are especially relevant to the MRE pouch application: wear, abrasion, delamination, and tearing. Furthermore, scratch properties will usually correlate with other properties of the material, such as tensile strength and puncture resistance. Another advantage of scratch testing is that it enables fast, reliable, and reproducible testing to be conducted under various environmental conditions.

Typical failure patterns observed in MRE packaging material frequently can be related to different damage transitions in the scratch test. The linear load increase type of Scratch test can identify and quantify three different MRE film failure modes, all with one single test. As well, it can provide well quantified information including scratch visibility, depth, width, and hardness.

-Cost Effectiveness

According to data gathered from the ration producers and from DSCP, there are over 100,000,000 packaged food items prepared each year. A potential saving of about 5-25% in packaging material cost and 10-20% in weight reduction (if non-foil packaging material is to be developed and utilized) can be expected if the project is implemented at NSC. This translates into a savings of over $500,000 each year. If the new material is more resistant to rough handling and to delamination (i.e., better seal), then savings can be doubled.

If the packaging material is non-foil based, there will be many more intangible benefits, as well. These benefits include (a) improved recyclability of packaging materials, (b) enhanced food safety via better, cheaper, and easier inspection techniques, (c) shorter lead times for commercially available films which may reduce inventory costs, (d) easier processing through the use of microwave heating, and (e) additional menu items.

Project Plan

The project was separated into three different phases listed below.

Phase I:

To collect and document data of all of the currently utilized laminated military ration packaging materials in order to identify the characteristics and performance requirements that allows them to meet severe service requirements, and document those requirements as the minimum requirements.

Phase II:

To identify simple procedures for confirming or validating the performance requirements that are essential for pouch and lid laminate materials.

Phase III: (to be jointly implemented at NSC)

To deliver a matrix listing ration packaging material performance requirements to be used by the Government as a guide for introducing new material specifications in the future. To include procedures for testing method procedures to train users who will be conducting material acceptance tests.

Objectives

(1) To identify the essential physical and mechanical properties and/or characteristics of military ration packaging materials that are crucial to endure the severe rough handling and hot or cold weather conditions required during service.

(2) To quantify the essential performance requirements for military ration packaging.

(3) To develop or identify one or a set of simple test procedures that can be conducted by material developers, pouch retorters, or NSC technical personnel to assure that the final product will meet military field performance requirements.

(4) To develop a standard test method for MRE packaging films and to work towards its adoption as an ASTM standard.

Definitions

MRE: Meal Ready to Eat

Low Temp.: -20°C

Room Temp.: 80°C

High Temp.: 180°C

R1: Cadillac Non-Retort Film #1: 98 GA OPP/15#PE/.0007F/3 mil sealant (0.15 mm)

R2: Cadillac Non-Retort Film #2: 48 GA PET/10#PE/.00035F/2 mil sealant (0.09 mm)

R3: Cadillac Non-Retort Film #3: 60 GA BON/10#PE/.00035F/3 mil sealant (0.13 mm)

R4: Cadillac Non-Retort Film #4: 98 GA PET/15#PE/.0007F/3 mil sealant (0.12 mm)

F1: Cadillac Non-Retort Film #1 lateral direction

F2: Cadillac Non-Retort Film #2 lateral direction

Smurfit Quad-Layer Retort Pouch: Nylon/PET/Foil/PP

Smurfit Tri-Layer Retort Pouch: PET/Foil/PP

R: front side tested with rubber backing

BR: back side tested with rubber backing

P: front side tested with plastic backing

BP: back side tested with plastic backing

M: front side tested with metal backing

BM: back side tested with metal backing

Mar/Scratch Resistance test: a recently developed test method used to determine the resistance of polymers against surface scratches

T_g: Glass Transition Temperature

T_m: Melting Temperature

Results and Discussion

All materials currently used in MRE packaging have previously been tested and certified for their current uses. Some of these prior tests, which are beyond the scope of this STP, include moisture and oxygen barrier properties, thermal properties, rough handling resistance, vacuum stability, hot fill durability, heat sealability, etc. Since all current films (including those chosen for this project) meet these criteria it is logical to conclude that new films (which may have unknown properties) can be compared to existing films (which are known to pass the aforementioned tests). The minimum material performance specification can therefore be stated as the minimum specification measured during this project.

The main critical defect and failure mode in packaging films occurs when the foil layer (that is responsible for minimizing diffusion of H_2O and O_2) is damaged and/or punctured. This type of a defect will cause the shelf life of the product to be greatly reduced. Rising load scratch testing was used to study at what point foil damage occurred on the sample films. As the scratch stylus traverses the film surface, the normal load increases. At some point the load will be sufficient to damage the foil within the film laminate.

The damage location along a given scratch can be correlated to the load that was applied. Therefore a quantitative measure can be made (Critical Normal Load for Foil Puncture) to compare between materials. Foil puncture was identified using two separate methods. First, optical macroscopic examination was conducted. For this test a strong, pinpoint, light source was aimed at the back of a scratched film specimen. The person conducting the experiment then looks for the point at which the light becomes visible through the film. Second, analysis using a scanning electron microscope (SEM) was employed in order to confirm and validate the above method. It can be seen in Figure 1 that the point at which the foil damage occurs is the same for both optical macroscopic testing and SEM testing. Because the macroscopic analysis yields the same results as the much more time consuming and costly SEM procedure, it is clearly a more logical choice.

Different critical loads at delamination, foil damage, and complete tear are shown macroscopically in Figure 2. In Figure 3 SEM images of different failure modes are shown, in which the critical points correlate 100% with those observed optically.

-Tensile Modulus and Tensile Strength

Tensile modulus and strength of the MRE films were calculated using the relevant ASTM standards. The results are shown in Figure 4. It can be seen that the minimum tensile modulus of the films occurs for film #1, both at low and room temperatures. The ultimate tensile strength for film #4 seems to be the minimum of the films and pouches both at room and low temperatures. This indicates that if a minimum requirement for tensile modulus and strength is to be defined as a standard, it should not be lower than any of the mentioned properties for the films #1 and #4.

-Scratch Test Vs. Puncture Test

In Figure 5, scratch and puncture critical loads are shown for tests performed at low and room temperature. One interesting finding to point out is the similar trend that these two material properties (scratch critical load and puncture critical load) show relative to each other. It can be seen that for every increase/decrease of puncture critical loads, critical loads for scratch of the material will also increase/decrease and this case is even observed for small differences such as the difference between film #1 and film #2. In this case there seems to be no need to perform both tests on a film and the puncture test can be substituted by the scratch test. Also for the standard basis, the minimum requirement for a non-retort film in terms of scratch and puncture critical loads would be that of film #2 at low and room temperatures. It should be noted that it is definitely necessary for one of the two tests to be performed and the exposure to low temperatures can depend on the final application of the film. If there is a possibility of the film being exposed to low temperatures, low temperature tests must also be run.

-Temperature Effect

In order to observe the influence of temperature on the performance of MRE pouch materials, scratch critical loads at delamination, foil damage, and complete tear for all of the tested films are shown at high (180°F), room (80°F), and low temperatures (-20°F) in Figure 6. These tests were conducted at the rate of 60 mm/s and the load range was 1-40N. It can be seen that all of the films show lower critical loads at high temperatures and again at low temperatures. The performance of the tested films are clearly optimum at temperatures close to room temperature. As well, the temperature issue is of more significance in hot weather compared to cold weather because of the sudden decrease in scratch critical loads of the films. The fact that temperature increase causes negative effects on the scratch resistance of the films can be explained partially by the drop of tensile modulus of the films due to the temperature increase. With the increase in temperature the polymer on the surface of the films becomes softer and more pliable, which allows the scratch tip to plough through the material with less resistance. The decrease in scratch critical loads due to the temperature decrease is smoother than the temperature increase and even film #3 shows better performance at low temperatures. Therefore, the temperature dependence of the films' scratch properties depends on the material. In some cases lowering the temperature would even cause the scratch critical loads to increase, although it should be also noted that in this case the difference between the scratch critical loads is in the same range as the standard deviation. However, because of the need to ensure the performance of films at temperature extremes it is important that testing be conducted at different temperatures. Because a scratch test can be conducted at any arbitrary temperature of interest it is possible to evaluate film durability in whatever environment it may be used.

-Scratching Rate Effect

Figure 7 illustrates the effect of scratch rate on film properties. In this case all tests were performed at 1-40N load range with plain (aluminum) backing. It should be noted that increasing the test rate (strain rate) results in a decrease of the measured critical loads properties. This test was conducted on one of the films and the trend is expected to be similar for all of the films. This trend is explained by the viscoelastic nature of polymers. It is well known that plastics become less ductile when it is subjected to high rate of testing. As a result, cracking and puncture can take place more readily. In reality the failures and defects that could occur on MRE packaging would most likely be caused with high rates. Dropping a box full of MREs and generalized rough handling (such as a crate shaking on the back of a truck or in an airplane) are both examples where the strain rate is relatively high. Therefore for our standard requirement consideration, the scratch tests should be performed at high speeds and it is recommended that the rate of 100mm/s be defined as our standard rate of testing. (The same rate as the industrial standard.)

-Film Backing Effect

Figure 8 shows the scratch critical loads for all of the tested films with different backings, both on their front (outer) and back (inner) sides. The scratch rate and load range for all of these tests were 60mm/s and 1-40N. The film backings were Neoprene rubber, PMMA sheet, and aluminum. Film backings are the surface on which the films are clamped for the scratch test. It can be seen that in most cases the rubber backed samples have better scratch resistance than the other two backings. Also, note that in most cases the front side of the films show higher scratch resistance compared to the back side. The lower scratch resistance of the plastic-backing films can partially be explained by the lack of friction between the films and the substrate. Other than the effect of the friction between the backing and the surface material of the film being tested, the backing can also play a role as a support and a stress distributor. In other words, if a 20 Newton normal force was pushed against a rubber backed specimen due to its' low tensile modulus the rubber backing would undergo a noticeable amount of strain (deformation) while this would not occur for a metal (aluminum) backed specimen. It seems that for MRE packaging the elastic modulus of the backing would have much more significance than the friction between the backing and the film. In other words, the food filled in the packaging could be emulated by the proper backing. In this case the friction between the two would not be of significant importance. As mentioned previously to determine the minimum requirements for the MRE packaging scratch standards the worst case scenario (which would be the metal backing) should be chosen. The fact that the front side of the films show better scratch resistance than that of the back sides is obvious and one of the explanations can be the closer distance of the Aluminum foil to the back side of the films. As well, the outermost layer of the films, which is PET, is designed for abrasion resistance, whereas the inner layer, which is PP, is intended for heat sealability rather than durability.

Scratch testing for standard purposes should be performed on both sides of the film. The outer surface is expected to have higher scratch resistance. In terms of the scratch critical loads, the standard requirements for the film is to have critical loads for foil damage, delamination, and complete tear of at least the minimum amount derived from all of the tested samples.

-Extrusion Orientation Effect

Scratch behavior with respect to molecular orientation is depicted in Figure 9. Note that testing the films along the lateral direction will only cause a slightly noticeable decrease in scratch resistance whereas the trends will remain similar to those cut along the melt flow direction. The direction of the cut samples and the performed scratch tests are shown in Figure 10. The testing rate and load range were 60mm/s and 1-40N. The issue of concern here is that in reality, the carrier of the box of MREs or the person who is in charge of handling them would not know which direction the melt flow would have been and definitely will not selectively drop or roughly handle them in one direction in order for the scratches or damages to be caused in the direction with the higher scratch resistance. The scratches and damages will occur in all of the possible directions and therefore the material has to be tested in both directions to find the minimum scratch resistance. In most cases the direction that is perpendicular to the melt flow direction will have a lower resistance to scratch and therefore its' critical loads should be considered to define the minimum requirements.

Conclusion and Recommendations

To establish a defining criterion for optimum MRE packaging, various material properties were examined for four sets of films provided by Cadillac Products. These films are used for *non-retort* applications, such as *wheat snack bread, shortbread cookies, and beef jerky*. Film #2 (48 GA PET/10#PE/.00035F/2 mil sealant) was chosen as the representative film for the material performance specification. This material was chosen because its properties were consistently lower than the other films studied during this project, therefore it is an ideal minimum standard.

Based on the scratch properties of this film, a set of minimum performance requirements for non-retort packaging has been established as follows:

Crit. Load Delamination	6.5 N
Crit. Load Foil Damage	4.5 N
Crit. Load Complete Failure	12 N
Ultimate Tensile Strength	42 MPa
Tensile Modulus	1 GPa

Scratch critical loads generally decrease as the test velocity (strain rate) increases. Therefore, scratch testing parameters, including rate, should be standardized. A standard test rate of 100 mm/s should be specified. This will disallow any confusion about the scratch rate and as well has the advantage of being the standard rate used in industrial applications.

Due to the direct relationship between tensile modulus and scratch resistance it is not necessary to conduct a specific tensile test. However, it is desirable for the film to possess an ultimate tensile strength of 42 MPa and a tensile modulus of 1 GPa.

It was also explained that the material scratch properties degrade with temperature increase. Therefore the environmental temperature in which the material is used is a factor that has to be considered and tests have to be performed at that temperature. For example, if the MRE packaging is supposed to be used in the hot deserts of Africa, it has to have the ability to withstand the high temperatures while maintaining minimum material performance.

The scratch resistance of the front side of the films is higher than that of the back side (this definitely would be predictable because of the film composition). Therefore, separate tests are required for both front and back sides of the film. One of the most important factors that has a significant influence on scratch critical loads is the film backing. In most cases the scratch critical loads would increase as the backing hardness decreases. Therefore, a hard backing material, such as aluminum or steel, should be used. The primary reason for this preference is the high modulus and hardness of these materials and that they would produce the lowest critical loads for the films. A secondary benefit is that these materials are more durable, and will result in a more reliable test apparatus.

One of the other important influential factors effecting our material scratch properties (for polymers) is the melt flow direction and its relation to the direction of the scratch test. It is known that, when polymers are molded or extruded, the chain-like molecules prefer to orient themselves in the direction in which the melt is flowing. This phenomena will result in an anisotropic material which will show different scratch resistance in the lateral and transverse directions causing the scratch critical loads to be lower than that of the film tested in the melt flow direction. The main idea for us is to develop the standard requirements and therefore it is also necessary to take into consideration the minimum critical loads that are obtained from our films.

Finally the standard deviation and error of the scratch testing has to be taken into consideration especially because the scratch test is a relatively newly developed standard. The standard deviation of the resulting critical loads typically has to be less than 10%. If standard deviations of 15% or higher are produced, retesting, increase in number of tests, and/or compatibilization of the test with various parameters has to be taken into consideration. Although as shown in Figures 7 and 8, the scratch test performed on the films were highly repeatable in our case with a maximum standard deviation of less than 10%.

Future Concerns

This STP succeeded in identifying the key properties of concern for packaging films, and in identifying a test methodology and standard for some packaging films. However, this project was also somewhat limited in scope. The standard should be better defined, and should be specific to packaging types that may be fundamentally different. For example, separate standards are needed for retortable and non-retortable packaging, yet this project focused primarily on non-retort films. As well, the explicit test procedures need to be defined, perhaps in accordance with ASTM procedures. This suggests that future work may be necessary.

Summary of Recommendations

- Scratch testing is advantageous in that it is efficient and may replace several other test methods. It is also uniquely suited to some properties of concern for packaging films (such as wear or puncture resistance)
- Scratch testing is repeatable and reliable for packaging film applications.
- The minimum performance specification for non-retort packaging films studied during this project should be specified as:

Crit. Load Delamination	6.5 N
Crit. Load Foil Damage	4.5 N
Crit. Load Complete Failure	12 N
Ultimate Tensile Strength	42 MPa
Tensile Modulus	1 GPa

- Scratch tests should be performed according to the ASTM standard D7027-05; specifically 100 mm/s rate and 1mm diameter spherical stainless steel tip.
- Scratch tests should be performed with a hard (aluminum or steel) backing material.
- Scratch tests should be performed on both the “outside” and “inside” surfaces of the film.
- Scratch tests should be performed transverse with respect to the melt flow direction.
- Scratch testing should take into account the intended use of the final product. Films that are expected to see use in temperature extremes should be tested to ensure that the above performance spec is met in those conditions.
- The Government (i.e., NSC) may wish to obtain its own scratch testing instrument in order to investigate new film materials and to qualify existing materials.

- Scratch testing machines may be used by film producers and packaging companies in order to perform quality assurance and qualification testing on films and pouch materials.
- Future study is recommended in order to consider expanding the standard to other applications, including retortable as well as non-retortable packaging, etc.

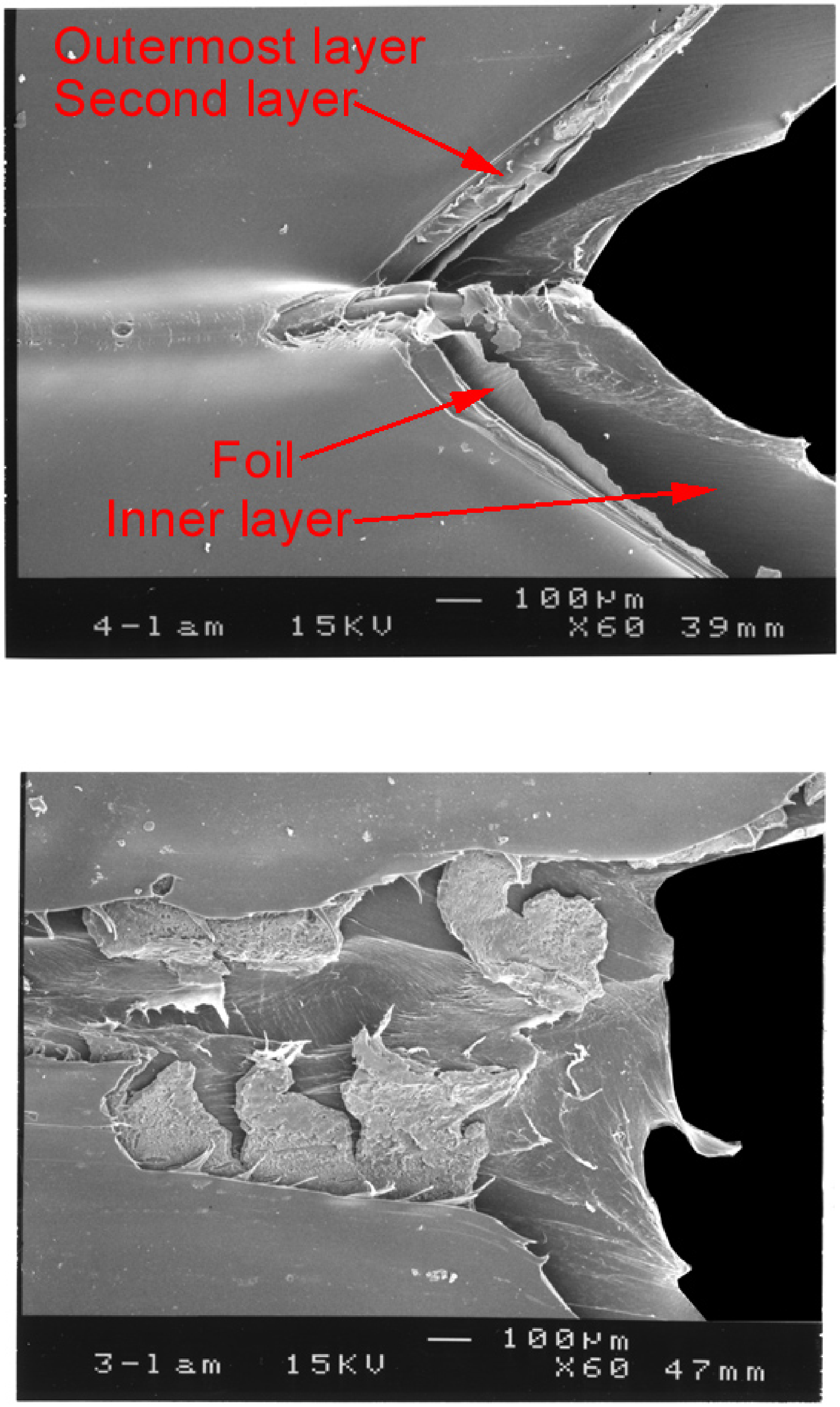


Figure 1: SEM images at critical load for foil damage.

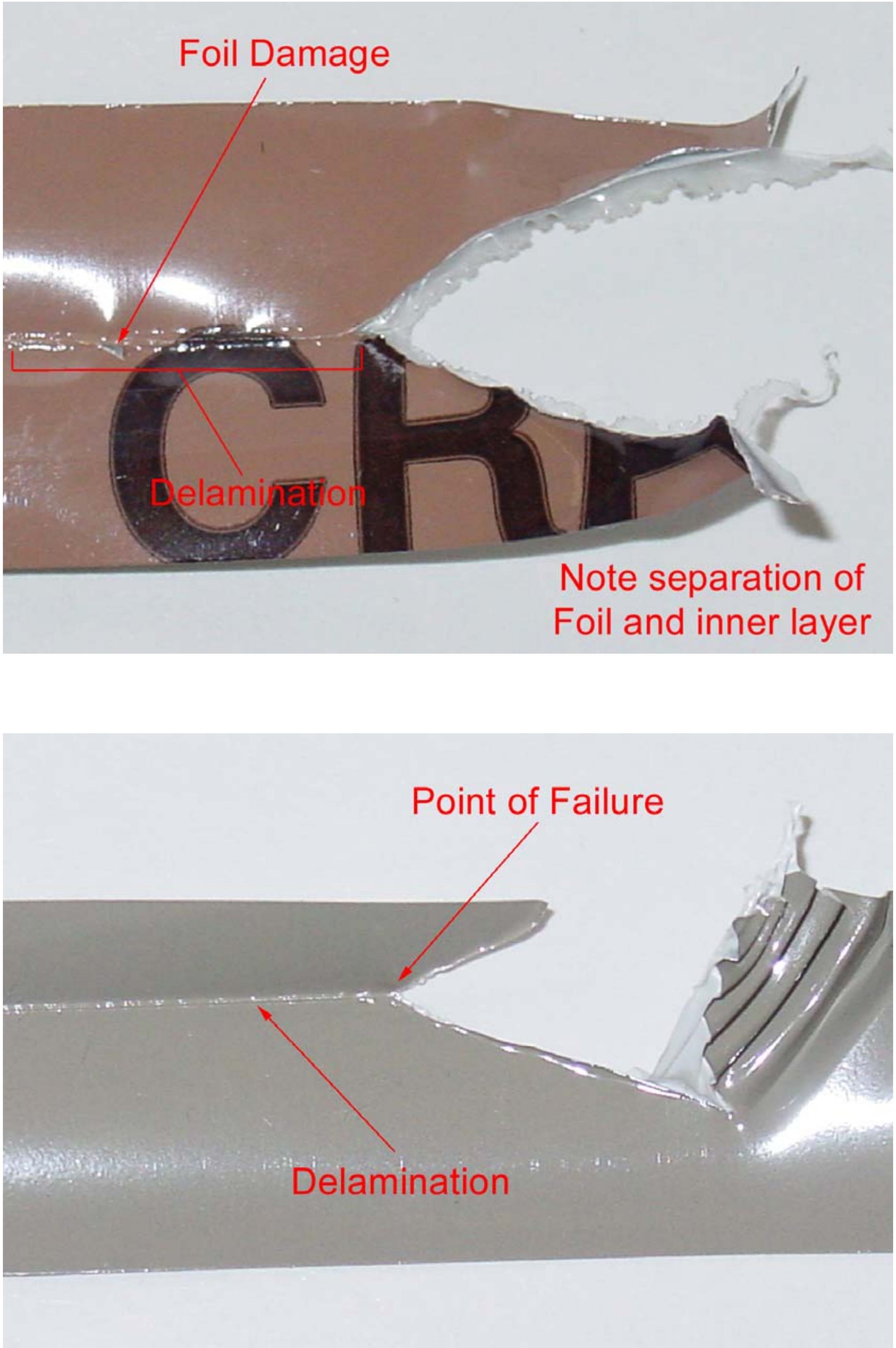
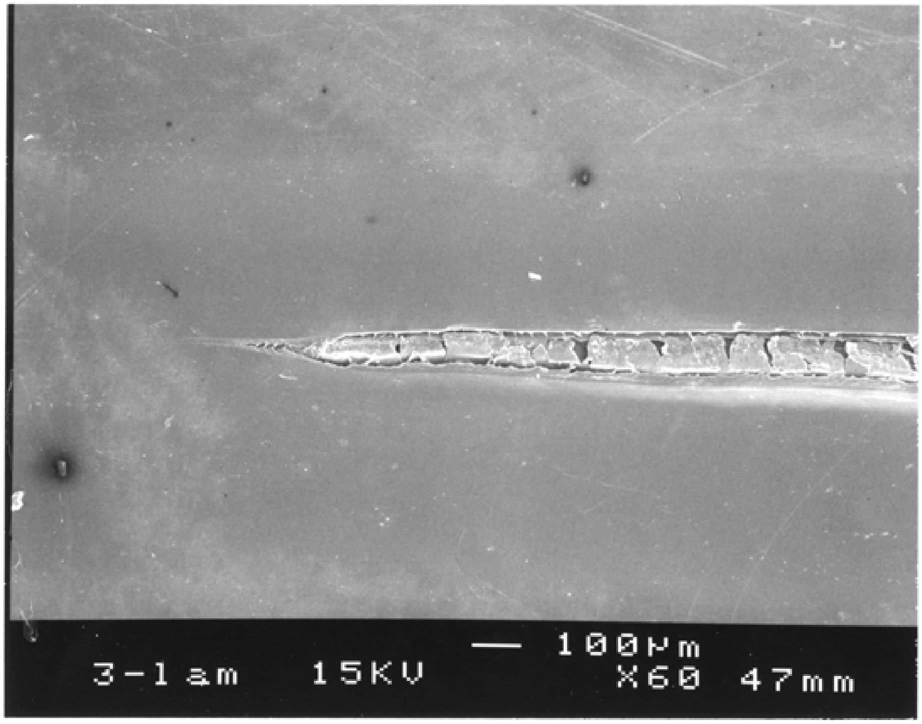


Figure 2: Macroscopic images of critical loads for failure.



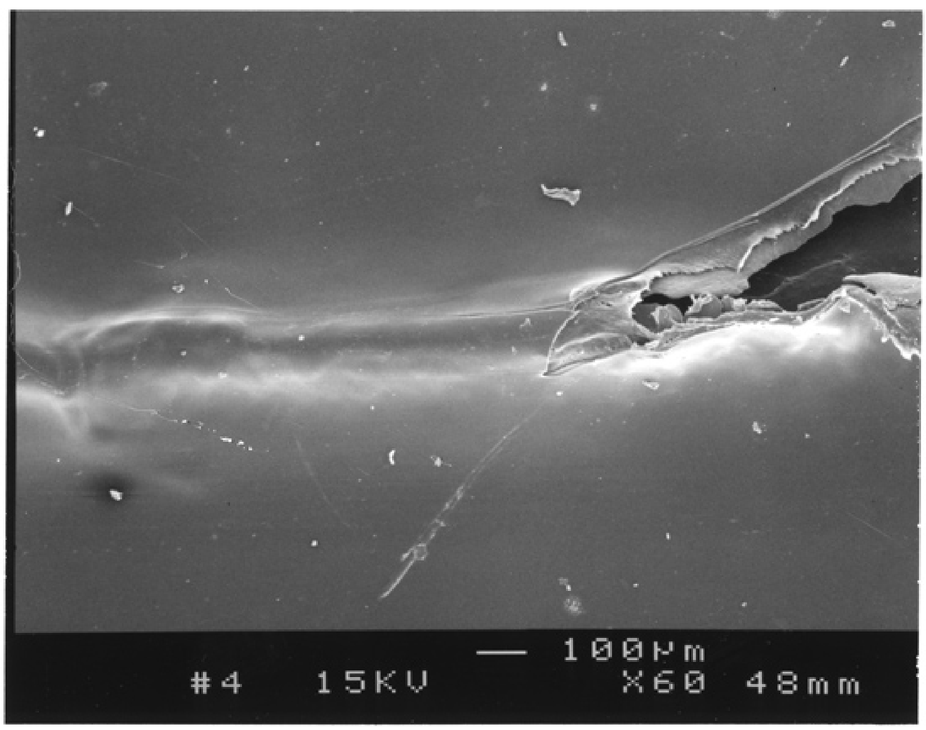


Figure 3: SEM images of Delamination and Complete Tear.

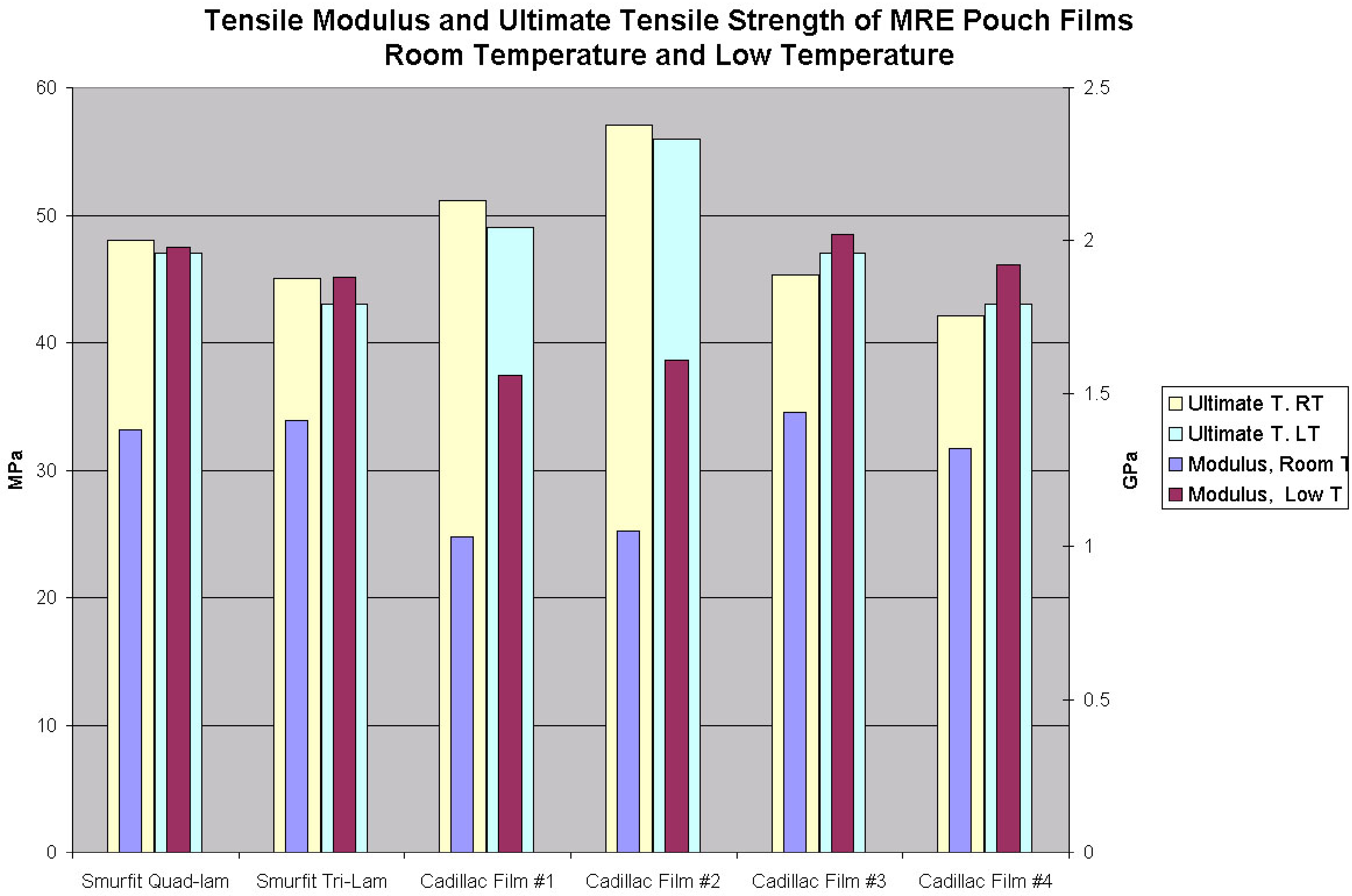


Figure 4: Comparison of Film Tensile Properties at low and room temperatures.

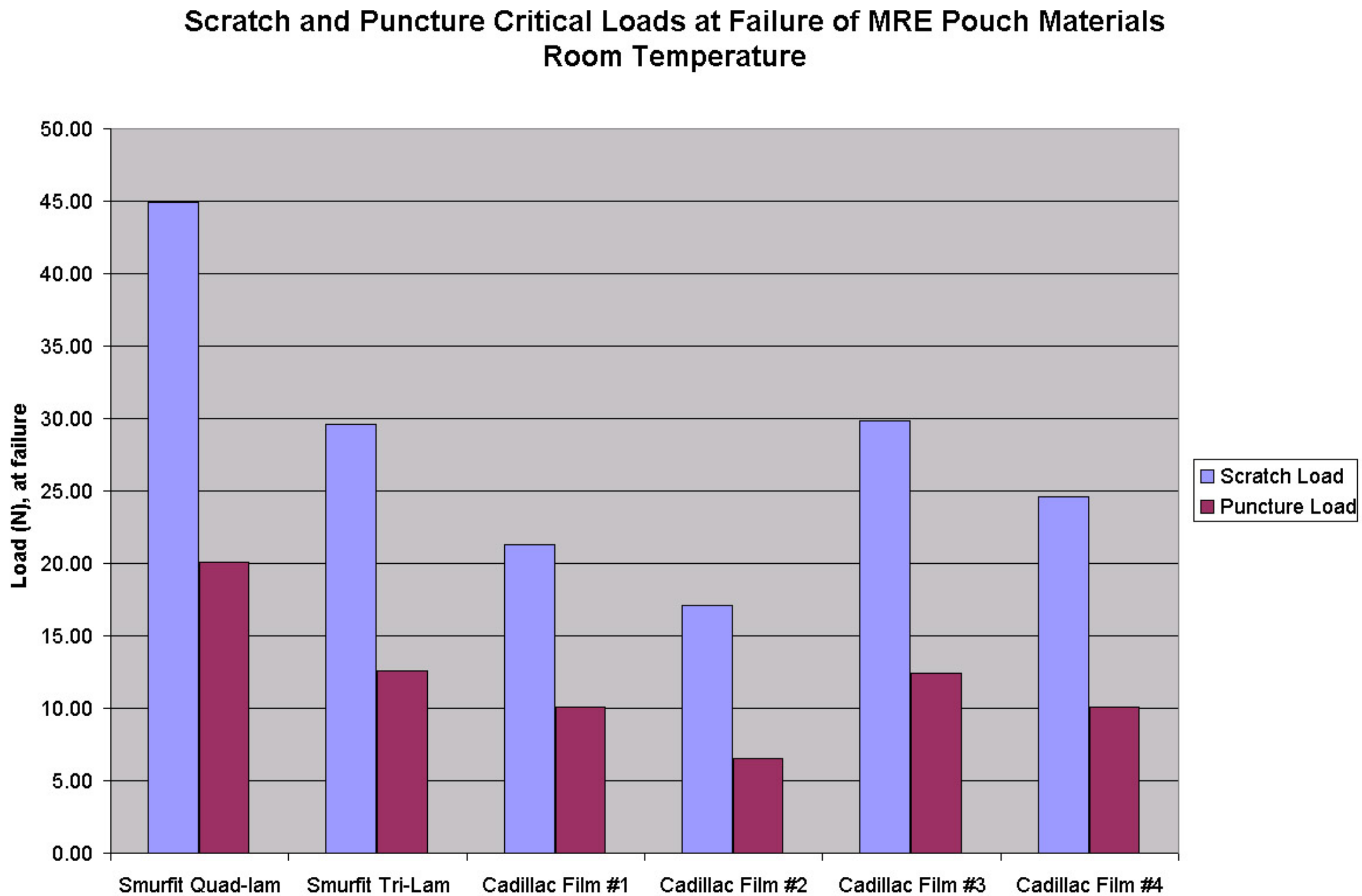


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Figure 5a: Scratch and Puncture Critical Loads at Failure for MRE pouch materials at room temperature.

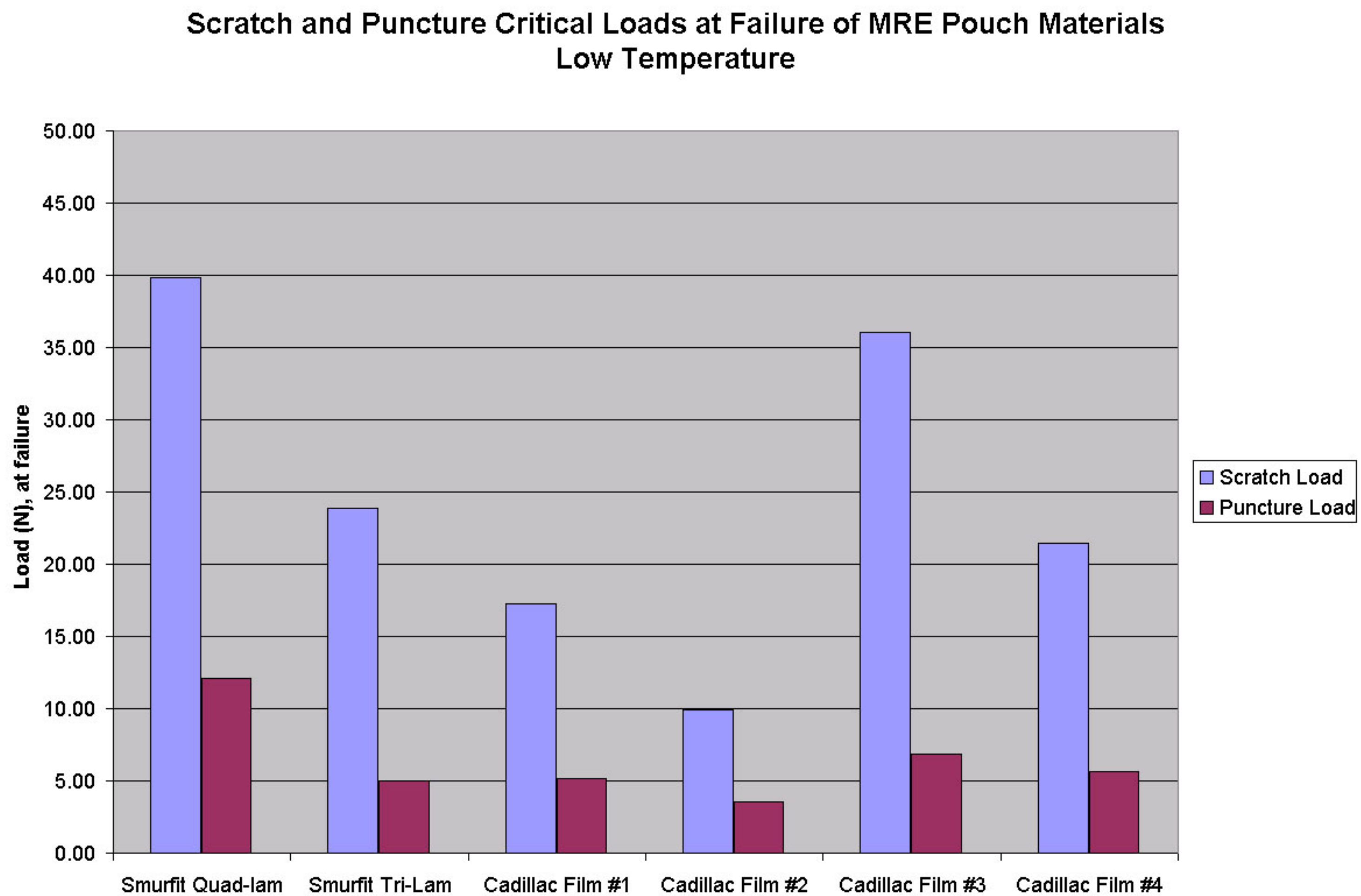

Figure 5b: Scratch and Puncture Critical Loads at Failure for MRE pouch materials at low temperature.

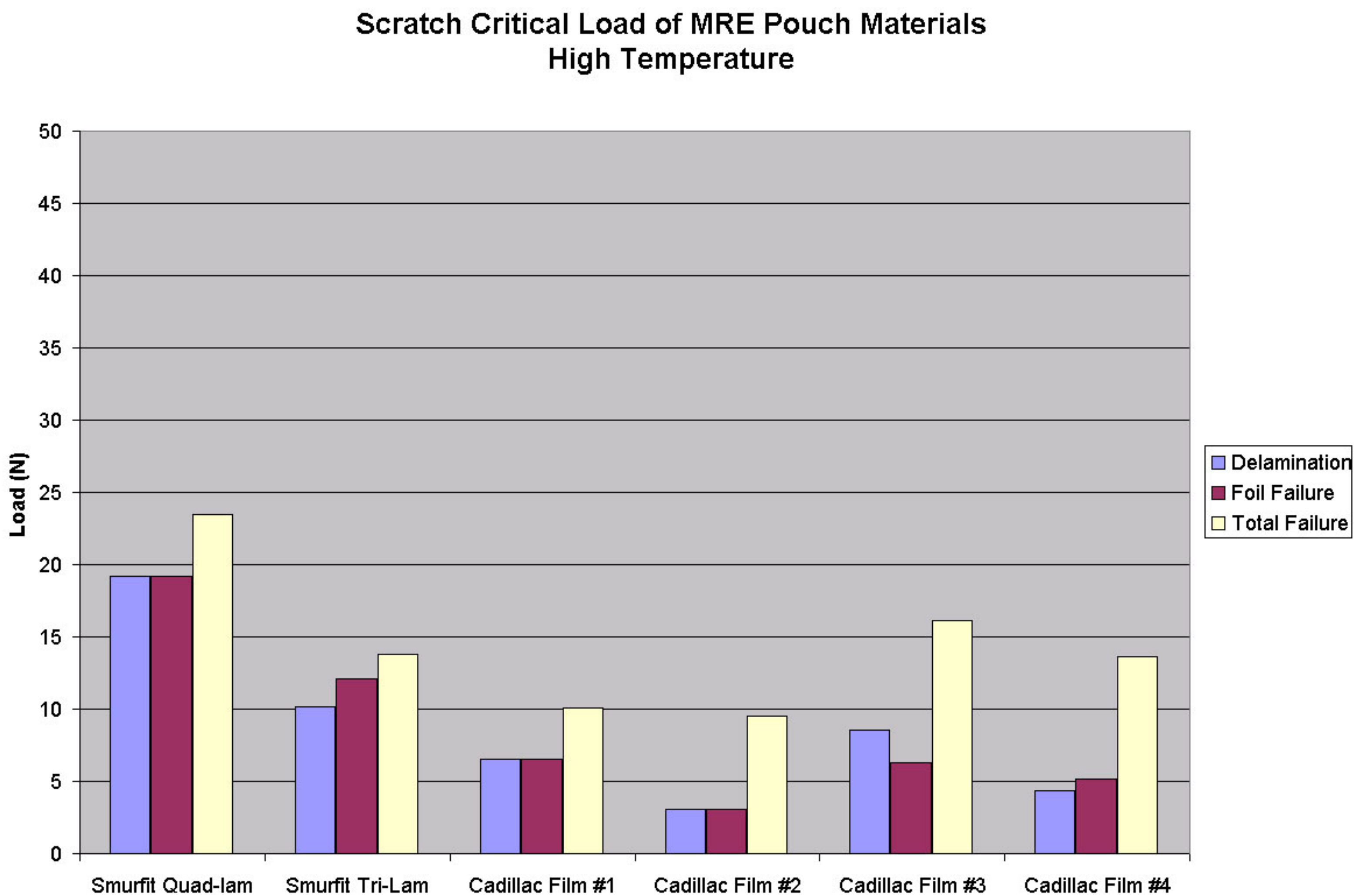

Figure 6a: Scratch Critical Loads at high temperature for MRE pouch materials.

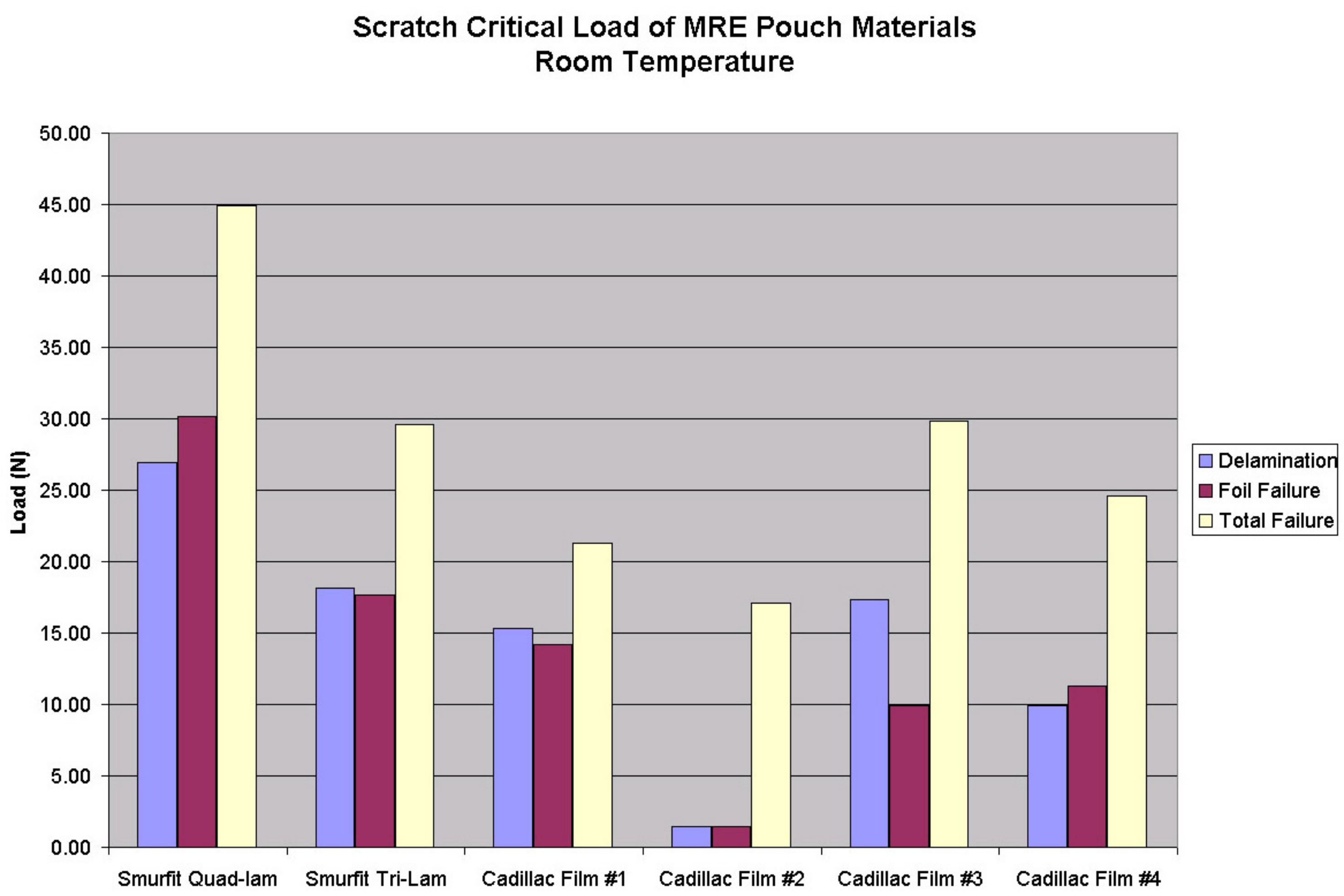

Figure 6b: Scratch Critical Loads at room temperature for MRE pouch materials.

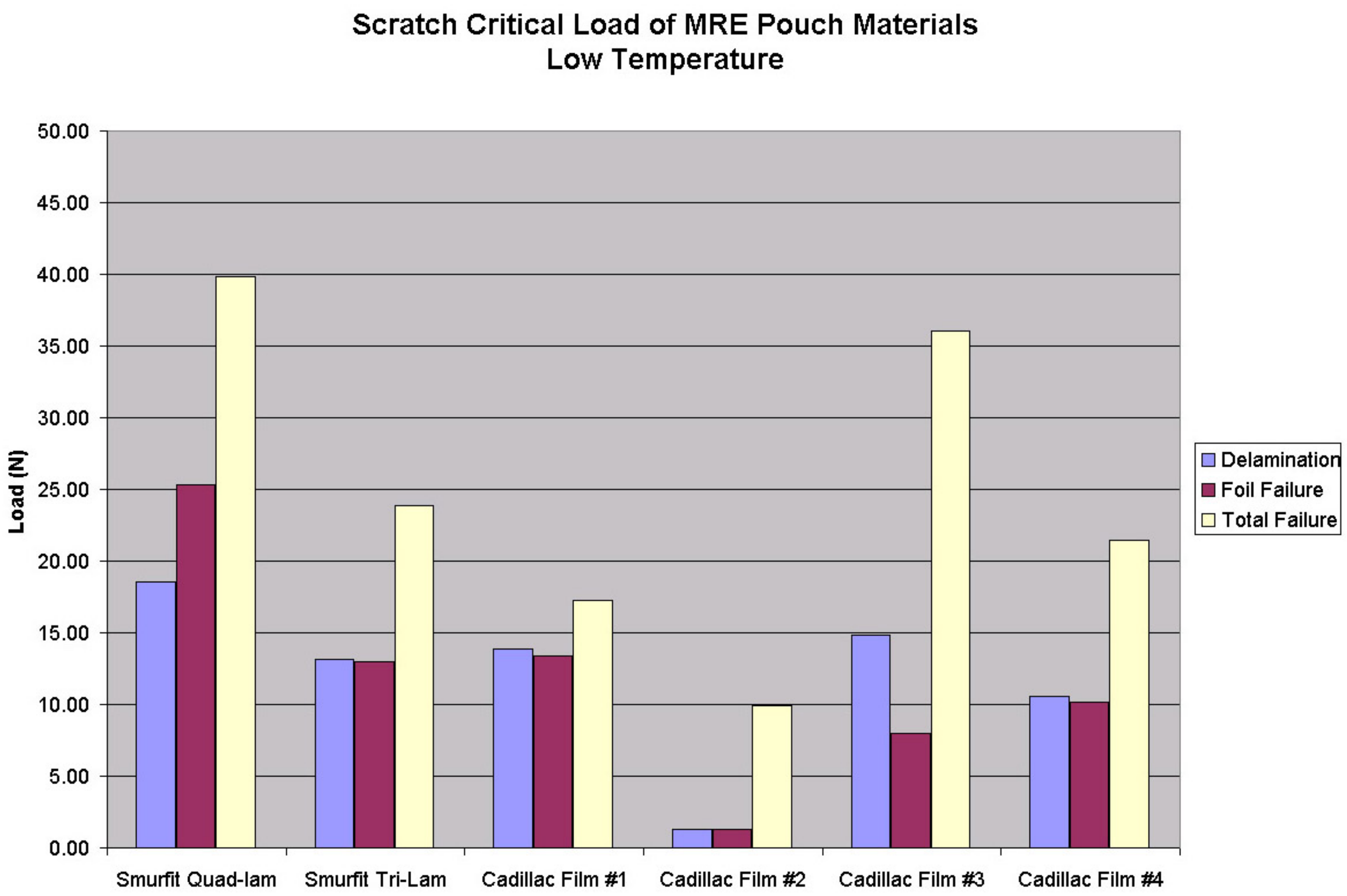

Figure 6c: Scratch Critical Loads at low temperature for MRE pouch materials.

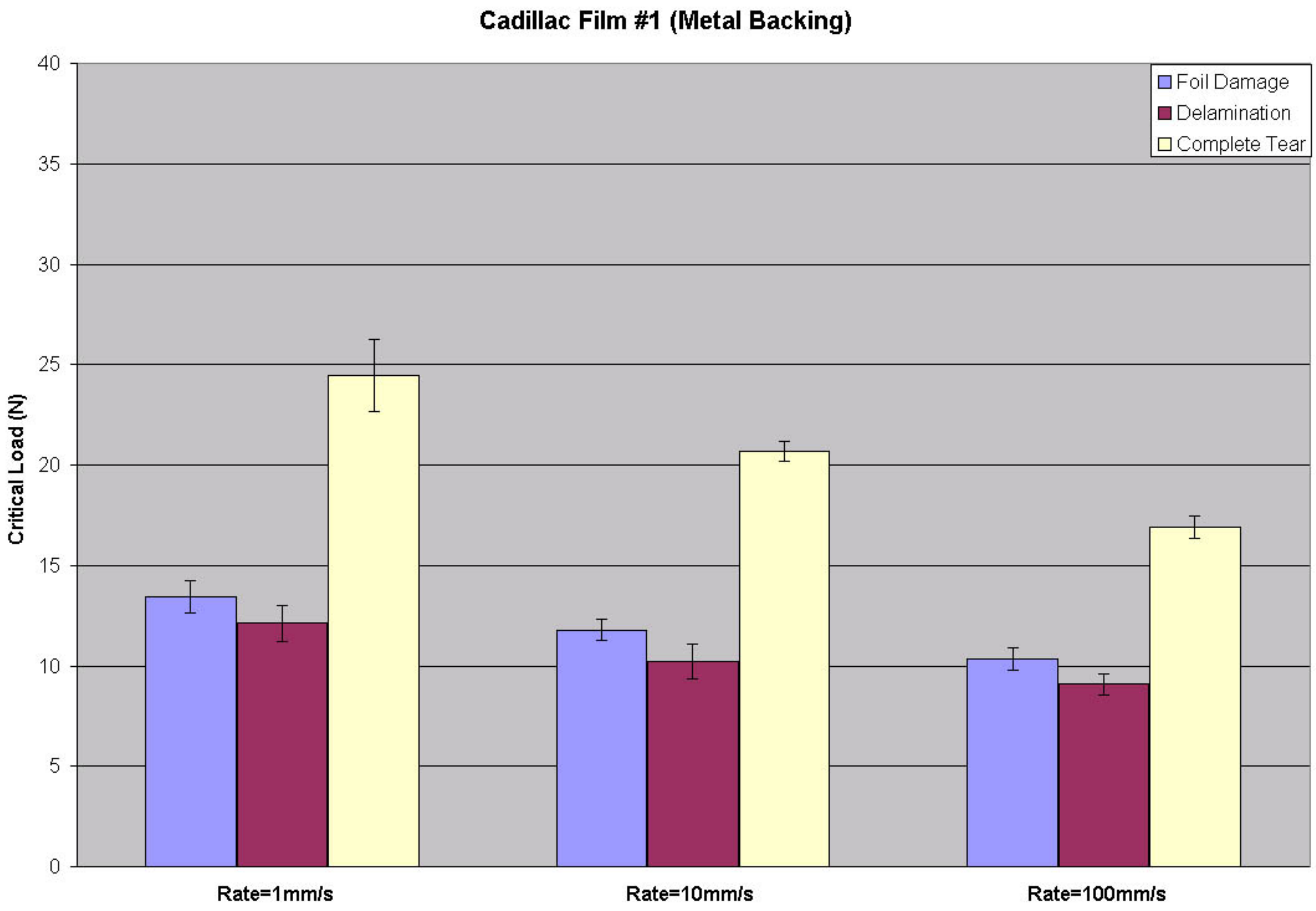

Figure 7: Testing Rate affect on Scratch Critical Loads for Film #1.

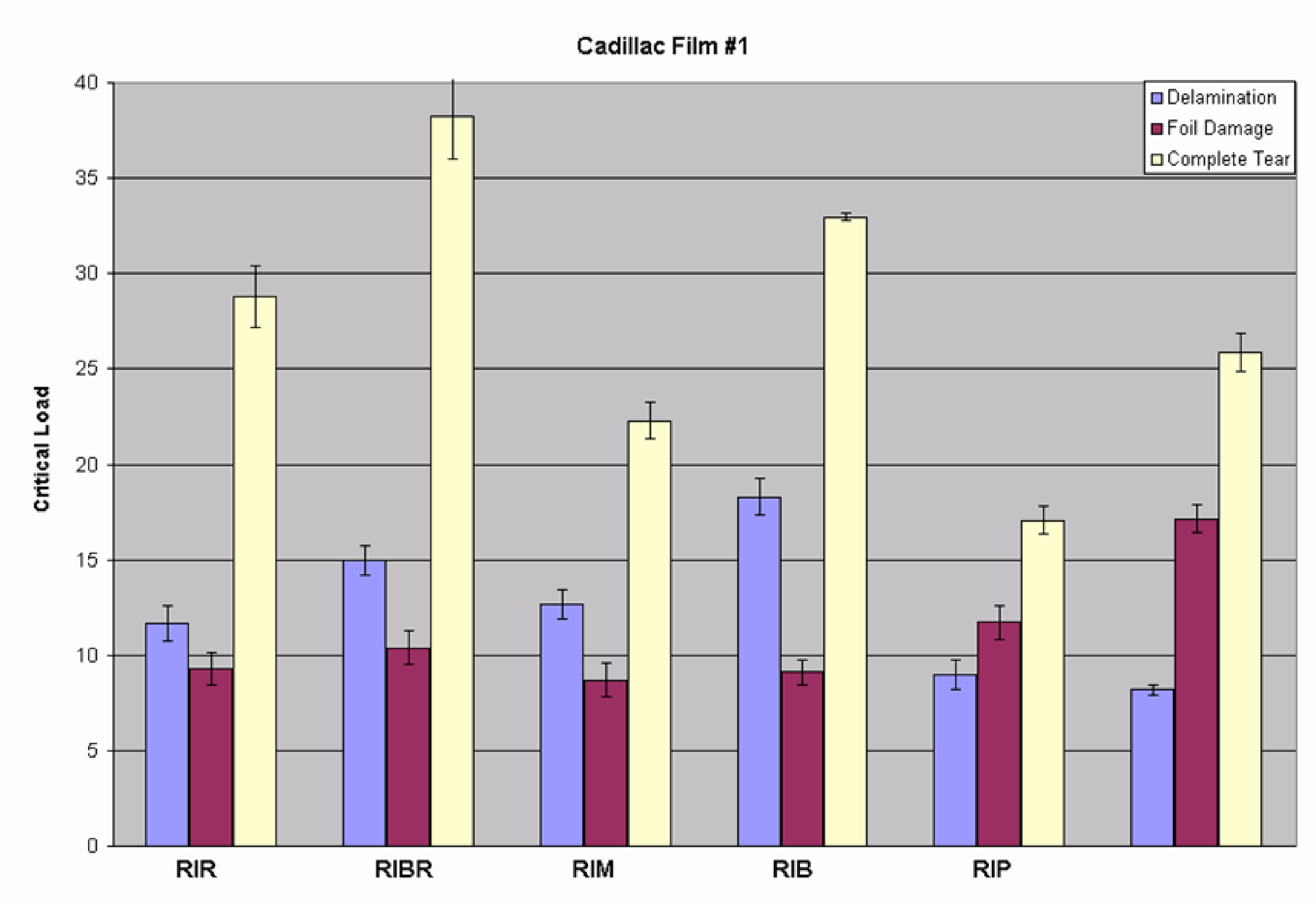

Figure 8a: Scratch Critical Loads with respect to backing and orientation.

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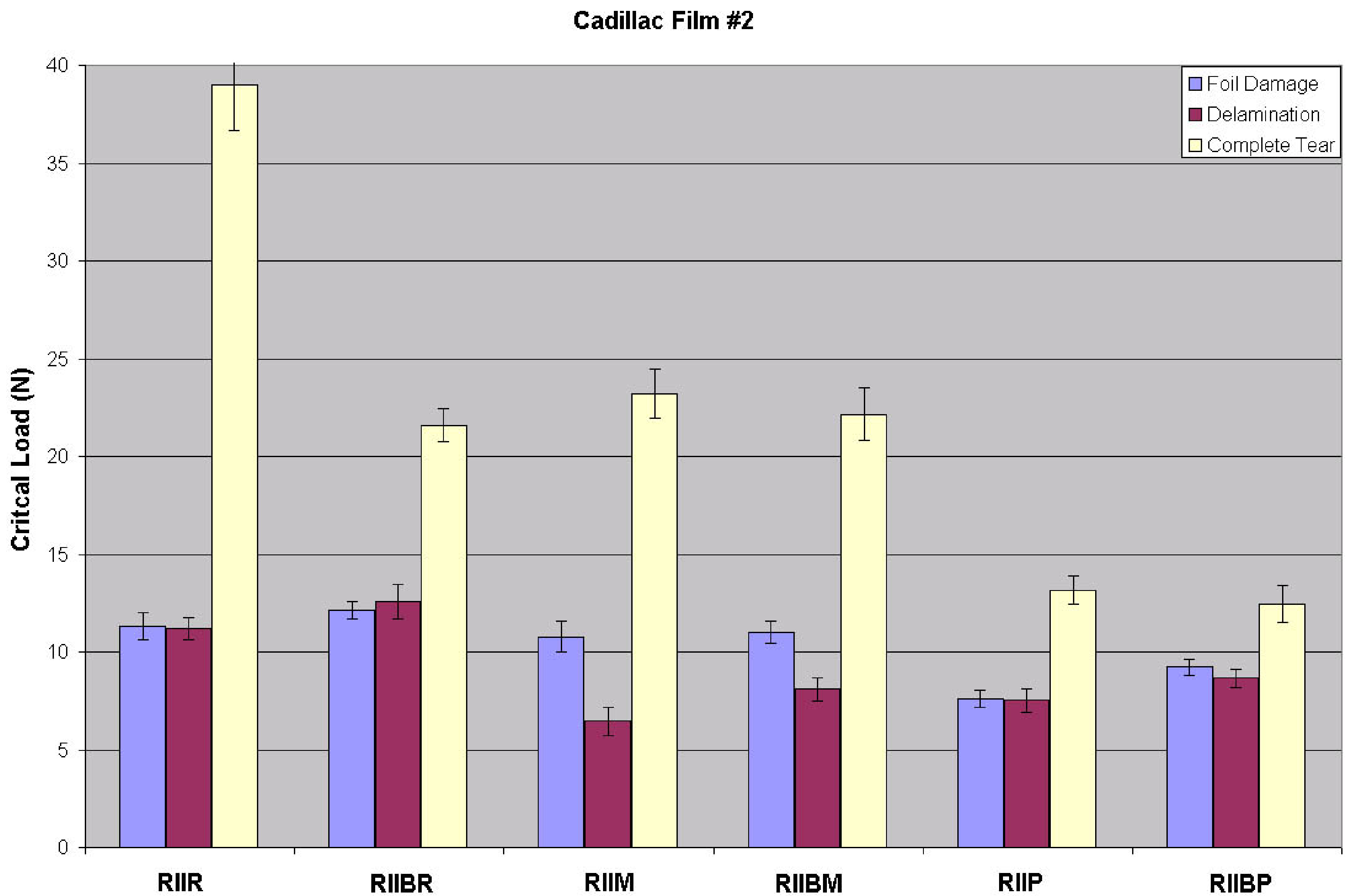

Figure 8b: Scratch Critical Loads with respect to backing and orientation.

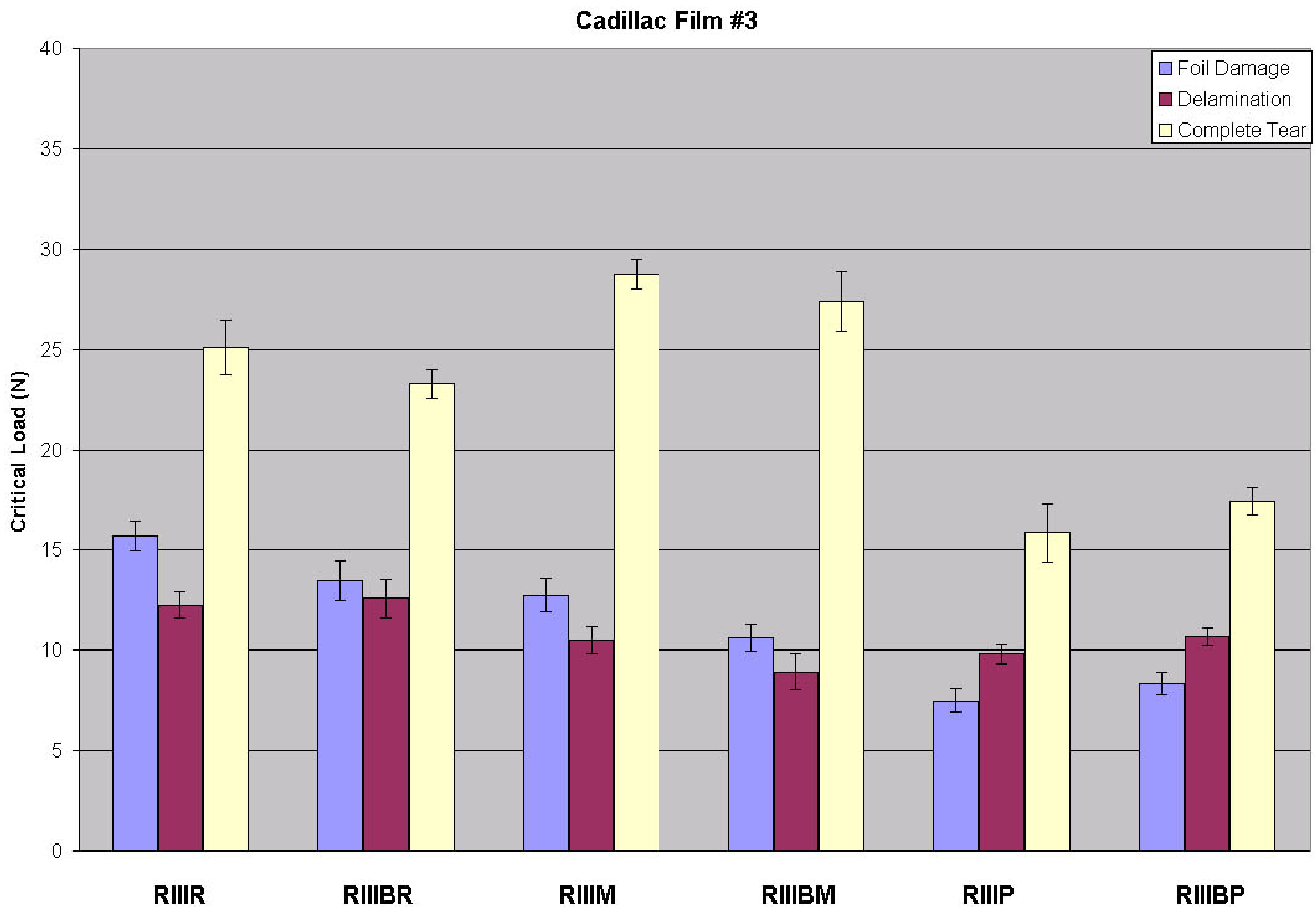

Figure 8c: Scratch Critical Loads with respect to backing and orientation.

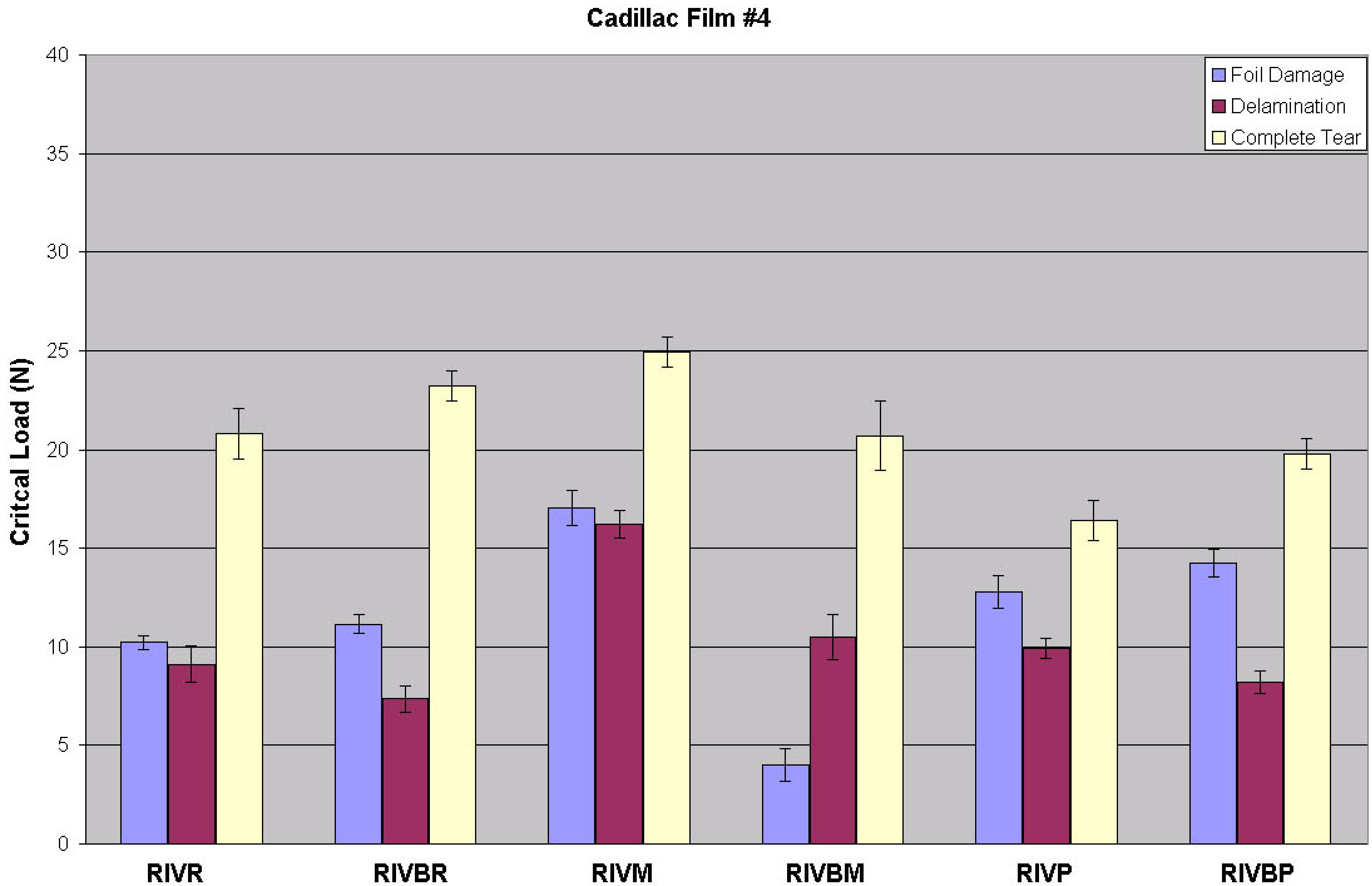

Figure 8d: Scratch Critical Loads with respect to backing and orientation.