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The Influence of the Incorporation

of

Flectron Metallized Layers

on the

Mechanical Properties of Composites

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ABSTRACT

For a composite it would be very advantageous to be conductive for an electrical current. A composite structure is then for example able to form a cage of Faraday, that protects the things being in it against electrical fields.

Thus fourteen composite plates have been made, all having a different special layer in the middle. This special layer consists of a substrate with, in some cases, an absorbed metal layer on it. This metal layer takes care of the conductivity of the composite.

In this research bending tests and roughness measurements have been performed on these materials. By doing this it has been tried to determine the shear strength of the middle layer and to find a possible relation between the roughness of the middle layer and it's shear strength. This has been done for humid (1% moisture absorption) and dry conditions. Besides this microscopic observations have been done in order to determine possible weak spots in the middle layers.

The results are that the shear strength of the middle layers is only a bit lower than the shear strength of the composite itselve and that the moisture absorption does not reduce the shear strength very much. A clear relation between the roughness and the shear strength of the middle layers could not be found. In the middle layers a weak spot seemed to be the border between the metal layer and the substrate.

Because about seven of the fourteen different materials did not break in the middle layer, the calculated shear strengths may differ from the real shear strengths. Therefore it is advisable to perform some more advanced tests than the bending test.

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CHAPTER 1: INTRODUCTION

Fibre composites combine a high strength and E-modulus with a low mass density. Consequently, the specific strength and E-modulus often are better than those of metals. Thus fibre composites are very suitable materials for the aircraft industry.

Nevertheless, these materials do have some disadvantages. One of them might be the fact that most composites cannot conduct electric current. This latter property can be very useful for certain applications. For example, a metal fuselage of an airplane or the metal carrossery of a car have the advantage to be able to form the so-called cage of Faraday, due to the electrical conductivity of the metal.

In order to get a composite, able to conduct an electric current, it has been tried to make a composite plate with a conductive layer in the middle. This layer consists of a non-conductive substrate on which a metal layer is absorbed. Such layers are being produced by Monsanto.

In this research, executed in order of Monsanto, a composite of carbon fibres and an epoxy resin is used. From this material a few unidirectional laminates have been made with in the middle the special (conductive) layer. The aim of this research is to analyze the shear strength of this layer by performing some bending tests. These tests have been done on both dry and wet specimen. Thus the influence of water absorption on the interlaminar shear strength can be determined. Linked up with the mechanical tests some microscopical research has been performed in order to investigate the course of the crack and to determine possible weak spots in the middle layer.

CHAPTER 2: TESTED MATERIALS

The specimen have been prepared in the following way. First, plies have been knitted from a roll with unidirectional carbon epoxy prepeg. These plies had a thickness of 0.125 mm and a width and length of 300 mm. The type of prepeg is a 913C Fibredux. It contains 40 weight percent resin (after curing) and is made of a high tensile strength carbon fibre (T300). For the mechanical properties of this composite, see table 1. Twelve of these plies have been put on each other, with the carbon fibres pointing in one direction (unidirectional). After this four different special layers have been placed, next to each other, on the twelfth ply (figure 1). And finally another twelve carbon epoxy plies have been put on these special layers, resulting in a laminate of twenty four plies with in the middle a special layer. In this manner four plates have been made. These plates and the different middle layers are shown in figure 3.

After laminating the plates, they have been put in an autoclave. The pursued cure cycle and the drafting in the autoclave are shown in figure 2.

The quality of the plates has been controlled by a C-scan. This apparatus sends ultrasone waves through the plates (fig 4). These waves are partly reflected by every interface material-air and by the glass plate at the bottom of the C-scan. These reflected waves result in a reflection peak. At each point of the plate the intensity of the reflected peak of the glass plate is measured. If there are no voids or delaminations present in the composite plate, the intensity of the reflected peak is at most. And if the intensity of the reflected peak is greater than a certain adjusted value, the corresponding point on the plate is painted black. On the other hand, if the intensity is lower then the point remains white. Thus the black points correspond with a good quality, and the white points with a poor quality. The number of black points will increase in case one lowers the adjusted intensity value.

The results of the C-scanning are printed in figure 5 and table 2. The adjusted

intensity value is seventy five. According to these results the plates containing a middle layer with a random structure seem to have more voids than the other plates. Later on, microscopic observations confirmed these conclusions. The middle layers that seem to have the largest number of voids, are the thick random aramide layers (one with copper (1.6 opsy) and one without copper (1 opsy)).

Charging the high adjusted intensity value (75), one may assume the quality of the plates to be rather good.

After C-scanning each plate has been cut into four smaller plates (13 × 13 cm). They are shown in figure 6. This resulted in sixteen plates. Fifteen of them having a different middle layer (figure 3). From these fifteen plates (named with the letters A-O), the specimen for the three- and four-point bending test have been cut.

In order to get specimen with zero % humidity, they have been put in a vacuum oven at a temperature of 60 °C until their weight remained constant. The specimen did not lose much weight. Thus the amount of water in the specimen had been rather low.

For a composite the absorption of water may have great influence on it's mechanical behavior. Therefore half the number of specimen have been put in a climate chamber for ± one month (37 days) at a temperature of 76 °C and a humidity of 70 %. After this the specimen were submitted to three-point bending tests.

If one wants to have the specimen fully saturated with water, they have to be put in a climate chamber for two or three months. This period is too long, as the time, available for this research, has been only three months. Our absorption data showed that after one month all specimen had absorbed already one percent of moisture. From the absorption rate after one month one could expect the specimen to be almost saturated with moisture. A saturation level of one percent seems to be a reasonable value [1], [2].

CHAPTER 3: DESCRIPTION OF THE EXPERIMENTS

The experiments that have been done are measurements of roughness values, measurements of the water absorption in the specimen and three- and four-point bending tests. After this some microscopic research has been performed. A general description of these tests follows below.

3.1. Measurement of the roughness

Before performing the bending tests, the roughness values of the used middle layers have been measured. The reason for this is the fact that the roughness of the middle layer may have great influence on the mechanical stitch of the middle layer to the carbon-epoxy laminate.

These measurements have been done by a Rodenstock RM 600 (figure 7). An infrared laser integrated into the sensor is focussed permanently on the surface to be measured by a movable objective. The position of the objective varies with the changes in height of the surface to be measured. Changes in the position of the objective are sensed independently of the focus control system by an inductive displacement transducer. From the signal of the transducer the roughness of the surface can be determined. The used roughness measure is Ra.

This Ra value represents the mean absolute deviation of the measured surface with respect to a certain zero level. This level is chosen in such a way that the mean absolute deviation is minimal. Thus:

$$Ra = \frac{1}{L} \int_0^L |y(x)| \, dx \qquad (1)$$

In which y is the height of the surface with respect to the zero level and L is the length over which the roughness is measured.

3.2. Three-point bending test.

The three-point bending test is a very widely used test method. If one chooses the right dimensions of the test specimen the apparent interlaminar shear strength can be determined. In this research the three-point bending test has been performed according to the SACMA SRM 8-88 standard. This norm has been derived from the ASTM METHOD D2344. Figure 9 shows the test formation and in figure 8 the used test specimen are shown.

For bending tests the ratio of the fixture span (L) to the thickness of the specimen (d) is very important. According to the SACMA standard L/d has to be four in our case (three-point bending). Then the material will break as a result of the interlaminar shear stress exceeding the interlaminar shear strength of the material (figure 10a). If the ratio L/d is chosen too high, the tensile and compressive stresses in the outermost fibres will be the limiting factor. As a consequence the material will break as shown in figure 10b.

The chosen cross-head speed is 0.5 mm/sec. And the tests have been performed on an Instron 1196.

In the specimen all the reïnforcing fibres are aligned parallel to the span length L and the principal axis of orthotropy coincides with the symmetry axis of the beam. The maximum shear stress (shear strength) occurs at the neutral surface and is obtained from elementary strength of material principles as:

$$\tau_{max} = 0.75 \frac{P}{bd} \qquad (2)$$

With:

P: maximum force (figure 9)

d: thickness of the specimen

b: width of the specimen

The use of this formula assumes the following things [3]:

1)   The material is homogeneous.

2)   Plane sections remain plane after deformation.

3)   The material exhibits a linear stress strain relationship.

The three-point bending test is not a difficult test and therefore it is widely used. Nevertheless there are some disadvantages that should be kept in mind, when performing the test:

1)   According to classical beam theory the distribution of the shear stress should be parabolically across the beam as indicated by the dashed curves in figure 11 (the so called Euler-Bernouilli solution which is used in norms as SACMA and ASTM and which results in formula 2 for the maximum shear stress, the shear strength). In contrast to this, finite element calculations show that the actual distribution of the shear stress may differ very much from the Euler-Bernouilli stress distribution [4] (figure 11). This can be explained by the fact that the Euler-Bernouilli solution does not take into account, the influence of the local stress distribution, due to contact forces, on the global stress distribution. Especially if the specimen are small these local stresses will play an important role.

2) Because of the contact forces the fibres under the loading nose might fracture by buckling [5] (figure 12). If the area in which this buckling takes place is rather large, it has an important influence on the shear stress distribution across the beam. Under these circumstances, formula 2 cannot be used for the calculation of the (interlaminar) shear strength. Fracture due to fibre buckling will appear less frequently if contact roles with a larger diameter are used. On the other hand a larger diameter results in a larger contact zone and therefore in a larger area of the specimen that is affected by the contact stresses.

Because of these disadvantages the three-point bending test is normally used as a screening test. It cannot be used as a test for design properties.

3.3. Four-point bending test

Besides three-point bending tests also some four-point bending tests have been performed (only on the dry specimen). The main reason for this is to check the shear strength values achieved by the three-point bending tests. In figures 13 and 14 the four-point bending test and the used specimen are shown.

In comparison to the three-point bending test the only disadvantage of the four-point bending test is not being an ASTM standard for it.

In a four-point bending test the transition from shear failure to tensile/compressive failure is shifted to a higher span to depth ratio (in figures 15 and 16 the theoretical curves and some experimental results of Rosensaft and Marom are shown [6]). The reason for this follows from figure 16. It shows that σ/τ is larger for a three-point bending test.

Besides this the shear strengths determined by the four-point bending test generally have a higher value than the ones calculated from the three-point bending test. This is accounted for by the fact that under four-point bending each loading roller transfers only half of the applied load. As explained in the previous section, the local stress field in the vicinity of the loading roller results in a rise of the actual maximum shear stress in the specimen above the nominal maximum shear stress calculated by formula 2. Consequently a reduction of the applied load (which corresponds to a reduction of the local stress field), results in a higher maximum nominal shear stress and in a smaller local buckling damage of the fibres under the loading rollers.

As a result of these considerations one may expect the shear values obtained by the four-point bending tests to be closer to the values that reflect the material properties.

3.4. Microscopic observations

After the bending tests some specimen (especially the ones with a crack in the middle layer) have been selected for analyzing the structure of the middle layer and the course of a possible crack.

Because of the cracks not being big enough to open them, the actual crack surfaces could not be looked at. In stead of this the lateral surfaces of the specimen (the surfaces in which the cracks are visible), have been analyzed by a light microscope (an MEF2 Reichert microscope).

CHAPTER 4: THE EXPERIMENTAL RESULTS

4.1. Roughness measurements

They are given in figure 17 and in table 3. For the middle layers with a woven structure, the roughness values have been measured in three directions (figure 18). Obviously, for the random structures the roughnesses have only been measured in one direction.

With respect to figure 17, the following remarks can be made:

1) For each kind of material (except aramide), the roughness value of the layer with a metal coating is lower than the value of the same layer without a metal coating (for instance: the Ra-values of b and c are smaller than the value of d).

2) For the woven structures the values measured in the 0° and the 90° directions, are rather the same (for one sort of material of course).

3) In most cases, the measured Ra-values in the 45° directions are higher than the values measured in the other two directions (0° and 90°).

4) The roughness values of the aramide structures are higher than those of the other materials. Especially the Ra values of the aramide layer with a copper coating.

4.2. Bending tests on the dry specimen.

4.2.1. Three-point bending tests.

These results are shown in figures 20 and 21 and in table 5. With respect to the figures a few things attract attention:

1)   From figure 20 one can see that the differences between the shear strengths are not very large. This also accounts for the calculated standard deviations in comparison to the shear strengths.

2)   Figure 21 shows the shear strength of specimen A (the specimen without middle layer) to be 100.6 MPa. The shear strength measured by the manufacturer is 92 MPa (table 1).

3)   For specimen with a random nylon structure in the middle layer, the calculated shear strengths are of the same order as the (interlaminar) shear strength of the specimen without middle layer (an exception must be made for the shear strength of the specimen with random nylon provided with a nickel layer).

4)   For specimen with a woven structure (polyester and nylon) in the middle, the shear strength has a higher value in case no metal layer has been absorbed.

5)   If a metal layer has been absorbed the specimen with a woven nylon structure in the middle layer have more or less similar shear strength values.

6)   The lowest shear strength values have been measured for the specimen with a middle layer consisting of woven polyester with an absorbed metal layer.

7)   The specimen with a thick aramide layer in the middle have higher shear strength values than the ones with a thin aramide layer.

4.2.2. Four-point bending tests.

These results are shown in figures 22, 23, 24 and in table 6. Again, a few things can be noticed:

1)   The specimen without a special middle layer (A) has the highest shear strength (100.88 MPa).

2)   The shear strength values do not differ very much. And the calculated standard deviations are small in comparison to the shear strengths.

3)   For the specimen with a random nylon structure in the middle (B,C,D) the shear strengths are almost as high as the shear strengths of the specimen without a special middle layer.

4)   Both the series with a woven middle layer (E,F,G and H,I,J,K) have a higher shear strength in case the substrate has not been metallized.

5)   From figure 24 one can see the shear strengths determined by the four-point bending tests to be of the same order of magnitude as those determined by the three-point bending tests. The values of the four-point bending tests are in almost all cases somewhat higher than the corresponding values of the three-point bending tests.

4.3. Moisture absorption by the specimen.

As mentioned before, half the number of specimen have been put in a climate chamber (70% humidity and T=76°C) for 37 days. The moisture absorption of the specimen without a special middle layer (the reference), during this period is shown in figure 19. The data of the specimen with a special middle layer were almost the same.

As one can see the absorption speed of the specimen diminishes in time (realizing that the x-axis represents √t). Through the data, a straight line can be drawn. Interpolation of this line shows that it does not cross the zero. At the end all the specimen contain plus minus 1 (weight) percent moisture (table 4).

4.4. Bending tests on humid specimen.

These specimen have been submitted only to three-point bending tests. The results of these tests are rendered in figures 25, 26, 27 ,28 and in table 7. The following things are important:

1)   All the shear strengths have been reduced as a result of the moisture absorption.

2)   The reductions of the shear strengths are not very high. The maximum reduction is 13%.

3)   The relative magnitude of the shear strengths within each material group (the materials having the same substrate in the middle layer), is plus minus the same for the dry and the humid specimen (figure 27). Only the shear strength of the specimen with nickel and polyester in the middle has reduced somewhat more than the shear strength of the other specimen with a

polyester layer in the middle. And within the group having a nylon substrate (B-D) the shear strength reduction of the specimen with nickel is much less than the shear strength reduction of the other specimen (figure 28).

4)   In comparison to the other groups of materials, the groups with an aramide and a polyester substrate in the middle show less reduction of the shear strength. (figure 28).

5)   The standard deviations of the humid specimen are somewhat lower than those of the dry specimen (figure 27).

6)   Remarks 1,4,5,6,7 made with respect to the dry specimen (section 4.3.1) also apply for the humid specimen.

4.5. Microscopic observations.

During this research both the tested and the untested specimen has been looked at. By doing so, one comes to know which cracks have arisen as a consequence of the bending tests and which cracks have arisen during the preparation of the specimen. Besides this, looking at the original specimen provides more information about the unharmed structure of the middle layer and about the influence of moisture absorption on the middle layer.

Below the results are presented per material cluster.

4.5.1.1. Dry specimen having a random nylon substrate in the middle layer.

Observations during the bending tests showed that almost all the specimen broke somewhere above and beneath the middle layer (figure 29a). Thus one could expect the middle layer to be rather unaffected.

A few things are important:

1)   The structures of random nylon with copper, without a metal layer and with nickel do not differ very much.

2)   The resin content of the middle layers is rather high, especially of the layer without metal.

3)   The middle layers of both the tested and the untested specimen contain a few voids.

4)   The specimen having a middle layer consisting of random nylon without a metal layer are hardly effected by the bending tests. Only a small space is visible between the resin and the nylon fibres. It is rather doubtful whether this is caused by the bending tests.

3.5.1.2. Humid specimen having a random nylon substrate in the middle layer

These specimen broke in the same way as the dry specimen (figure 29a). Therefore, during the microscopic research, hardly any differences with the dry specimen have been visible. A picture of such a humid specimen is shown in figure 30.

4.5.2.1. Dry specimen having a random aramide substrate in the middle layer.

Specimen with a thin aramide layer (materials L and N) in the middle often broke as shown in figures 29b and 29c. Especially after four-point bending tests, the middle-crack is clearly visible. In contrast to this the specimen having a thick aramide layer (materials M and O), hardly ever broke in the middle. They failed as shown in figure 29a. In figure 31 a picture of the middle layer is shown.

From the microscopic observations a few things became clear about the structure of the middle layer before and after testing:

1)   The aramide layers consist for a very great deal of epoxy resin.

2)   Before testing the structure of a thin layer is the same as the structure of the equivalent thick layer (the structure of L is the same as the structure of M and the structure of N is the same as the structure of O).

3)   Voids are visible in both the middle layers of the tested and the untested specimen.

4)   The layers consisting of aramide with a copper layer show that the copper is not lying very properly on the aramide fibres. Often holes or resin is present between the copper layer and the aramide fibres.

5)   In the middle layers without copper, the aramide fibres are surrounded rather well by the resin.

6)   In the thin layers the cracks are clearly visible. The fibres lying in the neighbourhood of these cracks have been deformed. The thin layer with copper shows cracks at the boundary between the middle layer and the carbon epoxy composite while the cracks in the thin layer without copper are such big that almost half the middle layer seems to have disappeared.

8)   In the thick middle layers no clear cracks are visible.

4.5.2.2. Humid specimen having a random aramide substrate in the middle layer.

Visual inspection after the bending tests learned that specimen with a thin middle layer often have a very small crack in this middle layer, just as shown in figure 28b and 28c. But these cracks did not occur as often as and were not as clear as the cracks visible in the dry specimen after the four-point bending tests. Generally none of the specimen with a thick middle layer broke in the middle layer (first ply failure). They broke in the carbon-epoxy matrix (figure 29a). Pictures made of these specimen are shown in figures 32, 33, 34.

A few things are remarkable:

1)   Possible damage as a consequence of moisture absorption is not visible.

2)   In the tested version having a thin aramide layer with copper, many voids are visible. Thus one can see a crack that consists of a connection of some voids. In this crack often the metal has been pulled of the fibre although at some places the metal fibre connection is still in tact.

3)   The crack in the tested specimen with a thin aramide layer without copper, is for a great deal coursing just through the resin.

4)   The specimen having a thick aramide layer without copper, has a crack at the boundary of the middle layer and the carbon-epoxy composite. One has to realize that this crack is not the first ply failure (the first crack occurred in the carbon-epoxy composite).

4.5.3.1. Dry specimen having a woven nylon substrate in the middle layer

During the bending tests most of the specimen with nylon and a metal layer in the middle, broke in this middle layer as shown in figure 29b. The specimen having nylon without a metal layer did not seem to have clearly visible cracks in the middle layer. A picture made of a specimen with a nylon substrate is shown in figure 35. Microscopic observations showed the following things:

1) The resin content in the woven middle layers is much lower than the resin content in the random middle layers (see previous sections).

2) The untested structures of woven nylon with copper and woven nylon with nickel are much the same. In both cases the metal layer is only present at the outside of the fibre bundles. Besides this at some places the fibres lying just above a fibre that is crossing perpendicular are somewhat deformed.

3) After testing, in the structures with a metal layer, the crack often goes through the fibre bundles and along the boundary area between the metal layer and the fibres lying under or above it. The fibres have been deformed and small cracks in the fibres are visible. Cracks can also be seen at the boundary of the middle layer and the carbon epoxy composite.

4) As expected (after visual inspection) the structure of the middle layer without a metal layer is rather unaffected by the bending test. In comparison to the same structures with a metal layer, the resin content is somewhat higher. Besides this, in the tested specimen a small crack is visible at the boundary of the middle layer and the composite material. For the untested structures, the only fibres that have been deformed are those which are crossed perpendicular by another fibre. Testing the specimen seems to enlarge these deformations. Finally, like the same structures with a metal layer, the structure without a metal layer contains a few holes.

4.5.3.2. Humid specimen having a woven nylon substrate in the middle layer.

According to visual inspection (without microscope) the specimen broke in the same way as dry specimen. A picture made during microscopic research is shown in figure 36.

This microscopic research revealed a few things:

1)   The damage due to moisture absorption is not visible at the used enlargements.

2)   All the tested specimen showed deformations of the fibres lying under or above fibres crossing perpendicular.

3)   In both the specimen with a copper and with a nickel layer the metal layer has been pulled of the fibres at some places. In these specimen clear cracks are visible between the fibre bundles and the fibres crossing perpendicular.

4)   Because the specimen without a metal layer did not break in the middle layer, no clear cracks or other irregularities could be observed in these specimen.

4.5.4.1. Dry specimen having a woven polyester substrate in the middle layer.

If a metal layer was present in the middle layer, the specimen breaks in the middle layer as shown in figures 29b and 29c. The structures without a metal layer did not clearly break in the middle layer. Pictures of the structures and of possible cracks are shown in figures 37 and 38.

Microscopic research revealed the following things:

1)   The middle layer contains much less resin than the middle layers having a random structure.

2)   In comparison to the woven nylon, the fibre bundles in the woven polyester contain much more fibres. Only the radius of the fibres is somewhat smaller.

3)   The untested structures having a metal layer are much the same. The metal layer is only present at the outside of the fibre bundles. Another eye-catching thing is the fact that the nickel copper layer is much thicker than the other metal layers.

4)   Testing the structures with a metal layer on the polyester, results in cracks running through the fibre bundles, along the boundary area between the fibres and the metal layer and along the fibres that cross the fibre bundles perpendicular. Besides this cracks are visible at the boundary area of the middle layer and the carbon epoxy composite. The fibres lying in the neighbourhood of a crack often are deformed and sometimes they are broken.

5)   The structure without a metal layer (only woven polyester) contains more resin than the same structures with a metal layer. The connection between the fibres and the resin seems to be rather good. Even after the specimen has been tested. In these tested specimen hardly any clear cracks or deformed fibres are visible.

4.5.4.2. Humid specimen having a woven polyester substrate in the middle layer.

According to eye observations right after the bending tests the humid specimen with a metal layer broke, just as the dry specimen, in the middle layer (figures 29b and 29c). The specimen without metal showed first ply failures in the carbon-epoxy composite (figure 29a). Pictures of the middle layer and of possible cracks are shown in figures 39 and 40.

A few things can be remarked:

1) The untested humid structures are rather the same as the untested dry structures.

2) In the neighbourhood of a crack the fibres often are deformed or cracked. This also accounts for the fibres crossing another fibre perpendicular.

3) Especially in the middle layer with copper the crack often courses through the fibre bundles. Thus the metal layer remains rather unharmed.

4) In the middle layer with copper and nickel the metal layer has sometimes been pulled of the resin. This did not happen very often.

5) Regularly, a small crack is visible at the boundary between a fibre bundle and a fibre crossing this bundle perpendicular. This accounts for all the specimen (even the one without a metal layer).

6) The specimen without a metal layer shows a small crack at the boundary of the carbon-epoxy composite and the middle layer. Besides this the crack also courses along the fibres in the bundles. One has to realize that the observed crack is not a first-ply-failure. As mentioned before, the specimen without a metal layer first broke in the carbon-epoxy composite.

CHAPTER 5: ANALYSIS OF THE RESULTS

5.1 Roughness tests.

During the roughness measurements the laser beam scans over the surface of the woven and random structures. The measured roughness values mainly depend on:

1)   The number and the deepness of the holes in the structure (the porosity). The diameter of the laser beam is 1 micrometer, while the holes in the structures are much larger (following from microscopic observations).

2)   The roughness of the different fibres.

3)   The roughness of the metal layer.

4)   The way the metal layer lies on the different fibres.

The woven structures show a higher roughness in the 45° directions. This is obvious because in these directions the structure is more irregular. That means, more deviations in surface height will be measured.

Because of the symmetry of the woven structures, the roughness values in the 90° and 0° directions are much the same.

In case a metal layer has been absorbed on the substrate, the structure is less porous. This results in a lower Ra value. The reductions of the Ra-values in the structures with a metal layer might also be the result of a possible lower roughness of the metal surface in comparison to the surface of the fibres.

The aramide structures (with and without a metal layer) have a higher Ra-value than the other structures. This is probably a result of the much higher porosity of these structures. The structures of the thick aramide layers are the same as those of the thin aramide layers. Only the first one is thicker. Therefore the differences between the highest and the lowest points of the scanned surface are larger. This latter fact explains the even higher Ra-values of the thick aramide structures.

The aramide structures with a copper layer are more rough than the ones without copper. This might be caused by the structure being less porous in case a metal layer has been absorbed. One has to realize that in a very porous structure the roughness will increase in case the porosity decreases. Another explanation might be that the roughness of the aramide fibres themselves is lower than the roughness of the copper layer. Besides this the way in which the copper layer has been absorbed on the aramide fibres seems to be rather irregular (microscopic observations). This may also contribute to the higher roughness values.

5.2. Moisture absorption.

The straight line in figure 19 shows that the moisture absorption rate reduces in time. Extrapolation of the line provides an estimation of the moisture amount in the specimen after three months. This is 1.72 %. This extrapolation does not take into account the existence of a saturation level, which is for carbon-epoxy composites of the order of 1 % [1], [2]. So the level of 1.72 % found by extrapolation will never be reached.

5.3. Microscopic observations

5.3.1. Dry specimen.

Observations of the broken structures with a metal layer show that for all these specimen the weak spots are:

1)   The boundary between the metal and the fibre.

2)   The fibre bundle itselve. This means that often a crack courses right through the fibre bundle. Therefore the resin impregnation in the fibre bundles might not be optimal. This can be explained by the fact that the fibre bundles are covered by a metal layer.

3)   The boundary of the middle layer and the carbon epoxy composite.

The fractures and deformations of the fibres lying in the neighbourhood of a crack is probably caused by slipping of the crack surfaces over each other.

The fibres crossing each other perpendicular (in the woven structures) have only a small tangent plane. Therefore the stresses might be rather large at these places. This explains the clear visible deformations of these fibres. Especially after testing.

The irregularity of the aramide substrate with a copper layer, might be caused by a bad adhesion of the copper to the aramide fibres or by the production of the aramide-copper layers.

5.3.2. Humid specimen.

All the cracks appearing in the middle layers of the humid specimen were not as visible as those appearing in the dry specimen. Reasons for this might be that the humid middle layer is less brittle or that the cracks in the humid specimen arose at lower shear stresses. The latter fact takes care that the energy release rate is less.

The course of the cracks in the humid specimen is globally the same as in the dry specimen. This means that the weak spots in the humid middle layers are the same as in the dry middle layers. Only in the middle layer with a polyester substrate and a copper layer, the metal layer remained relatively unharmed. From this one may conclude that the adhesion between the copper layer and the polyester fibre is better resistent to humid conditions than the adhesion of nickel to polyester.

In the thin aramide layers, the shear strength reduction of the middle layer without copper is less than the shear strength reduction of the layer with copper. This can be explained by the fact that in the middle layer with copper, the adhesion between the copper and the resin reduces.

5.4. Bending tests.

5.4.1. Dry specimen.

During the bending tests the specimen without a special middle layer (A) almost always broke in or in the neighbourhood of the middle layer. Knowing that the material is homogeneous, one may conclude that the maximum shear stress is working in the middle layer. Just as predicted by the Euler-Bernouilli solution. The value of the shear strength is 100.66 MPa (three-point bending). The value given by the manufacturer of the carbon-epoxy composite is 92 MPa. Only this

latter value has been determined by a three-point bending test with a span to depth ratio of five. The ratio used in our tests is four. Looking at figure 15 learns that the higher the span to depth ratio the lower the determined shear strengths. Knowing this, the value of 100.66 MPa is very reasonable.

In case the specimen has a special layer in the middle the situation changes. With respect to these specimen two conclusions are possible :

1)   The Euler-Bernouilli solution is still valid.

2)   The distribution of the shear stress does not agree with the Euler-Bernouilli solution, and will probably be as shown in figure 11. A possible reason for this might be that the material with a middle layer cannot be supposed to be homogeneous anymore.

For the specimen that did not seem to break in the middle layer (B, C, D, G, J, M, O), it is rather doubtful whether conclusion one is valid. Because if the middle layer would be stronger than the rest of the material, the calculated shear strength should be higher than the shear strength of the reference material (A). Both the three-point and the four-point bending tests show that the reference material has the highest shear strength. If this Euler-Bernouilli solution is not valid, strong doubts will rise with respect to the usefulness of equation two for the calculation of the shear strength.

The random nylon structure with a metal layer does not break in the middle layer while the woven nylon structure with a metal layer does. Knowing that an important difference between those materials is their porosity (the porosity of random nylon is much higher), a possible conclusion might be that the resin content in the middle layer has an influence on the kind of first ply failure that occurs.

The structures with a woven substrate (polyester or nylon) have the highest shear strengths and do not break in the middle layer, in case no metal layer has been absorbed. These none-metallized structures have the highest roughness value and the highest porosity (in comparison to the other structures with the same substrate). These observations subscribe that the resin content in the middle layer has an influence on the kind of first ply failure that occurs.

The woven nylon structures with a metal layer all showed first ply failure in the middle layer. Because in both the three- and the four-point bending tests, the structure with a copper layer has a higher shear strength than the one with a nickel layer, it seems that the adhesion of the copper to the fibre or to the resin is better.

The specimen with a woven polyester substrate and a metal layer also break in the middle layer. Only in this case the specimen with the nickel layer has a higher shear strength (in both the four- and the three-point bending tests). Therefore it seems that the adhesion between the fibres and the metal layer plays an important role. Besides this, in the structure with a polyester substrate the adhesion of the nickel layer to the polyester fibres seems to be better than the adhesion of copper layer to these fibres.

Microscopic observations showed indeed that the adhesion between the fibres and the metal layers is weaker (and therefore more important) than the adhesion between the metal and the resin.

Because within each group the metallized middle layers have the same structure (microscopic research and measurement of roughness values) and the differences between the shear strengths are rather low, one can conclude that the differences in adhesion strength of the different metal layers are not very large.

The shear strength of the specimen with a polyester substrate and a copper and nickel layer lies in all cases closer to the shear strength of the polyester structure

with a copper layer than to the value of the structure with a nickel layer. In the copper nickel structure the copper is lying against the fibre and the nickel layer is lying on the copper layer. Thus again the boundary between the fibre and the metal layer is important.

In the aramide structures the thick layers have a higher shear strength than the thin layers and only the thin layers break in the middle. A possible reason might be that the thick layer is more able to resist the imposed deformations during the bending tests.

In order to discover a possible relation between the measured roughnesses and the shear strengths, the shear strengths of the different materials have been plotted against the roughness. This is shown in figure 41. It follows that a positive relation might exist for the aramide and for the woven nylon substrate. This is more a first speculation than a serious conclusion. From the fact that not in all cases a higher roughness corresponds with a higher shear strength (compare the thin random aramide with the random nylon), one can conclude that other mechanisms like chemical adhesion also influence the (apparent) interlaminar shear strength as it is determined by the bending tests.

In figures 42, 43, 44 the shear strengths have been plotted against the different types of layers on the substrate. From this one can see that in almost al cases (three- and four- point bending and dry and humid circumstances) the random nylon substrate seems to have the highest shear strength. Only looking at the metallized layers shows that for nickel and copper the best substrate is random nylon and the worst is woven polyester. And for the copper, the aramide (thin) is better than the woven nylon.

5.4.2. Humid specimen.

After moisture absorption the distribution of the shear strengths over the different materials is almost the same as before absorption. Because of this the different mechanisms that determined the initiation of the different cracks in the dry specimen may possibly be the same in the humid specimen.

Only now the specimen having a polyester substrate with a nickel layer has a lower shear strength than the same specimen with a copper layer. Therefore the adhesion of the nickel layer to the polyester fibre seems to be more sensitive to moisture absorption.

Under humid conditions the adhesion between the aramide fibres and the resin is expected to reduce a lot. This is not shown by the results of the different bending tests. A reason might be that the shear strength of the middle layer is for a great deal determined by the resin. Indeed microscopic observations showed that the resin contents in the middle layers with an aramide substrate are very high.

CONCLUSIONS AND RECOMMENDATIONS.

As a consequence of this research a few important final remarks can be made:

1)   The reduction of the apparent interlaminar shear strength as a consequence of the existence of a special middle layer in the unidirectional carbon-epoxy composite is not very large. This accounts for all the middle layers. Thus the composites with such special middle layers may be applicable in practice.

2)   Knowing that in the metallized middle layers, the boundary between the fibres and the metal layers seems to be a weak spot (under humid and dry conditions), it is advisable to try to improve the adhesion between the fibres and the metal layers. Only for the aramide substrates a metal layer seems to improve the ILSS. Further investigations should be done to know the exact reason for this latter fact.

3)   The amount of resin in the aramide middle layers is very high. Therefore the influence of the stiff and strong aramide fibres on the mechanical properties of the middle layer is probably not very great. To have more profit of the mechanical properties of the aramide fibres it might be better to use a woven aramide structure.

4)   From the middle layers with a metal layer and a nylon substrate the random structures have the highest apparent shear strengths (in both the three- and four point bending tests and under humid and dry circumstances). The worst substrate seems to be the woven polyester (figures 42, 43, 44). As the random nylon has a lower roughness value but generally a higher interlaminar shear strength than the random aramide one can say that for the random structures not only the roughness but also the type of fibre has an important influence on the interlaminar shear strength.

5)   In the woven structures only the outside of the fibre bundles is covered by a metal layer. For the electrical conductivity of the middle layer, it would be better to have a structure in which each separate fibre is covered with a metal layer. The metal layer makes it also less convenient to impregnate the fibre bundle with resin which makes that the fibre bundle is a weak spot in the middle layer.

6)   The middle layer seems to have an influence on the shear stress distribution in the specimen. Therefore the measured apparent shear strength might deviate from the real shear strength of the middle layers. Therefore it is sensitive to perform some more advanced tests than the bending tests on the composites with a special middle layer. For example mode I and mode II fracture toughness tests.

7)   Under humid conditions the specimen with the special middle layers do not have large reductions in shear strength (not more than the carbon-epoxy material itselve). Therefore these materials seem to be applicable in humid circumstances.

8)   Maybe it would be better to produce carbon fibres that are covered with metal layers. From these metallized carbon fibres one can make prepegs (unidirectional). These prepegs can be worked up in a composite structure. The advantages of this might be a better covering by the metal layer, a better impregnation of the metallized layers by the resin and a higher ILSS (if the adhesion between the metal layers and the carbon fibres is good).

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[5]   JAMES, J.M., BROWNING, C.E., Materials Characterization for Matrix-Dominated Failure Modes, Effects of Defects in Composite Materials ASTM STP 836, 1984, 104-124.

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APPENDIX A: FIGURES AND TABLES

	22°C	80°C
interlaminar shear strength [MN/mm²] (span:depth = 5:1)	92	72
0° Flexural strength [MN/mm²] (span:depth = 40:1)	1500	1100
0° Flexural modulus [GN/mm²] (span:depth = 40:1)	130	120
90° Flexural strength [MN/mm²] (span:depth = 25:1)	83	
90° Flexural modulus [GN/mm²] (span:depth = 25:1)	7	

TABLE 1: The mechanical properties of the carbon-epoxy composite (913C Fibredux).
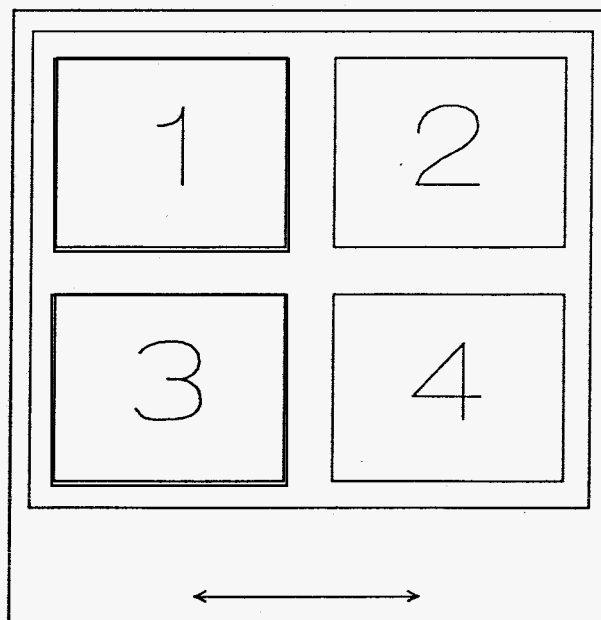
FIGURE 1: The position of the four middle layers on the twelfth plie. The arrow represents the fibre direction.
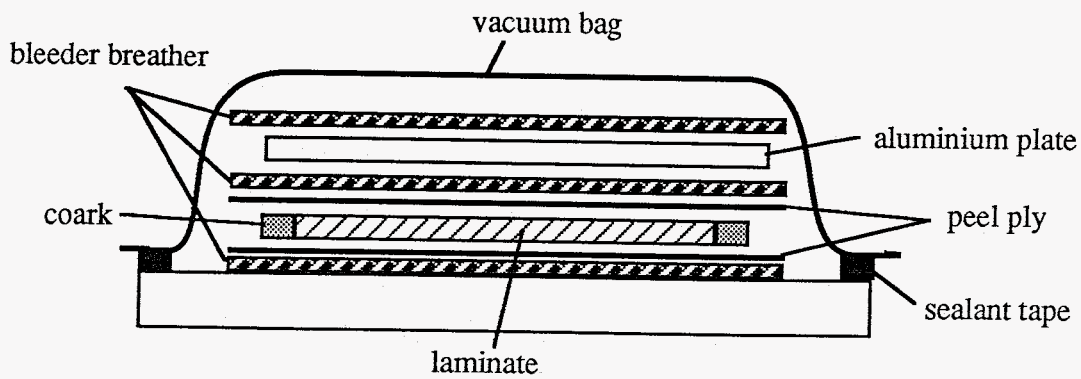
FIGURE 2A: The formation in the autoclave.
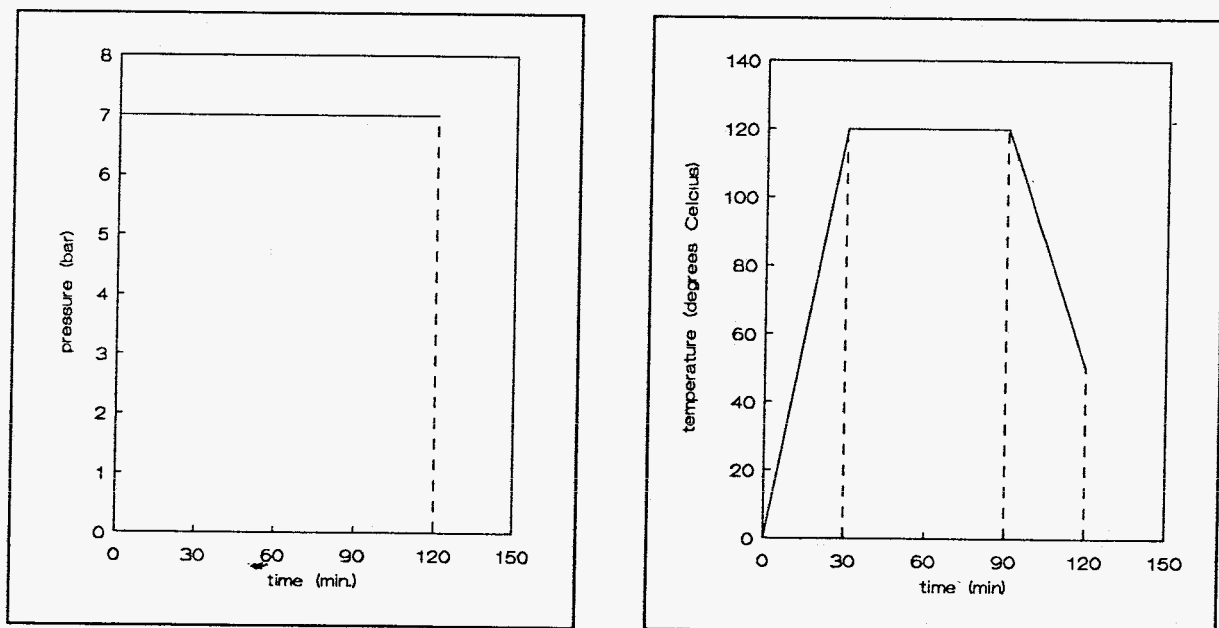
Figure 2B: The cure cycle. During this cycle the plates have been at a vacuum of 100%.
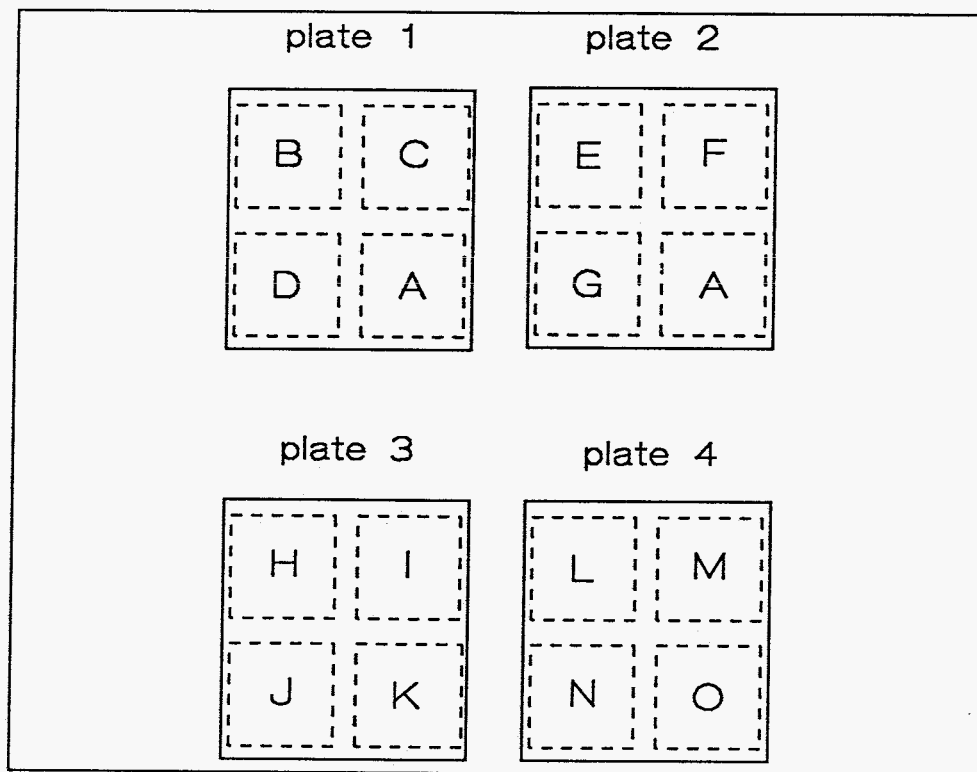
A= no special middle layer (reference material).

B= random nylon substrate with a nickel layer.
C= random nylon substrate with a copper layer.
D= random nylon substrate without metal layer.

E= woven nylon substrate with a nickel layer.
F= woven nylon substrate with a copper layer.
G= woven nylon substrate without a metal layer.

H= woven polyester substrate with a nickel layer.
I= woven polyester substrate with a copper layer.
J= woven polyester substrate without a metal layer.
K= woven polyester substrate with a copper and a nickel layer.

L= random aramide substrate with a copper layer (thin).
M= random aramide substrate with a copper layer (thick).
N= random aramide substrate without a metal layer (thin).
O= random aramide substrate without a metal layer (thick).

FIGURE 3: The structure of the plates as they come out of the autoclave.
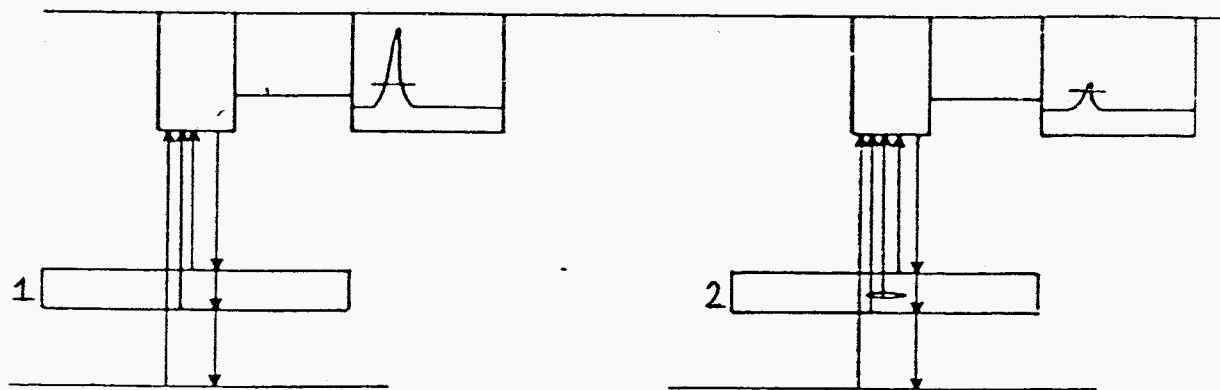
FIGURE 4: The principle of the ultra sone c-scan.

1) = unharmed specimen   2) = specimen with a delamination
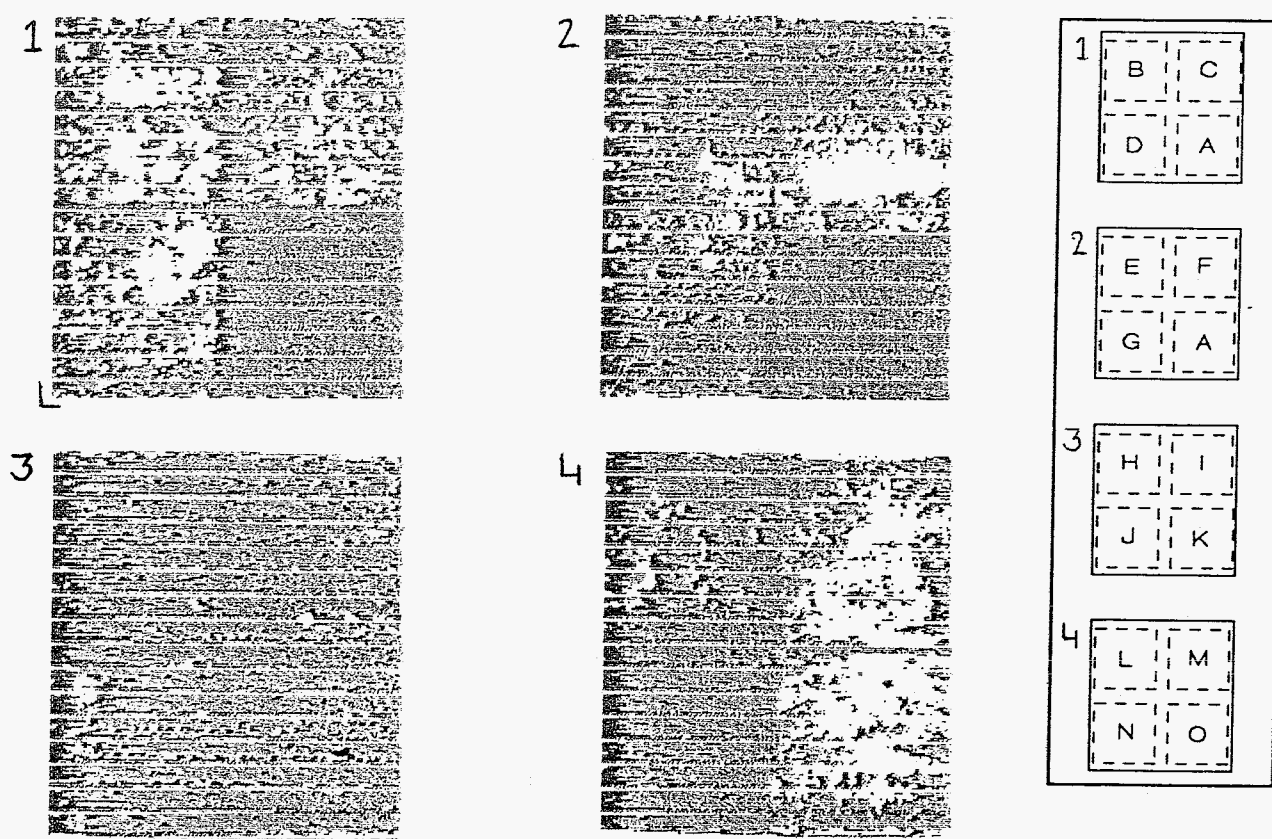
FIGURE 5 : The results of c-scanning the plates.

	plaat 1	plaat 2	plaat 3	plaat 4
damage area (%) at level 75	31.5	22.3	13.8	32.5

TABLE 2: The damage area of the different plates (%) at a level of 75. The position of different middle layers in the plates is given in figure 3.

One of the four plates that have been cured in the autoclave

The four smaller plates being cut from one big plate

The four middle layers
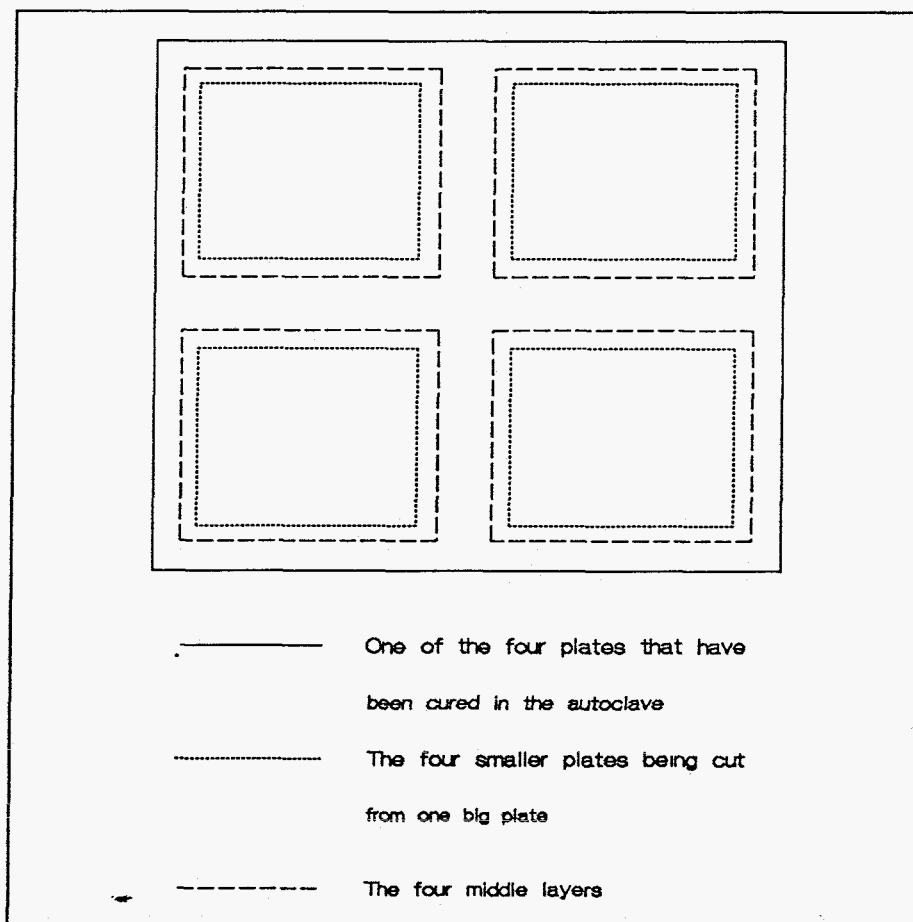
FIGURE 6: A schematic representation of the way in wich the four smaller plates (each having only one sort of middle layer) have been cut from one big plate.
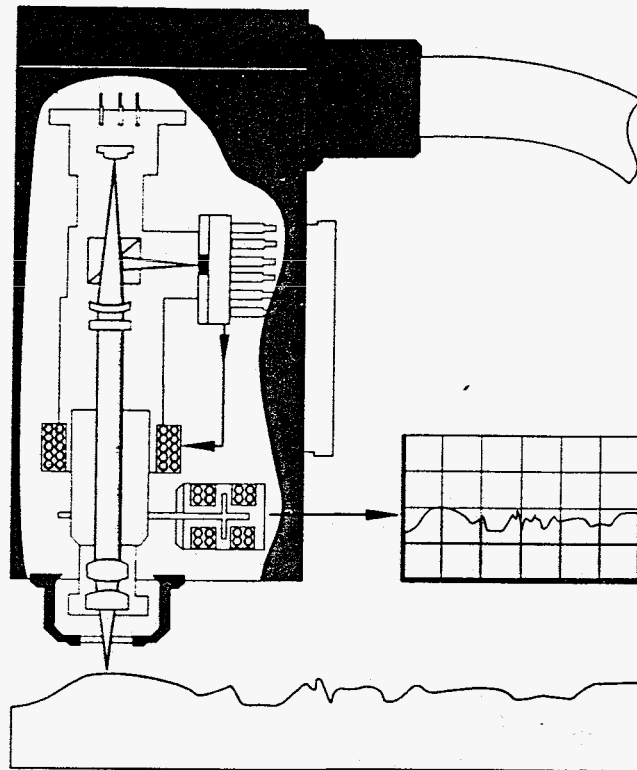
FIGURE 7: The principle of the Rodenstock roughness measurer.
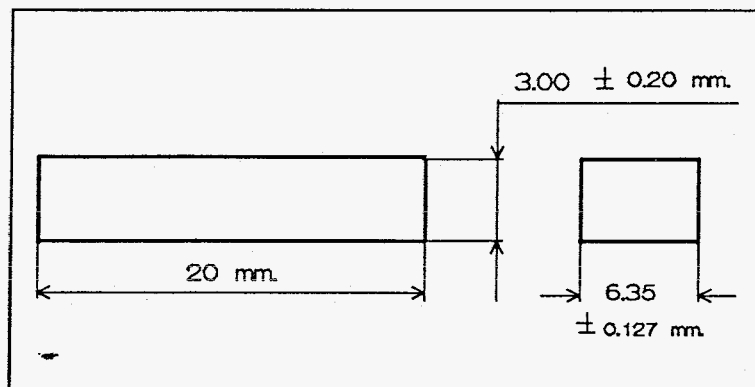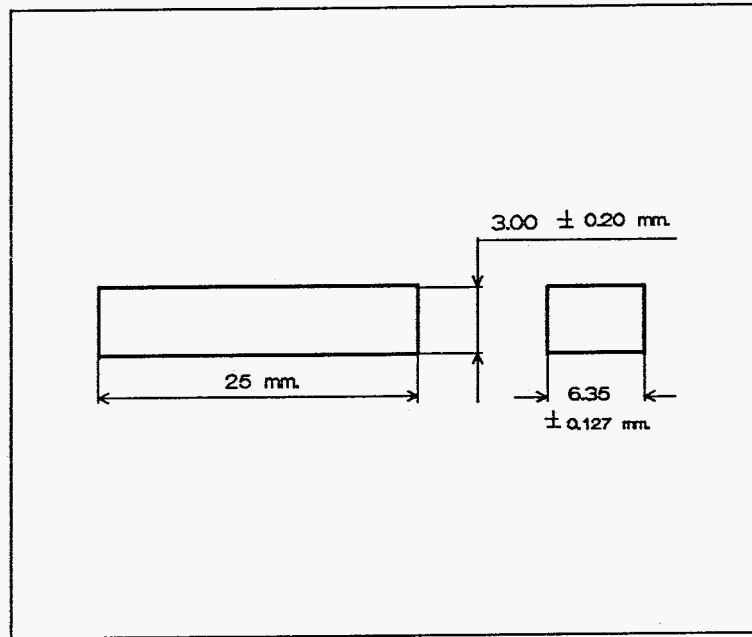

FIGURE 8:   The dimensions of the specimen, tested in the three-point bending test
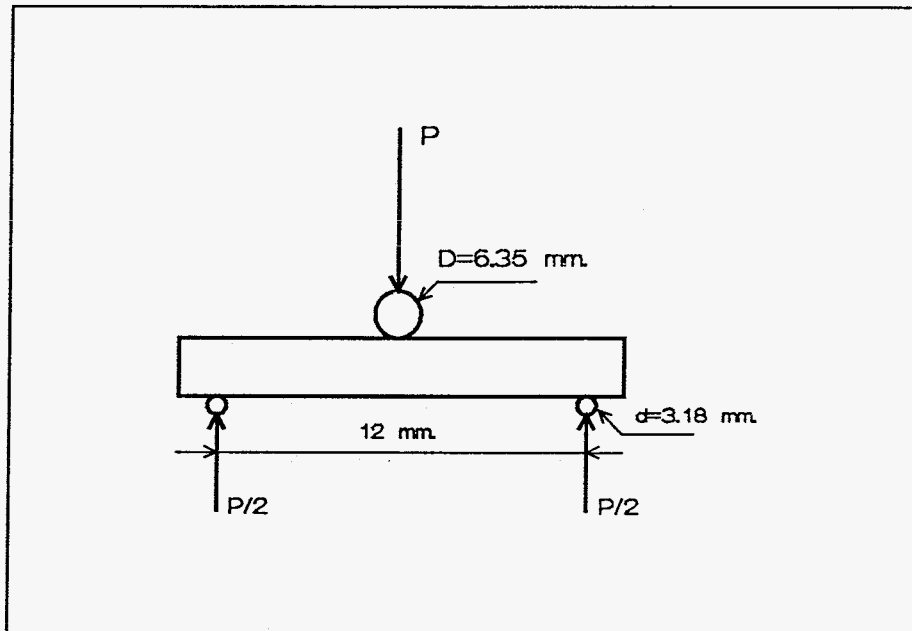
FIGURE 9: The three-point bending test.
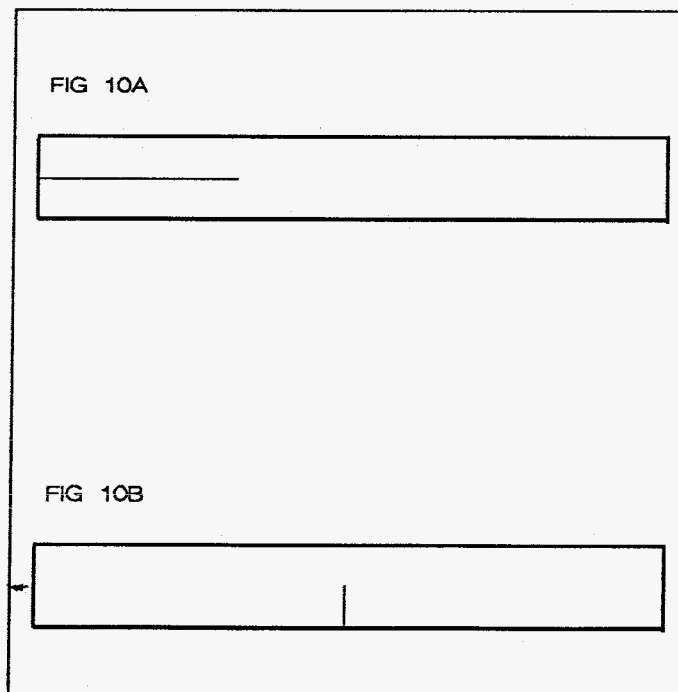
FIGURE 10 A/B: Possible cracks that may occur as a result of a bending test.
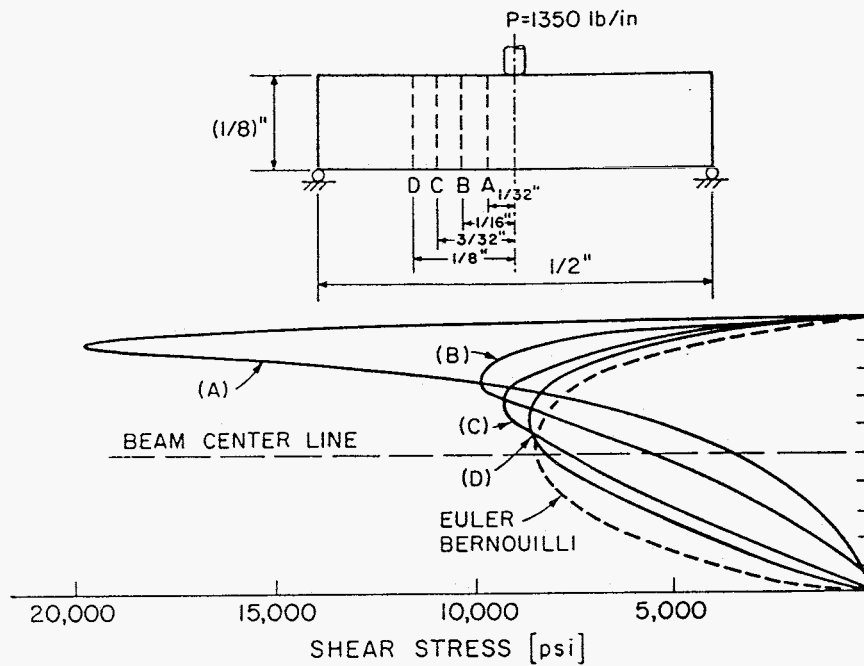
FIGURE 11: The shear stress distribution in a model of a graphite/epoxy beam (unidirectional) as calculated by an EEM programme [4]. L/d=4.

FIGURE 12: An example of fibre buckling under the loading nose, during a three-point bending test [5].

3.00 ± 0.20 mm.

25 mm.

6.35
± 0.127 mm.

FIGURE 13: The dimensions of the specimen tested in the four-point bending tests.

P/2   P/2

10 mm.

D=6.35 mm.

d=3.18 mm.

20 mm.

P/2   P/2
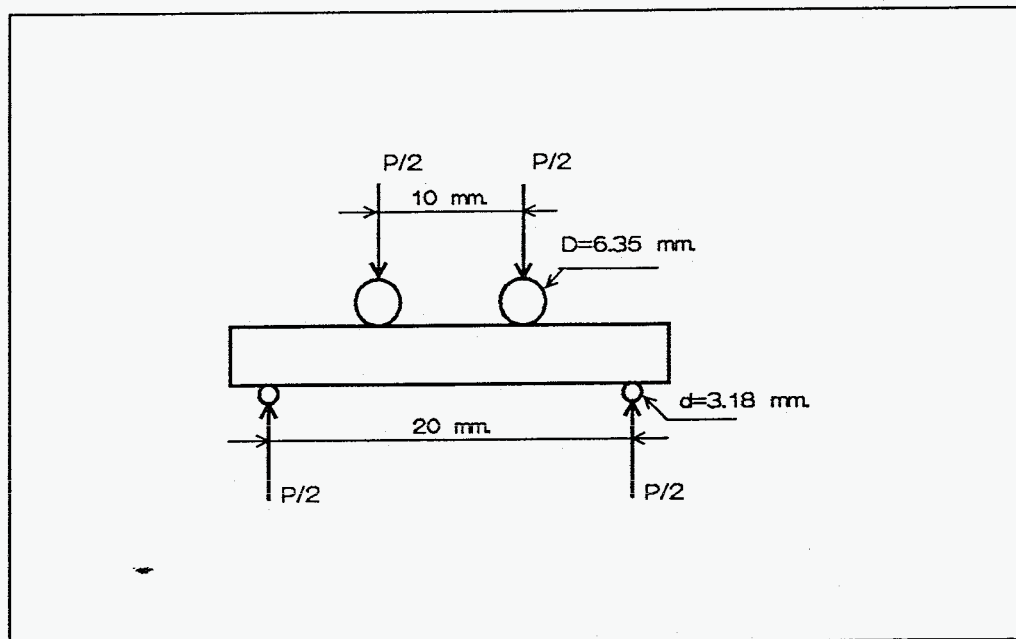
FIGURE 14: A schematic representation of the four-point bending test.
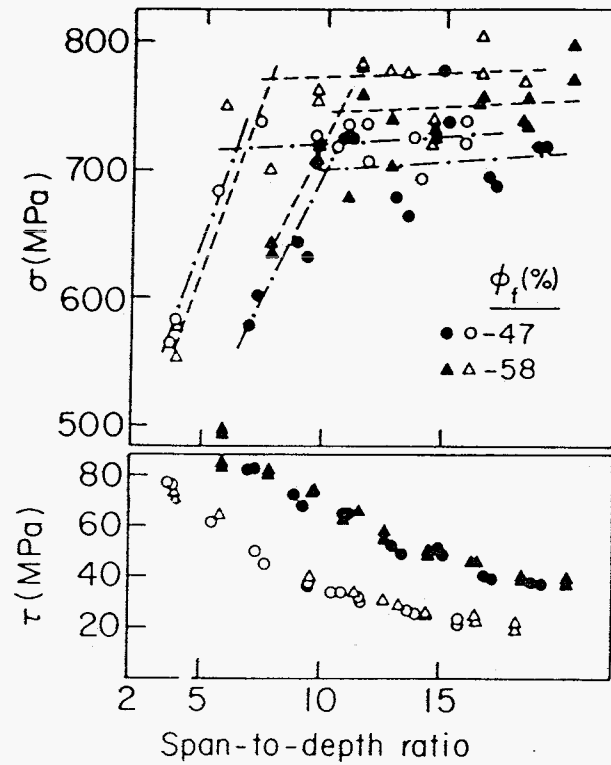
FIGURE 15: The tensile and shear stress versus span-to-depth ratio. For the same span-to-depth-ratio the shear stress/tensile stress ratio is higher in case of the four-point bending test (represented by the filled symbols, the unfilled symbols represent the three-point bending test) [6].
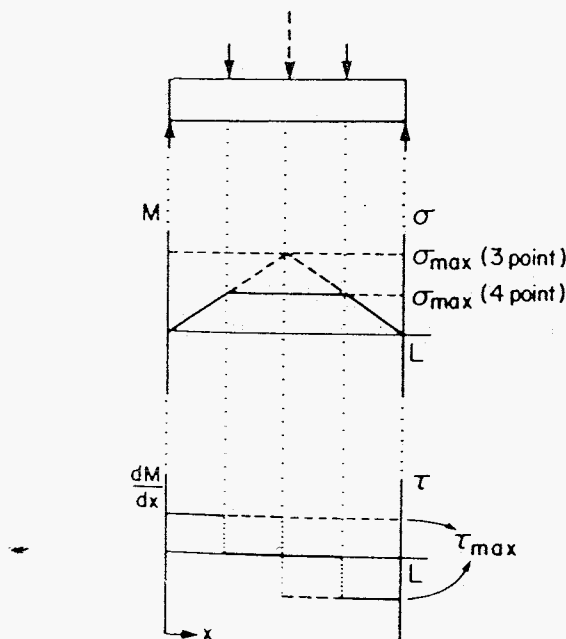
FIGURE 16: The moments and shear stresses in the specimen during a four- and a three-point bending test (according to beam theory) [6].
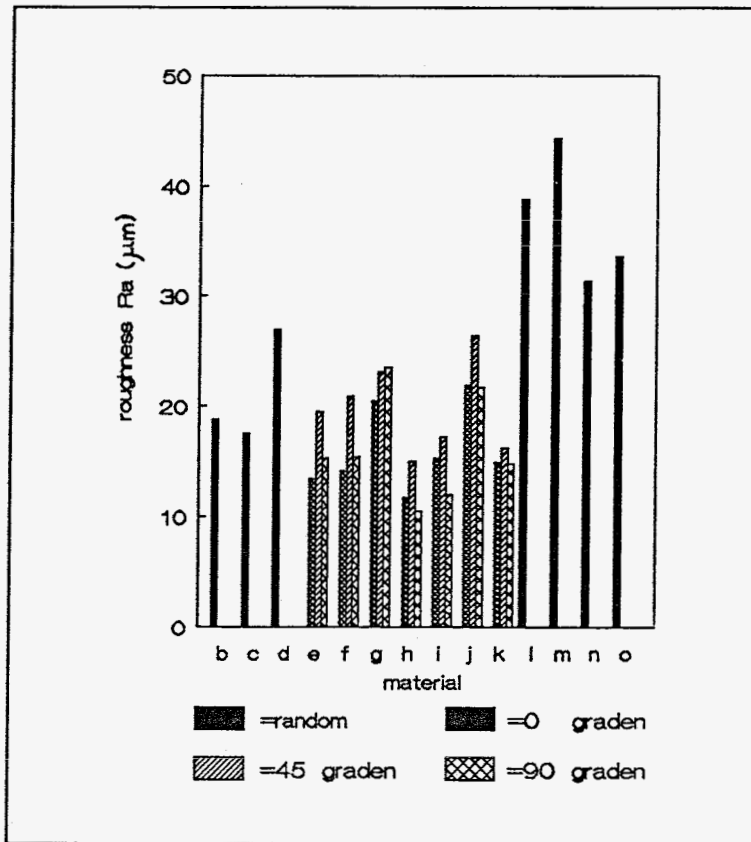
b= random nylon substrate with a nickel layer
c= random nylon substrate with a copper layer.
d= random nylon substrate without a metal layer.

e= woven nylon substrate with a nickel layer.
f= woven nylon substrate with a copper layer.
g= woven nylon substrate without a metal layer.

h= woven polyester substrate with a nickel layer.
i= woven polyester substrate with a copper layer.
j= woven polyester substrate without a metal layer.
k= woven polyester substrate with a nickel and a copper layer.

l= random aramide substrate with a copper layer (thin).
m= random aramide substrate with a copper layer (thick).
n= random aramide substrate without a metal layer (thin).
o= random aramide substrate without a metal layer (thick).

FIGURE 17: The results of the roughness measurements.

material	direction	Ra [μm.]
b	arbitrary	18.8
c	arbitrary	17.5
d	arbitrary	26.9
e	0° 45° 90°	13.4 19.5 15.3
f	0° 45° 90°	14.1 20.9 15.4
g	0° 45° 90°	20.5 23.1 23.5
h	0° 45° 90°	11.7 15 10.5
i	0° 45° 90°	15.3 17.2 12
j	0° 45° 90°	21.9 26.4 21.7
k	0° 45° 90°	14.9 16.2 14.8
l	arbitrary	38.8
m	arbitrary	44.3
n	arbitrary	31.3
o	arbitrary	33.6

TABLE 3: The results of the roughness measurements
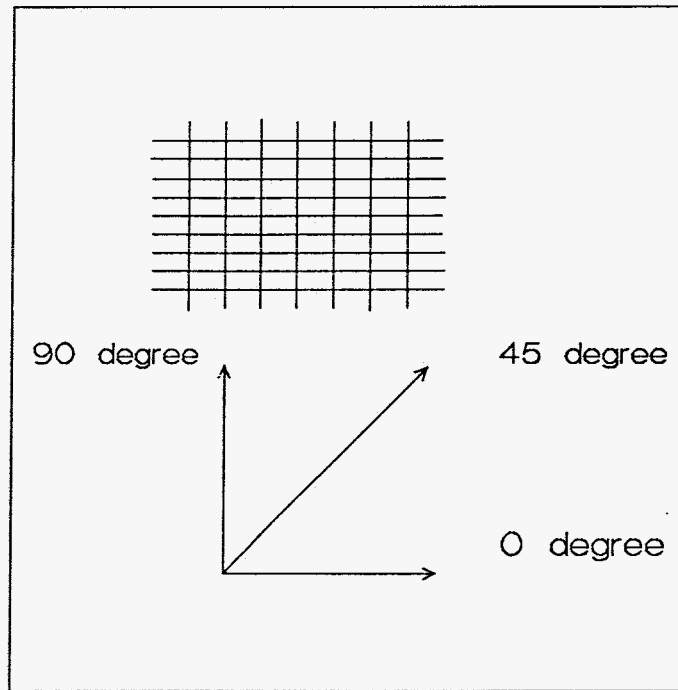
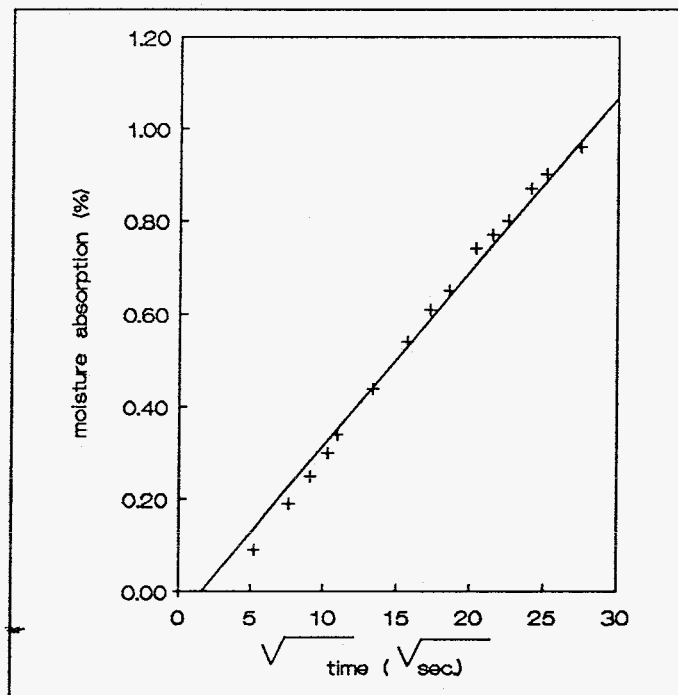**FIGURE 18:** The different directions in which the roughnesses have been measured.

FIGURE 19: The average course of the moisture absorption during one month, for the specimen without a special middle layer.

material	number of specimen	average total moisture absorption (%)
A	5	1.06
B	5	1.05
C	6	1.08
D	5	1.05
E	5	1.06
F	5	1.10
G	5	1.04
H	5	1.02
I	5	1.01
J	5	1.06
K	5	1.05
L	5	1.07
M	5	1.09
N	5	1.07
O	5	1.08

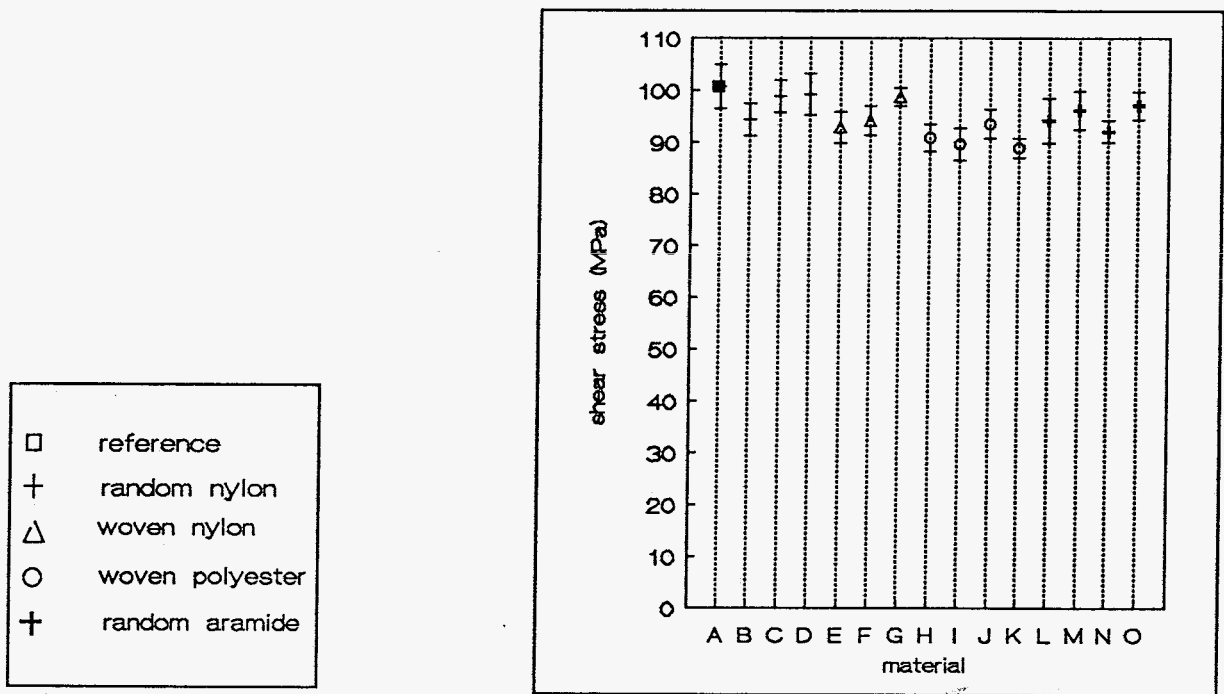**TABLE 4:** The average total moisture absorption for the different specimen.

FIGURE 20: The shear strengths of the different materials, determined by three point bending tests (dry specimen). Which material corresponds to which letter is given in figure 3.
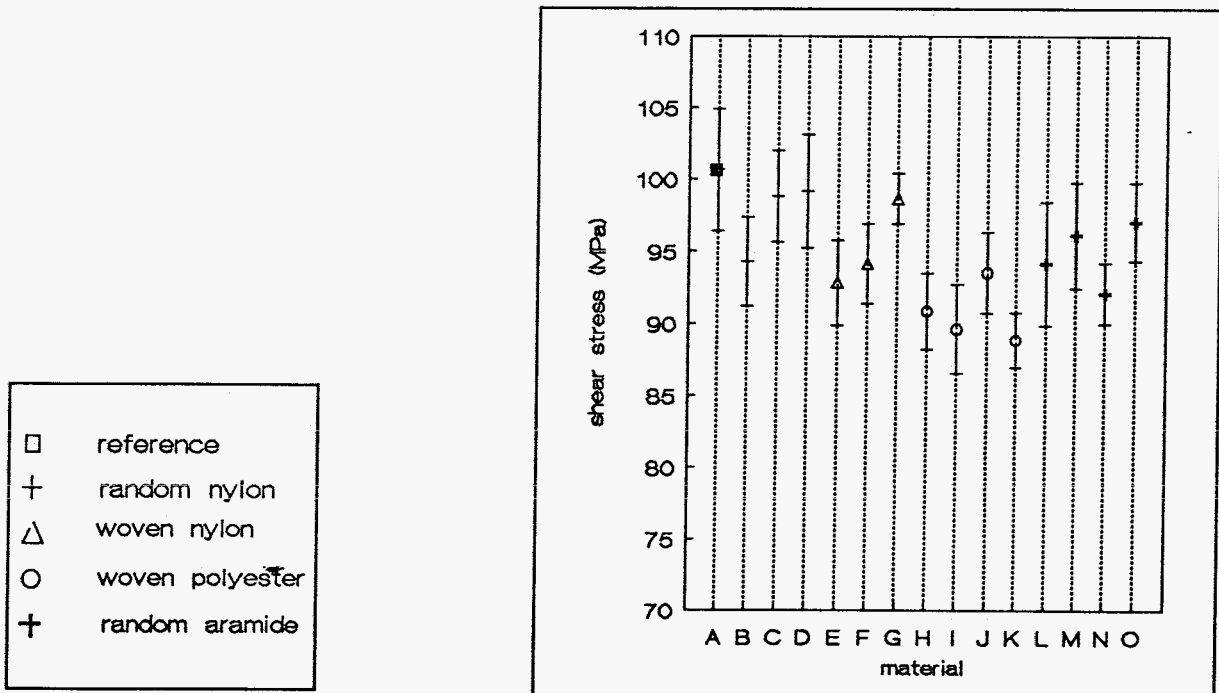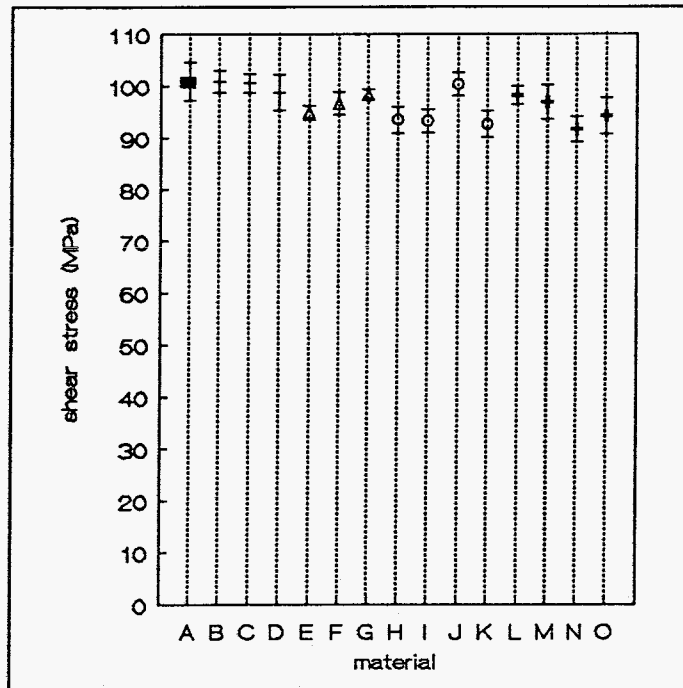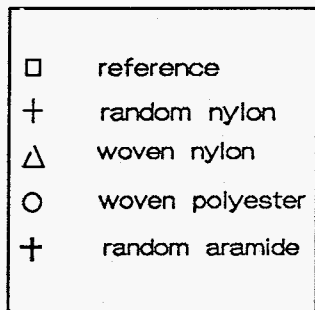

FIGURE 21: A closer look at the results of figure 20.

material	ILSS [MPa]	standard deviation	number of tests
A	100.66	3.1	12
B	94.29	3.16	13
C	98.82	3.95	12
D	99.18	4.24	13
E	92.84	2.95	13
F	94.16	2.77	13
G	98.67	1.73	13
H	90.84	2.62	13
I	89.6	3.08	13
J	93.5	2.81	13
K	88.85	1.89	13
L	94.14	4.28	13
M	96.11	3.66	14
N	92.07	2.11	11
O	97.05	2.71	12

TABLE 5: The results of the three-point bending tests (dry specimen). The materials corresponding to the different letters are given in figure 3.

FIGURE 22: The shear strengths of the different materials, determined by four-point bending tests (dry specimen). The different materials corresponding to the different letters are given in figure 3.
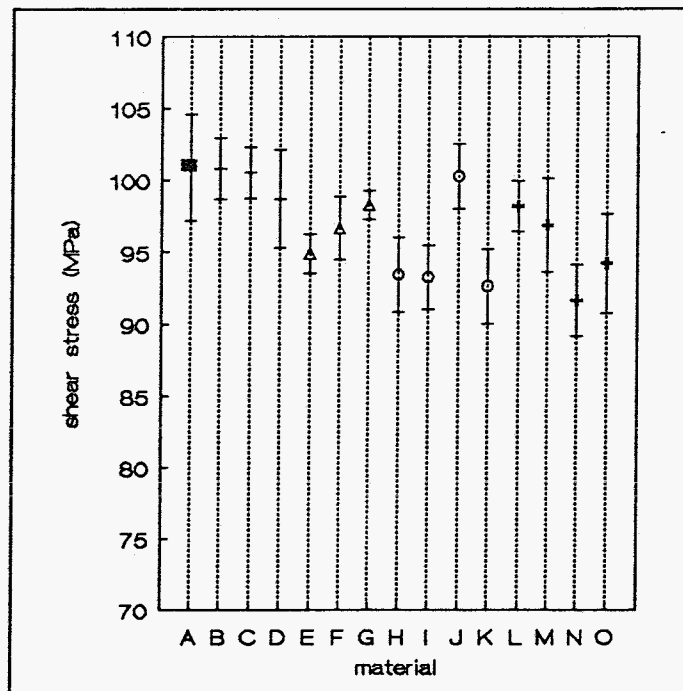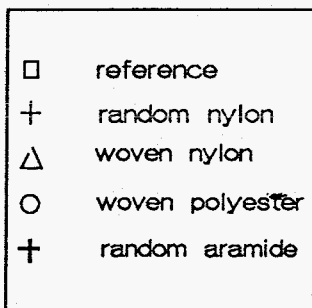

FIGURE 23: A closer look at the results of figure 22.

material	ILSS [MPa]	standard deviation	number of tests
A	100.88	3.69	8
B	100.82	2.13	6
C	100.53	1.8	5
D	98.7	3.43	5
E	94.87	1.37	5
F	96.65	2.2	5
G	98.27	1	5
H	93.43	2.58	5
I	93.25	2.21	5
J	100.27	2.26	5
K	92.63	2.58	5
L	98.19	1.78	5
M	96.87	3.28	5
N	91.65	2.47	7
O	94.21	3.49	7

TABLE 6: The results of the four-point bending tests (dry specimen).The materials corresponding to the different letters are given in figure 3.
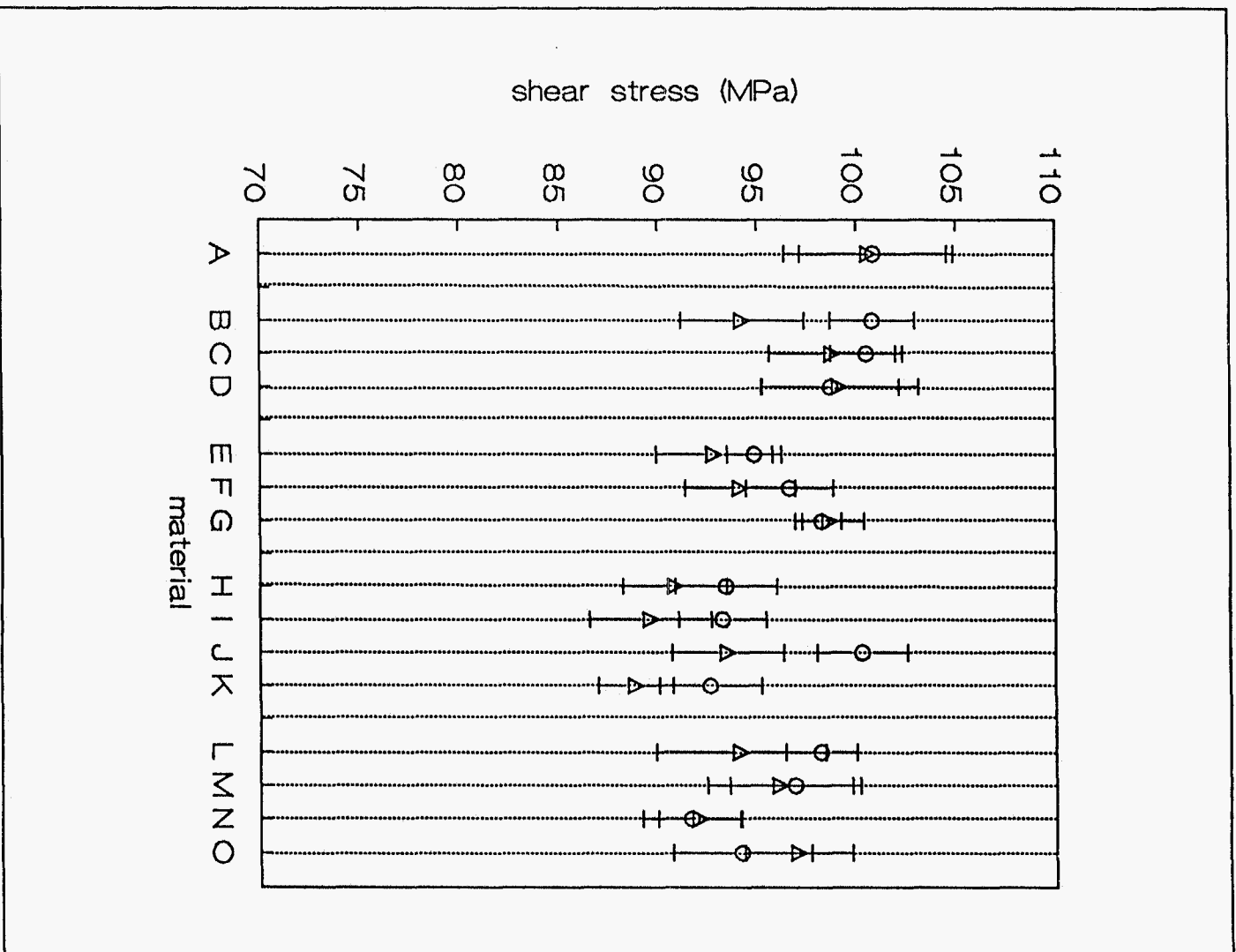
FIGURE 24: A comparison of the shear strengths determined by the four-point bending tests and those determined by the three-point bending tests (for dry specimen). The materials corresponding to the different letters are given in figure 3.
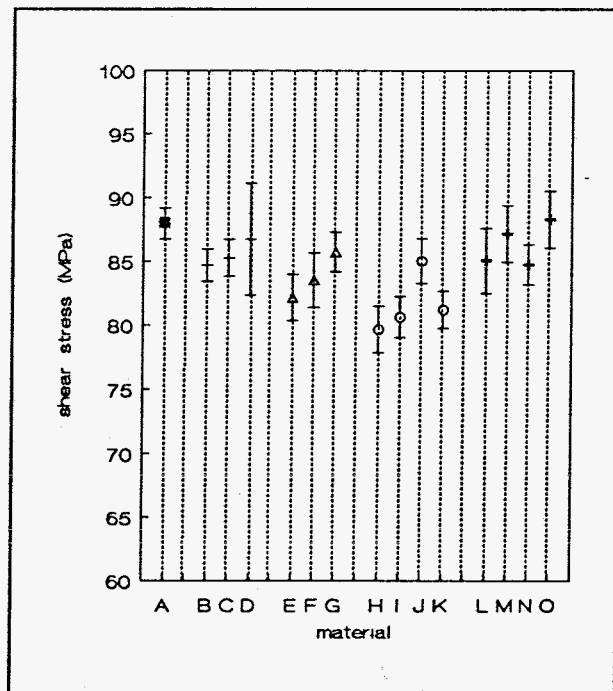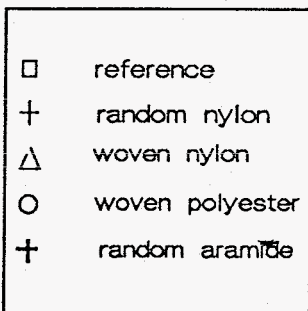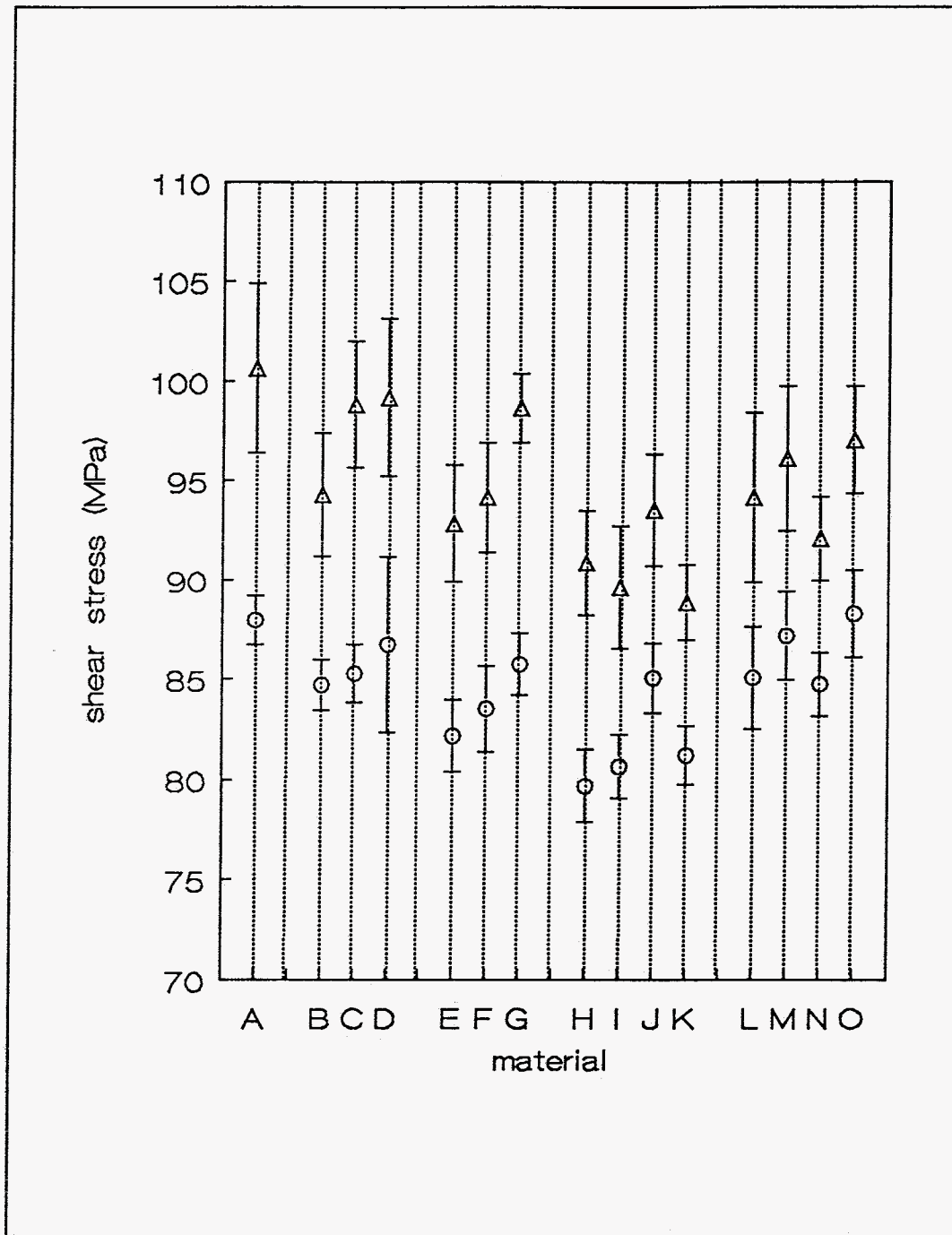
△=three-point bending   ○=four-point bending

material	ILSS [MPa]	standard deviation	number of tests
A	87.97	1.23	11
B	84.71	1.26	12
C	85.27	1.46	12
D	86.74	1.42	11
E	82.18	1.79	13
F	83.52	2.14	15
G	85.75	1.56	14
H	79.68	1.83	13
I	80.64	1.59	14
J	85.05	1.75	14
K	81.21	1.46	13
L	85.07	2.55	13
M	87.18	2.23	14
N	84.75	1.58	13
O	88.29	2.2	12

TABLE 7: The interlaminar shear strength of the humid specimen as determined by three-point bending tests. The materials corresponding to the different letters are given in figure 3.
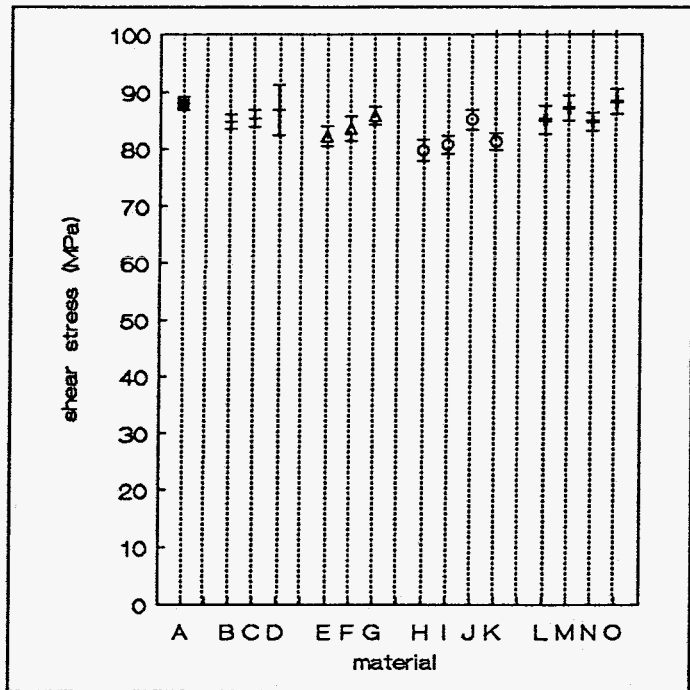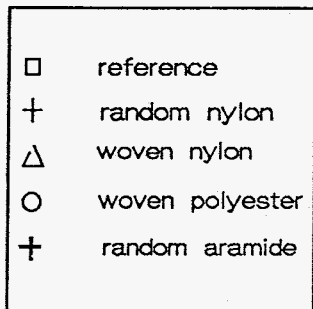
FIGURE 25: The interlaminar shear strengths of the humid specimen, as determined by the three-point bending tests. The materials corresponding to the different letters are given in figure 3.

FIGURE 26: A closer look at the results of figure 25.

FIGURE 27: A comparison of the shear strengths of the humid and the dry specimen (determined the three-point bending tests). The materials corresponding to the different letters are given in figure 3.

△=dry   ○ =humid
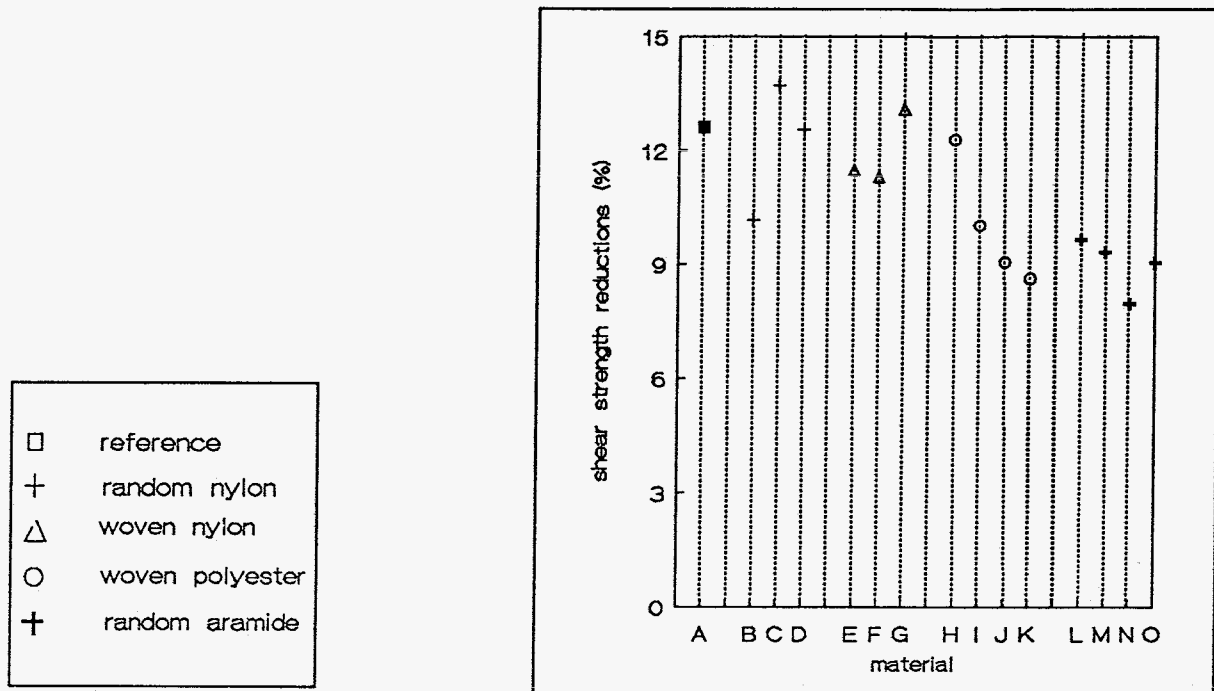
FIGURE 28: The reductions of interlaminar shear strength as a result of moisture absorption.
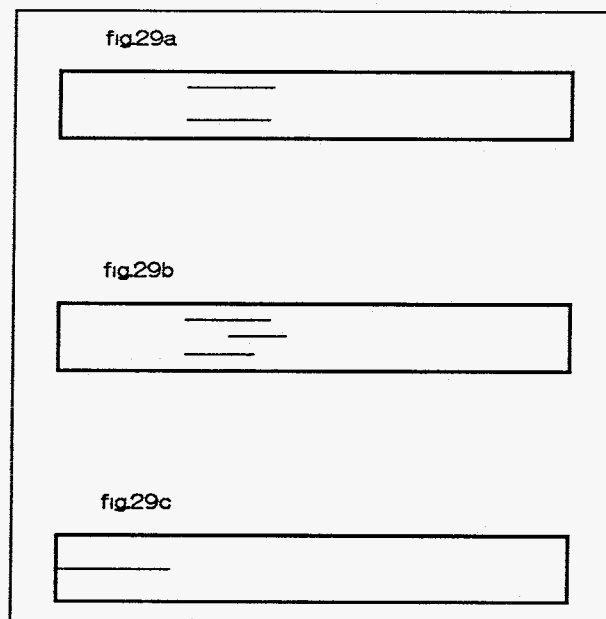
FIGURE 29: The different cracks occurring after the bending tests.
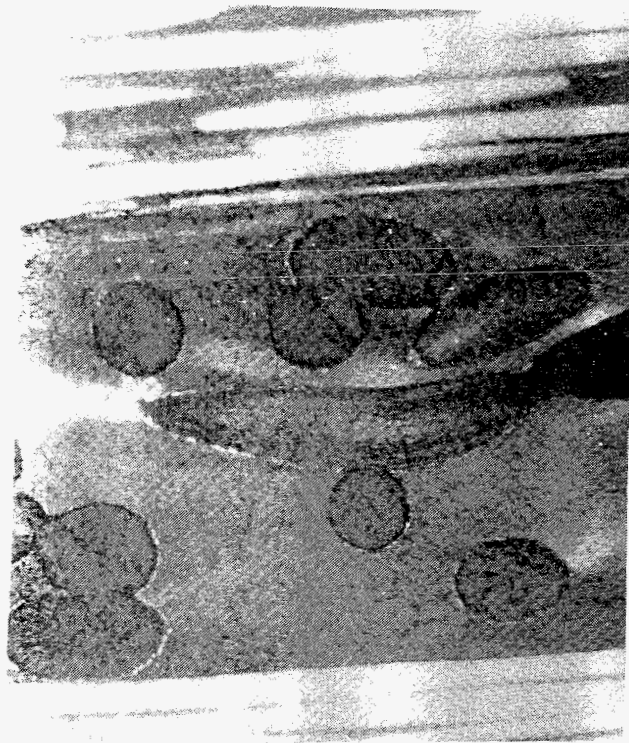
FIGURE 30: A humid untested random nylon substrate with a copper layer (enlargement is 630).
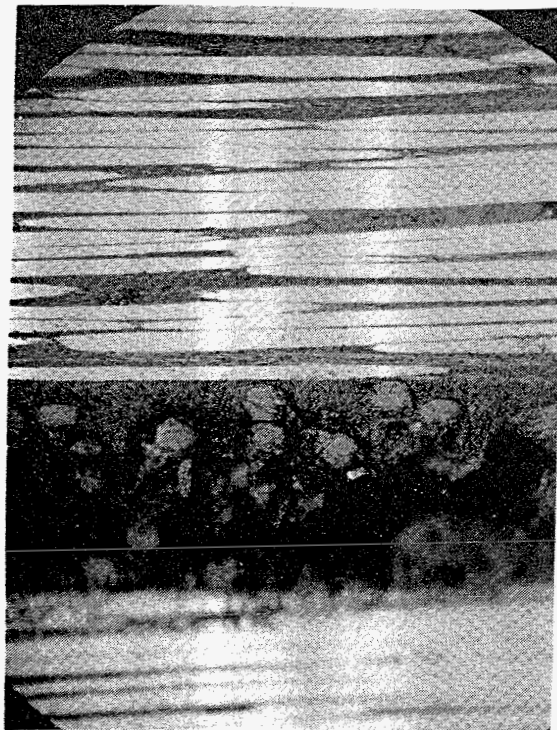
FIGURE 31: A tested dry random aramide substrate without a metal layer (thin, enlarge ment is 315).
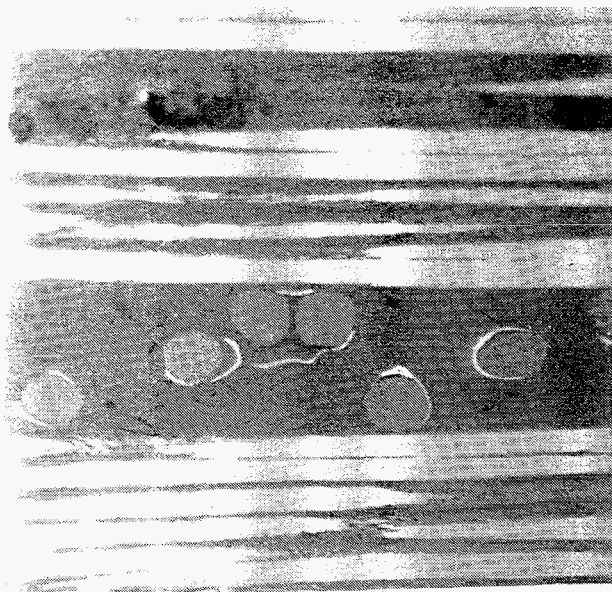
FIGURE 32: An untested humid random aramide substrate with a copper layer (thin, enlargement is 630).
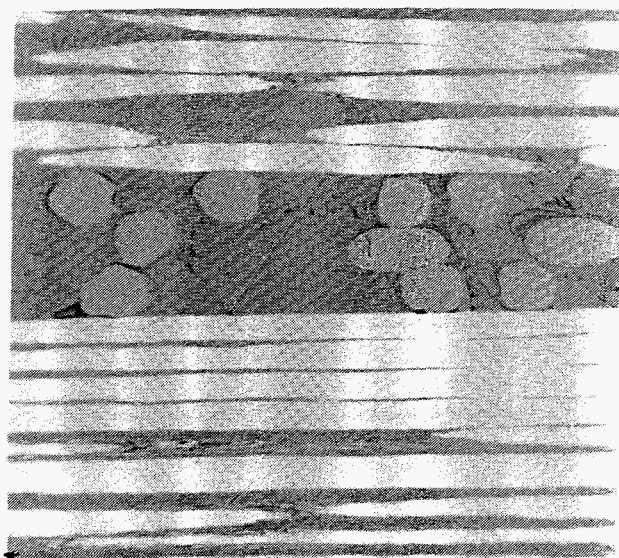
FIGURE 33: An untested humid random aramide substrate without a metal layer (thin, enlargement is 630).
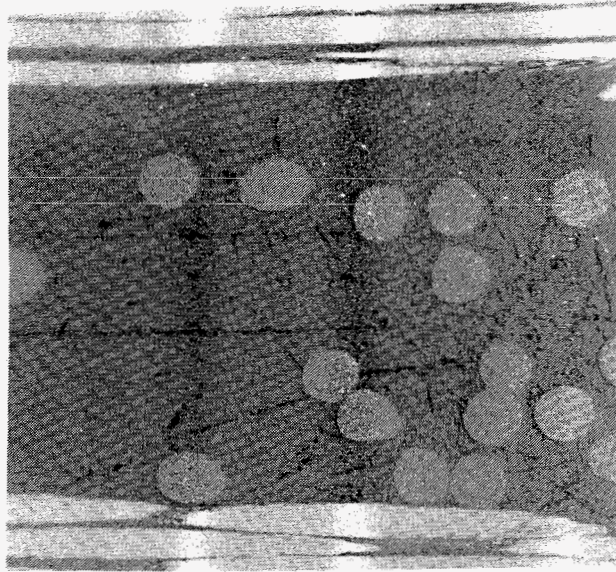
FIGURE 34: An untested humid random aramide substrate without metal layer (thick, enlargement is 630).

FIGURE 35: A tested dry woven nylon substrate with a nickel layer (enlargement is 315).
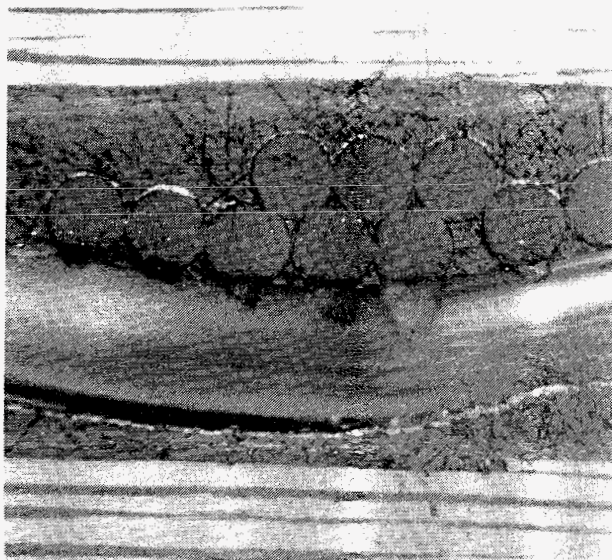
FIGURE 36: A tested humid woven nylon substrate with a copper layer (enlargement is 630).
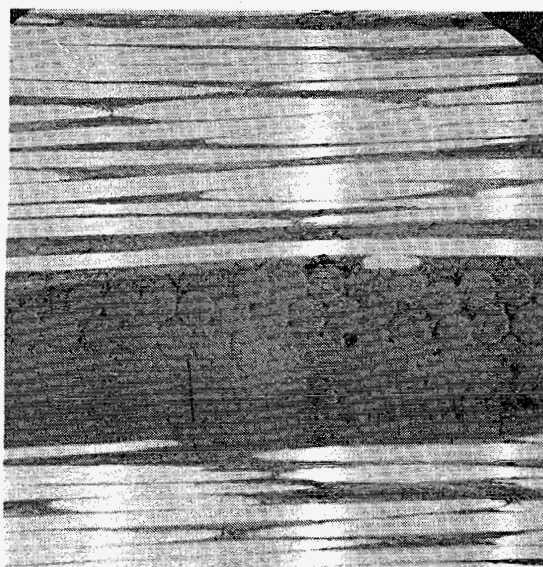
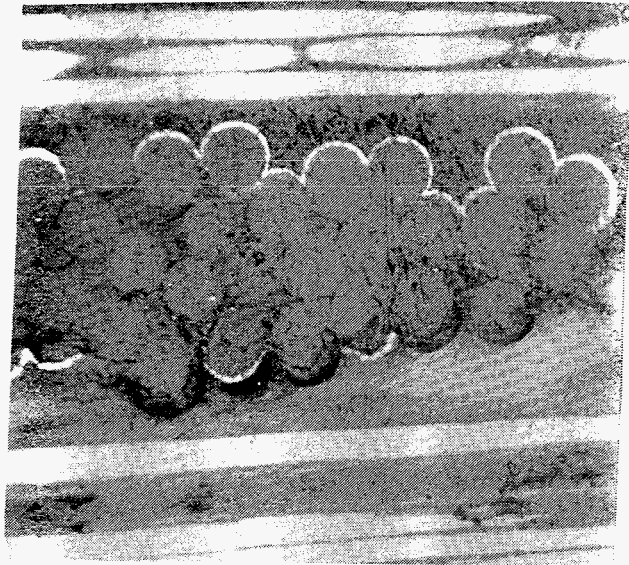**FIGURE 37:** An untested dry woven polyester substrate without a metal layer enlargement is 315).
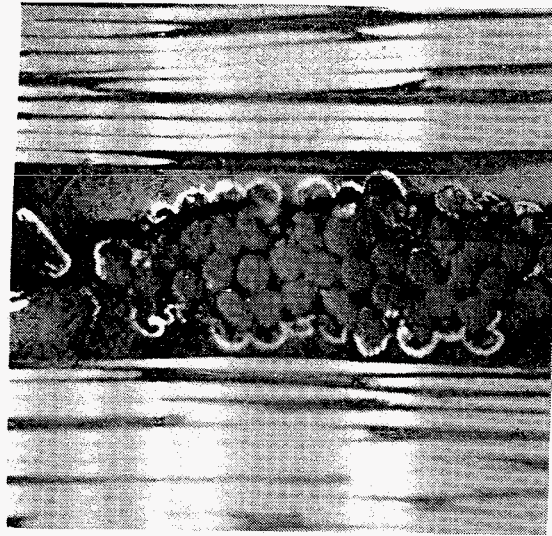
FIGURE 38: A tested dry woven polyester substrate with a copper-nickel layer (enlargement is 315).
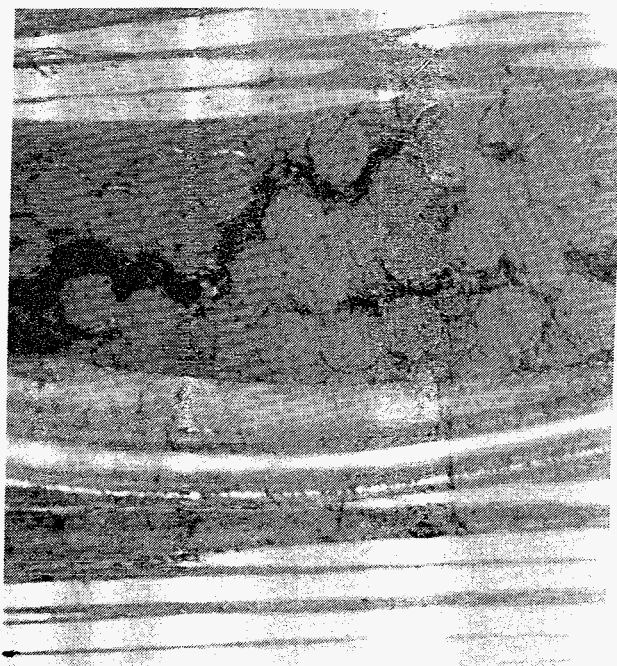
FIGURE 39: A tested humid woven polyester substrate with a copper layer (enlargement is 630).
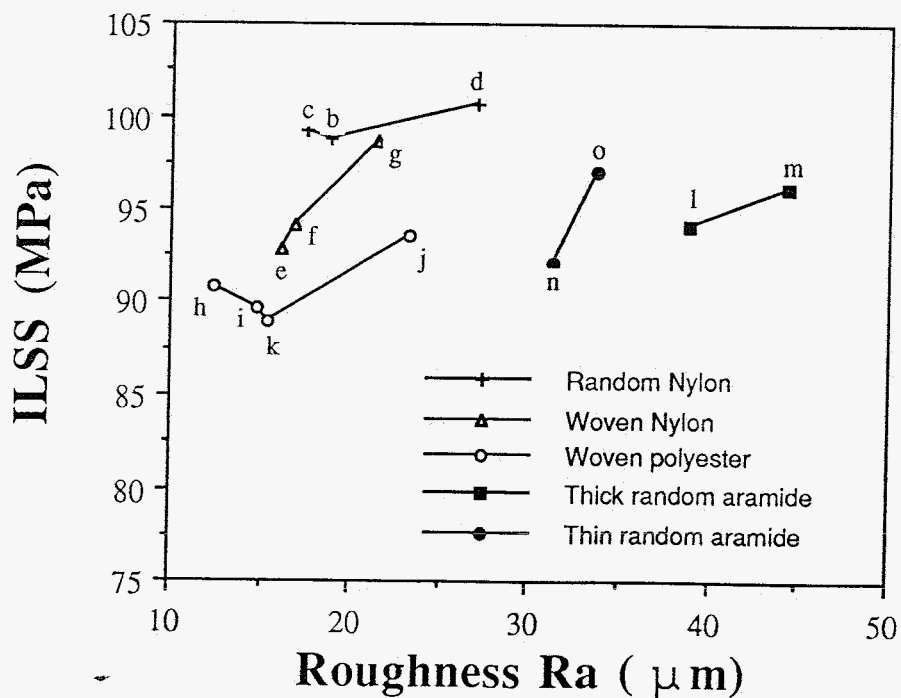
FIGURE 40: A tested humid woven polyester substrate with a copper-nickel layer (enlargement is 630).

FIGURE 41: The shear strength determined by three-point bending tests (table 5), as a function of the roughness (porosity) of the different middle layers (table 3). The materials corresponding to the different letters are given in figure 3.
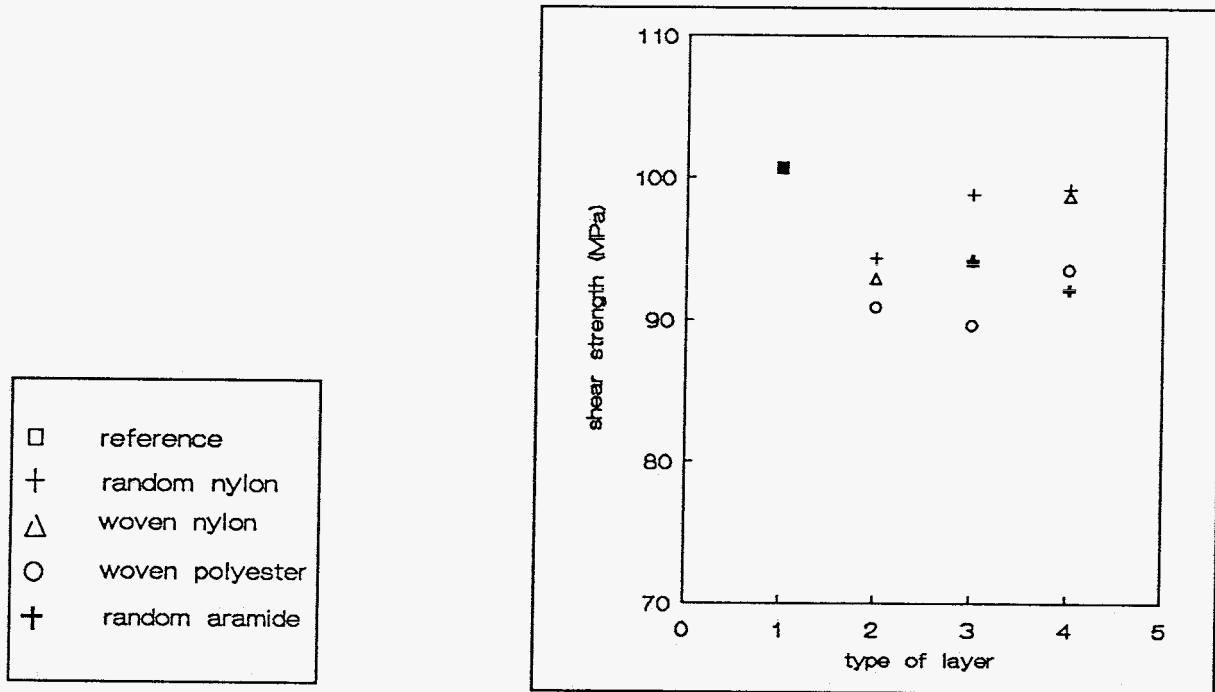
FIGURE 42: The shear strength of the dry specimen determined by the three-point bending tests (table 5) as a function of the different layers on the substrates:

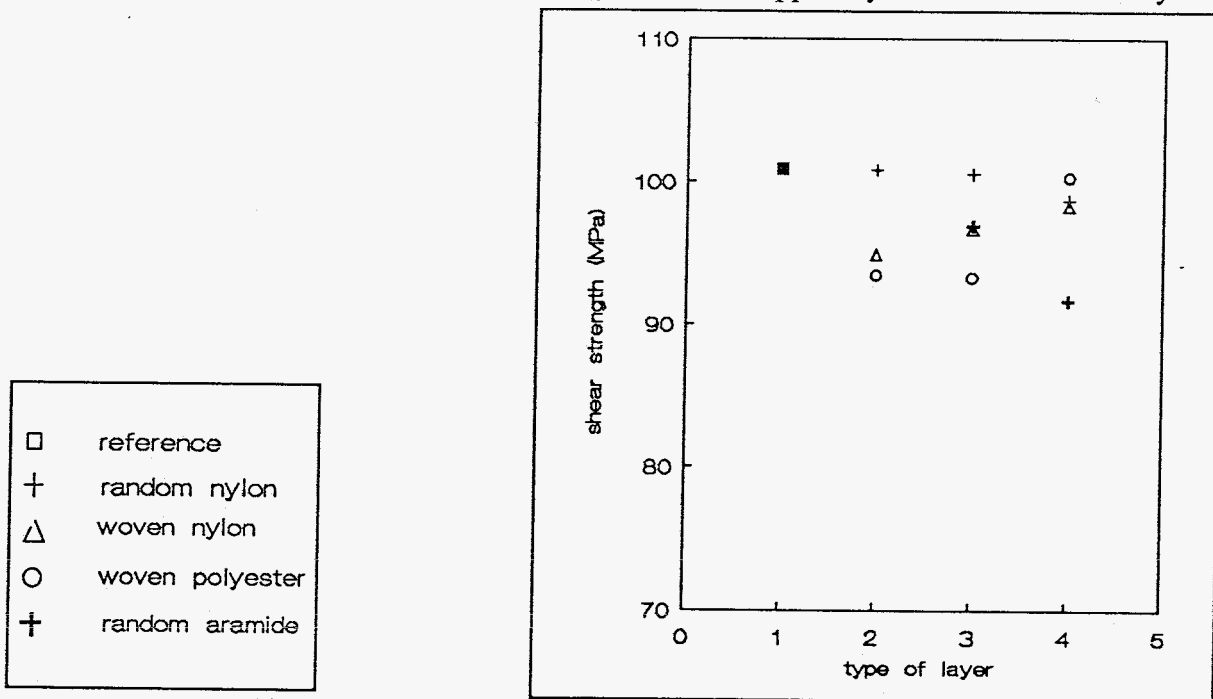1=reference   2=nickel layer   3=copper layer   4=no metal layer
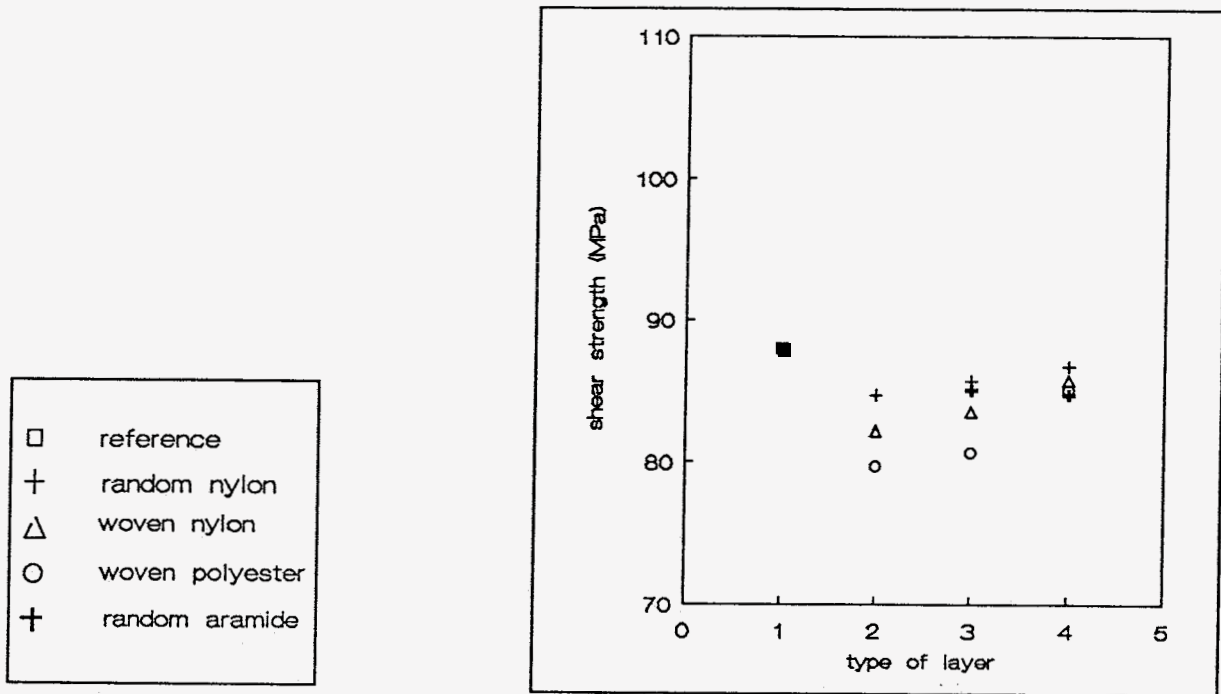

FIGURE 43: The shear strength of the dry specimen determined by the four-point bending tests (table 6) as a function of the different layers on the substrates:

1=reference   2=nickel layer   3=copper layer   4=no metal layer

FIGURE 44: The shear strength of the humid specimen determined by the three-point bending tests (table 7) as a function of the different layers on the substrates:

1=reference   2=nickel layer   3=copper layer   4=no metal layer