Influence of pre-deformation
on the adhesion of PET on steel

Mirre Janssen
Internship, Eindhoven University of Technology, MT04.22

Ir. M.J. van den Bosch
Prof. Dr. Ir. M.G.D. Geers
Contents

1 Introduction 2

2 Methodology 3
  2.1 The material 3
  2.2 Pre-deformation 4
    2.2.1 Experimental setup 4
    2.2.2 Specimen geometry and preparation 4
    2.2.3 Experimental plan 5
  2.3 Roughness 7
    2.3.1 Experimental setup 7
    2.3.2 Specimen geometry and preparation 8
    2.3.3 Experimental plan 9
  2.4 Adhesion 9
    2.4.1 Specimen geometry and preparation 9
    2.4.2 Experiments 10

3 Results 12
  3.1 Relation between pre-deformation and surface roughness 12
  3.2 Relation between pre-deformation and adhesion 14
  3.3 Relation between surface roughness and adhesion 15

4 Discussion 16

A Manual of the LSM510 17

B Height images and RMS values 18
Chapter 1

Introduction

Worldwide about a billion cans are produced every day. Because of the quantity of cans that are produced, any adaption in the production process can save a lot of money. Therefore, research is done to make the production process cheaper. A possible way of making the production cheaper, is to apply a PET layer on the steel sheets before a deformation process. It protects the steel from corrosion and the paint is sprayed on top of it. One of the problems that occurs during deformation of thin metal sheets with a PET layer, is that the PET layer delaminates from the steel. With increasing deformation, the PET coating delaminates easier. The reason is that the surface roughness of the steel increases with increasing deformation. A rougher surface means that the PET is less attached to the steel and thus delaminates easier.

The goal of this report is to define a relationship between surface roughness of steel and adhesion strength of PET.

The relation between the surface roughness and adhesion strength can not be determined directly. It is possible to determine the relation between the pre-deformation and the surface roughness and the relation between the pre-deformation and the adhesion strength.

The pre-deformation of the specimens is induced by uni-axial tension parallel to the rolling direction of the steel. The roughness measurements methods that can be used are limited to optical methods because of the presence of the PET layer. After a conversation with Dr.Ir. Willem-Pier Vellinga it is decided to perform the surface roughness measurements with a laser scanning confocal microscope. The adhesion strength is measured with uni-axial tensile peel-off tests.

In the next chapter the material, experimental setups, specimen preparation and experimental plans are discussed. After that, the experimental results are given and explained. Finally, there is a discussion.
Chapter 2

Methodology

2.1 The material

The material is made and provided by Corus. The name of the material is Protact P585. It consists of a steel substrate, coated on two sides with a PET layer by direct extrusion. The thickness of the steel substrate and the PET layer is respectively 240 µm and 30 µm. To increase the adhesion of the PET there is an chromium layer which is partially oxidated between the steel substrate and the PET. The structure of the material is schematically shown in figure 2.1. The materials yield point is between 400 and 450 MPa and the material is suitable for large deformations due to its ductility. In an uni-axial tensile test it behaves almost ideal plastic.

Figure 2.1: schematic build-up of the material
2.2 Pre-deformation

2.2.1 Experimental setup

The pre-deformation is induced by uni-axial stretching in the rolling direction. The specimens are deformed with a Zwick 100kN tensile stage. The specimens are deformed until a defined extension. There is an extension-meter that measures the exact elongation of the specimen in a specified domain. The maximum extension can be pre-defined, stopping the tensile test when it has been reached. The extensionmeter is set to an initial length of 40mm. This length is chosen because of the geometry of the specimens. The test speed that is used is 10mm per minute. To clamp the specimen, the clamps with the smallest teeth are used, this to avoid fracture in or near the clamps. Sandpaper is put between the specimen and the clamps to protect the specimen for the clamp teeth and prevent slipping.

2.2.2 Specimen geometry and preparation

The specimens that are used are cut out of thin sheets of Protact 585. To protect the metal from scratching a protective layer is applied to the sheets on one side. First the sheets are cut into rectangles with a dimension of 150×35mm. Approximately fifteen of these rectangles are then stacked and milled in the desired shape. To prevent the specimens from slipping out of the clamps during the milling, holes are drilled in the specimens where a bolt is put through. The geometry of the specimens is given in figure 2.2. The lines in the neck of the specimen in figure 2.2 represent the domain of the extensionmeter. The protective layer is removed from the specimen before inducing pre-deformation for reasons that are outlined in section 2.2.3. Ethanol makes it easier to remove the protective layer from the steel.

![Figure 2.2: geometry of the specimens](image-url)
2.2.3 Experimental plan

It would be convenient to induce the pre-deformation in the specimens while the protective layer is still on the surface of the PET. To do this, it must be certain that the protection layer has no influence on the experiments.

To examine whether the protective layer has any influence, six specimens are deformed. Three of them are deformed with the protective layer on the surface (specimens A) and the other three are deformed without the protective layer (specimens B). All the other conditions were the same. Any differences that occurred during and after the deformation are due to the protective layer.

The first difference between specimens A and B was noticeable during the deformation on the 100kN tensile stage. Two of the three specimens A fractured before reaching the expected strain. Specimens A showed brittle fracture. Specimens B had a more ductile fracture and reached higher strain levels. Figure 2.3 shows the stress-strain curves of the samples.

![Figure 2.3: The stress-strain curves of the samples](image)

Another difference is noticeable when a specimen A and a specimen B are placed under an optical light microscope. Figures 2.4 and 2.5 show images of the specimens under the microscope. When the polarisation mode is used the shear bands are visible in specimen B, while there are no shear bands visible in specimen A. The cracks in the PET layer of specimen A were smaller than the cracks in the PET layer of specimen B. This can be due to the difference in strain.

The protective layer is removed before inducing the pre-deformation, because it is concluded that it influences the material behavior and its damage mechanisms. However, how much and exactly why has not been studied. More research is needed to quantify the influence. After the pre-deformation the protective layer is applied again.
Because the investigated quantities (surface roughness and adhesion strength) are related to the deformation, it is important that the deformation is homogeneous. For example, the local surface roughness of the specimen depends on the local plastic strain. Discussions with Corus revealed that it is difficult or even impossible to obtain a homogeneous strain field. Reasons for this are the small thickness of the specimens, the low amount of hardening and possible manufacturing inhomogenities. The deformation field is visualized to investigate whether the deformation can be considered homogeneous.

To visualize the deformation field a uni-axial tensile test is performed on a specimen with a grid on it. The size of the grid is 0.5 mm by 0.5 mm. The bar is extended until a extension of 20 percent is reached. After that the bar is examined under an optical microscope and the deformation appeared to be homogeneous to a large extent. The specimen has also been photographed with a digital camera. This picture is shown in figure 2.6. The grid is clear on the photograph but quantitative data can not be recovered from it. The only conclusion that can be made is that the deformation is optically homogeneous. Therefore, it is assumed that the deformation field in the neck of the specimen is homogeneous.

The percentages of elongation that are chosen are 0.0, 5.0, 7.5, 10.0 in pre-
defined extension is this respectively 0, 2, 3 and 4mm. These percentages are chosen because of the fact that a lot of bars fractured above 10 percent elongation and the bars where limited available. For the measurements of the surface roughness there is chosen to deform three specimens of each pre-deformation. For the measurements of the adhesion of the PET on the surface of the steel there is also chosen to deform three specimen of each pre-deformation. The same pre-deformations were chosen as for the measurements of the surface roughness. After the pre-deformation of the specimens a couple of specimens were left. From the specimen that were left two of them are pre-deformed until an elongation of 12.5% and one of them is pre-deformed until an elongation of 15%. These are used in the tests to measure the adhesion of PET on the surface.

2.3 Roughness

2.3.1 Experimental setup

The methods to measure the surface roughness of the steel are restricted by the PET layer. A possibility could be to remove the PET-layer from the steel. This can be done by peeling of the PET layer from the steel. A disadvantage of this technique is that pieces of the PET layer remain on the surface of the steel and influence the roughness measurements. Another option is to remove the PET layer chemically. However the department in Eindhoven has no experience in removing PET layers from steel chemically and it seems to be too cumbersome. A measurement method that measures trough the PET layer the surface of the steel is needed, so optical measurement methods remain. After a conversation with Dr.Ir. Willem-Pier Vellinga it is chosen to perform the surface roughness measurements with a confocal microscope. A disadvantage of this method is that it is time demanding, each surface roughness measurement takes approximately half an hour. The working principle of this roughness measurement method is briefly explained here.

There are two differences between a confocal microscope and an ordinary light microscope. The confocal microscope images a smaller part of the sample in the z-direction due to a pinhole, parts above and below the focus plane are rejected by the pinhole. The part of the sample that is imaged is perfectly in focus. Also lights the confocal microscope a smaller part of the sample, which leads to a better lateral resolution. In figure 2.7 is the working principle of the confocal microscope schematically described. A 3D image can be made by scanning an area in a series of planes, generating a stack of 2D slices. The data from the 2D slices is then analyzed by a Matlab program. This program compares the light intensities of each data point for all slices. The light intensity is the highest when the point is in focus. This is shown in figure 2.8 were the light intensity is plotted against the focal height. With
this information the program can calculate the height of each data point and produce a height image. This method is at 0.1 \( \mu \text{m} \) accurate. With the height of each data point known the RMS value of the roughness can be determined. The RMS value is calculated with the following equations,

\[
RMS = \sqrt{\frac{1}{n^2} \sum_{x=1}^{n} \sum_{y=1}^{n} (A(x, y) - \bar{h})^2} \tag{2.1}
\]

\[
\bar{h} = \frac{1}{n^2} \sum_{x=1}^{n} \sum_{y=1}^{n} A(x, y) \tag{2.2}
\]

where \( A(x, y) \) is the height of the data point, \( \bar{h} \) is the mean surface height and \( n \) is the number of data points in the \( x \) and \( y \) direction.

The confocal microscope that is used to measure the surface roughness is a Zeiss LSM510 confocal microscope. A manual with all the parameter adjustments that were done on the microscope to measure the surface roughness can be found in appendix A.

### 2.3.2 Specimen geometry and preparation

Normally the PET layer is transparant, so the light of the confocal microscope can go through it. However, when the specimens are deformed, cracks appear in the PET layer which scatter the light from the microscope. On these locations no data is detained from the specimen and they remain black. This leads to inaccurate roughness measurements. To prevent this, the
specimens are given a heat treatment after the pre-deformation. This heat treatment consists of heating the specimens on a plate which has a temperature of 200°C. The glass transition temperature of PET is 70°C. This means that the PET is then in rubbery state. After approximately 30 seconds the specimens are immediately cooled down with water. The cracks and shearbands in the PET layer have disappeared. The pre-deformed specimens are too large to be placed in a sample holder of the confocal microscope. Besides that only the deformation in the neck is assumed to be homogeneous. Therefore the ends are cut of and a rectangle piece of 40 × 25mm remains. This specimen is divided into 4 parts by drawing lines. The geometry of the specimen that is used to measure the surface roughness of the steel is shown in figure 2.9

![Figure 2.9: roughness measurement specimen](image)

### 2.3.3 Experimental plan

The roughness measurement specimens are divided into four sections. The surface roughness is measured ones in the middle of each section. From each pre-deformation are three roughness measurement specimens available. This means that for each pre-deformation twelve corresponding surface roughness measurements data point are obtained. All the roughness measurements have a name that consists of four numbers. The first number stands for the batch in which the specimen is milled out the steel, the second number represents the sheet of which the specimen is cut out, the third number stands for the place on the sheet where the specimen is cut out and the last number gives the location of the roughness measurement on the specimen.

### 2.4 Adhesion

#### 2.4.1 Specimen geometry and preparation

Out of each pre-deformed tensile bar two adhesion test specimens are made. These are cut out of the neck of the pre-deformed tensile bar in a manner that is shown in figure 2.10. The dimensions of the specimens are 10×40mm. After the specimens have been cut out a groove is milled into them. This groove controls the location of fracture of the steel substrate. The depth of the groove is about 120 µm. If the groove is deeper more heat is generated
during the milling, which influences the PET layer. If the groove is less deep
the fracture of the steel substrate is not controlled. The groove in the spec-
imen is shown in figure 2.11. Subsequently, the specimens are polished in
groups of four. This is done with four different polishpapers with respec-
tively 320, 800, 1200 and 2400 particles per \( cm^2 \). The polishing removes
imperfections in the edge of the PET layer that were caused by the cutting
of the specimen. If this was not done the imperfections in the edge of the
PET-layer could initialize fracture of the PET layer when a tensile force is
applied to the PET layer. The protective layer is then carefully removed
from the specimen. Finally the steel substrate of the specimen is broken
in an uni-axial tensile test. This is done on a Deben micro tensile stage
equipped with a 2150N loadcell. The test speed that is used is 0.2mm per
minute. Directly after the fracture of the steel substrate the tensile test is
stopped. The specimens are placed in a hand made holder from foam. The
specimens are now delicate and should be handled with care, otherwise the
PET layer will fracture.

2.4.2 Experiments

The adhesion strength of the PET on the steel is measured by an uni-axial
tensile peel-off test. In this test the steel substrate is broken while the PET
layer is still intact. During the test, the PET layer stretches and delaminates
from the steel. The force is measured. From a certain point this force remains
constant. This constant force represents the adhesion strength of the PET
layer on the surface of the steel. A picture of the experimental setup is shown
in figure 2.12.

The tensile stage that is used is a Kammrath&Weiss micro tensile stage
provided with a 20N loadcell. It is important that the speed at which the
clamps are moving from each other in the uni-axial tensile test is the same
at every test, this because of the fact that the adhesion strength is related
to this speed. The test speed that is used is 20\( \mu m \) per second. The adhesion
strength tests can now be performed on all the specimens.
Figure 2.12: experimental setup
Chapter 3

Results

3.1 Relation between pre-deformation and surface roughness

The height images and the results of the surface roughness measurements are given in appendix B. On some of the height images a noise is visible. This is probably due to the PET layer. It could be that the PET layer was heated to long, at a too high temperature and cooled down too slow. So, the structure of the PET has changed. Only the measurements with a high quality height image were taken into account. These images are indicated by "good" in table B.1. In figure 3.1 are the surface roughness values of the good measurements plotted against the elongation of the specimens.

![Figure 3.1: results of roughness measurements](image)

The variation of the data points is large. Especially the data points from the specimens with 7.5 percent elongation are widely spread. It could be that the roughness measurement method that was used was not suitable for PET-coated steel. Another explanation is that the samples were not as homogenous deformed as was assumed. Despite the spread of the data points
the roughness increases with increasing deformation, as is expected. This can be seen in figure 3.2 where the means and the standard deviations of the roughnesses are plotted against the elongations. A second degree polynomial is fitted to the data points. This curve cannot be extrapolated because the surface roughness cannot continue to increase with increasing pre-deformation, because there is a limit to the surface roughness. The maximum surface roughness will probably be located near the crack after fracturing the tensile specimen.
3.2 Relation between pre-deformation and adhesion

An example of a curve obtained in a peel-off test is shown in figure 3.3. The measured force is divided through the width of the peel-off sample to get comparable results. After approximately a minute the measured force remains constant, at least at a large scale. On a smaller scale level the force slightly decreases in time. Perhaps this is due to material properties but to determine an exact cause research is needed. To obtain reliable results the mean adhesion strength in a domain between 350 and 450 seconds is calculated. The mean adhesion strengths and the standard deviations are plotted against the pre-deformation in figure 3.4. It is clear that the adhesion strength decreases with increasing pre-deformation. There are not a lot of data points, because during the peel-off tests, the PET layer of many specimens fractured before delamination started. The exact reason is unknown.

Figure 3.3: peel-off curve
Figure 3.4: adhesion strength as a function of pre-deformation
3.3 Relation between surface roughness and adhesion

The relation between the pre-deformation and the surface roughness and the relation between the pre-deformation and the adhesion strength are known. The relation between the surface roughness and the adhesion strength can be determined and is shown in figure 3.5.

![Figure 3.5: relation between surface roughness and adhesion strength](image)

The reliability of this relation can be questioned, because there are many uncertainties in the previous results. It should be treated as an rough estimation of the relation between the surface roughness and the adhesion strength.
Chapter 4

Discussion

As expected, the adhesion strength of PET decreases with increasing surface roughness of the steel substrate. The scattering in the surface roughness data and in the adhesion test data is large. This is because a homogeneous deformation field is difficult or even impossible to obtain. For a better result more tests need to be done. A better result can also be obtained if the relation between the surface roughness and the adhesion strength can be determined directly.

The confocal microscope surface roughness measurement method is perhaps not the most suitable method for PET coated steel. A lot of the height images were of inferior quality and were not taken into account. Other methods should be investigated.

Many of the peel-off specimens fractured during the peel-off tests before delamination started. In future this can be taken into account and more peel-off specimens can be made.

Related topics that need more research are the exact influence of the protective layer, the influence of the peel-off test speed on the adhesion strength of PET and the reason why the adhesion strength of PET decreases in time during a peel-off test.
Appendix A

Manual of the LSM510

Turn the confocal microscope and the PC on with the master on/off switch. When the PC is ready, double click the LSM510 pictogram. Click on scan new images and start expert mode. Make a new file by clicking on File → new.

Go to acquire → laser. Turn on the Argon laser, set the output on 50%. Next click on acquire → micro. Choose the 2.5× objective. Set the reflector turret on Fset 14 and the transmitted light on 50%. Go to acquire → configuration. Toggle on the first three lines in the excitation screen and set them on 50% transmission. Provide channel 1 with the LP 505 filter and provide the lowest channel with the HFT488/543 filter.

Turn on the 100W lamp and set the microscope in VIS mode. Place the specimen under the microscope and move it so that the edge of the specimen is in the field of the microscope. Focus the microscope on the edge of the specimen by turning the large wheel on the right side of the microscope. Then change the objective for the 20× objective. Focus again and find a good spot.

For scanning this spot set the microscope in LSM mode. Click on the scan icon. With the fast xy icon a live image of the specimen can be seen on the PC. Click on the z-stacking icon. Select the lower and upper plane between which the specimen is being scanned. Set the pinhole at 11. The gain is chosen with respect to the histogram. On the domain that the microscope is scanning the histogram should not contain too many points that have an intensity over 250. If this is the case the pictures are too bright and the gain should be smaller. If this is all set the scan is started by clicking the start button.

When finished, turn off the laser and wait for a ”click”, which will be heard ± five minutes after turning off the laser. Turn off the PC and turn off the master on/off switch.
Appendix B

Height images and RMS values

Undeformed specimens

Figure B.1: In reading direction: nr 0.3.2.1, nr 0.3.2.2, nr 0.3.2.3, nr 0.3.2.4, nr 0.3.2.5
Specimens with 5.0% elongation

Figure B.2: In reading direction: nr 1.1.2.1, nr 1.1.2.2, nr 1.1.2.3, nr 1.1.2.4, nr 1.2.7.1, nr 1.2.7.2, nr 1.2.7.3, nr 1.2.7.4, nr 1.3.5.1, nr 1.3.5.2, nr 1.3.5.3, nr 1.3.5.4
Specimens with 7.5% elongation

Figure B.3: In reading direction: nr 0.5.5.1, nr 0.5.5.2, nr 0.5.5.3, nr 0.5.5.4, nr 0.5.6.1, nr 0.5.6.2, nr 0.5.6.3, nr 0.5.6.4, nr 2.2.4.1, nr 2.2.4.2, nr 2.2.4.3, nr 2.2.4.4
Specimens with 10.0% elongation

Figure B.4: In reading direction: nr 1.1.7.1, nr 1.1.7.2, nr 1.1.7.3, nr 1.1.7.4, nr 1.3.7.1, nr 1.3.7.2, nr 1.3.7.3, nr 1.3.7.4, nr 1.4.5.1, nr 1.4.5.2, nr 1.4.5.3, nr 1.4.5.4
Table B.1: roughness measurements of the samples

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