Preparation for An Experimental Study of Fractures in Gels

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Abstract

This paper describes the concept, design and execution of an experiment to study fractures in gels made of water, Laponite and NaCl. The behaviour of solutions made from these materials is dictated by a phase diagram where the concentrations of the respective constituents define which phase the solution is in. Two of four possible phases are gels, where one is isotropic and the other is nematic. The experiment is based on a peel-test like method. The experimental concept is that a strip of filter paper is placed on top of a Laponite gel, either in the nematic of isotropic phase, which is in the form of a horizontal column. The end of the paper is attached to a line which goes to a positioner $160mm$ above the experimental stand. The positioner is moved upwards at a constant velocity, which causes a fracture to move through the sample at a constant velocity. This fracture is then studied.

The experimental objective was to study the development of patterns on the fracture surfaces. Due to a limited time frame only one experimental session and two tests of the system were carried out. The low amount of experiments conducted and the currently underdeveloped technique for analyzing the fracture surfaces made the tests inconclusive, even though the observed fracture surface on one of the test samples showed a very interesting pattern. Further experiments with variations in fracture velocity and constituent concentrations may shed light on the current results.

Preface

This report is written for the required project in the ninth semester of the program for a Master degree in Physics (Sivilingeniør) at the Norwegian University of Science and Technology (NTNU). The project was done at the Complex Materials Group at the Department of Physics as a preparation for my Master Thesis, which will be done as a continuation of this project. The work on this project was done from the middle of September to the middle of December.

I would like to acknowledge Professor Jon Otto Fossum, my academic supervisor, for giving me the opportunity to do this interesting project. I am also grateful for the advice and support I have received from him throughout my work on the project. I am also indebted to Dr. Ahmed Gmira for advice and assistance. Furthermore, I would not have been able to carry any of my work without the help of Arnolf Bjølstad and the engineering workshop for fine mechanics. I am also grateful to my fellow students for many enlightening discussions and suggestions.

Trondheim, December 20th 2006 Christian A. Nielsen

Contents

List of Figures

Chapter 1

Introduction

Fractures have a great impact on the world and can cause benign damages, such as a broken ruler, or huge catastrophes, such as planes falling apart in mid flight or the collapse of buildings. For this reason the subject of fracture mechanics has been thoroughly researched for the last century. The main focus has however been on solid materials, while soft materials have received much less attention. The interest in fractures in soft materials has however increased over the last few decades, as many new soft materials such as polymers and plastics are used for an ever increasing range of applications.

One type of soft material is clays. Clays have been used by humanity for millennia for diverse tasks such as construction materials and pottery. Clays have also had an impact on peoples lives through mudslides and ground stability, and for the last 100 years clay has been studied by geologists and geophysicists in association with oil drilling and pre-building preparations at construction sites. More recently material physicists have taken an interest in clays due to their complex material properties and the growing availability of clean synthetic clays has made the study of clays an important part of modern materials science [1]. Thus it is a natural continuation of this trend to study the fracture properties of clay gels. The particular clay gel which is investigated in this project is made from water, NaCl and Laponite RD[1], a synthetic clay.

The main objective of this project was to design and assemble an experimental setup to investigate the fracture properties of Laponite gels. A secondary objective was to test the setup and start experiments. The experiment was designed to reproduce the peel-test like method used by Tanaka et. al. [2] to investigate fractures in acrylamide gels. The basic concept of this experiment is that a strip of paper is attached to the top of a horizontal gel column, the end of the paper is attached to a positioner a distance above the sample and that moving the positioner upwards at a constant velocity will cause a fracture to propagate horizontally through the sample at the same velocity. The individual pieces of equipment used for the experiment are described in Section 3.4.

As the fracture propagates through the sample it will create two mirrored frac-

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[1] For simplicity, Laponite RD will simply be referred to as Laponite in the rest of this text.

ture surfaces, one on top of the sample and the other attached to the strip of filter paper. The fracture surfaces may form patterns that reflect the mechanics of the fractures. Thus the fracture patterns, or the lack thereof, may provide useful insight into the material. The setup may also be used to study the propagation of the crack through the material. This may be done by placing a camera below the sample and filming as the experiment is conducted. Some more work on the experimental setup is however required before this becomes an effective tool.

The setup has so far been used for two tests of the experimental setup and one longer session of experiments. The tests were primarily conducted to test the experimental setup, though one of them did produce a rather interesting fracture pattern. Currently the process for preparing samples is somewhat complicated, and thus only one of four sessions to produce samples was successful. This session produced 6 samples, and 5 of these were used. The process of extracting information from the fracture surfaces did however turn out to be rather difficult due to the transparent nature of the Laponite gel, and thus the results are not as clear as it would be preferred. The process of recording and analyzing the fracture surfaces after the experiment has been conducted is the part of the experiment which is in the most urgent need of improvement.

Chapter 2

Theory

This is a brief presentation of some of the theoretical background of the project[1].

2.1 Fractures in Soft Materials

The field of fracture mechanics has been actively researched since the first world war, and is today one of the pillars of mechanical and civil engineering. It is also of great interest to physicists and material scientists. The main focus of fracture mechanics has however been on solid materials, and thus such materials have been the subject to the majority of the research on fractures. Fractures in soft materials, such as gels, is nevertheless a very important field of research, and a great deal of energy has lately been devoted to this subject by an growing number of researchers.

The basic principles of fractures in soft material is, like in solid materials, the growth of cracks due to a stress σ. The fracture is assumed to start at a crack tip where the stress is elevated above the average stress. Stress will build up around this crack tip and the material will eventually fail, allowing the crack to propagate through the material [3].

Force may be applied to a material in three basic ways to enable a crack to propagate. The most common crack mode is Mode I, which is an opening mode. For a Mode I crack, a tensile stress is applied normal to the plane of the crack. The second mode is Mode II, where a shear stress is acting parallel to the plane of the crack and perpendicular to the crack front. Mode III cracks are cracks where a shear stress acts parallel to both the crack front and the plane of the crack. Pure Mode I, II and III cracks are illustrated in Figure 2.1. Real cracks are seldom purely of only one mode. An actual crack may frequently have a combination of different crack modes at the same time, and the combination of modes may change as the fracture propagates [3].

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[1] The main focus of the project has however been the practical task of designing and assembling an experimental setup, and thus the theory will not be presented in depth in this report.

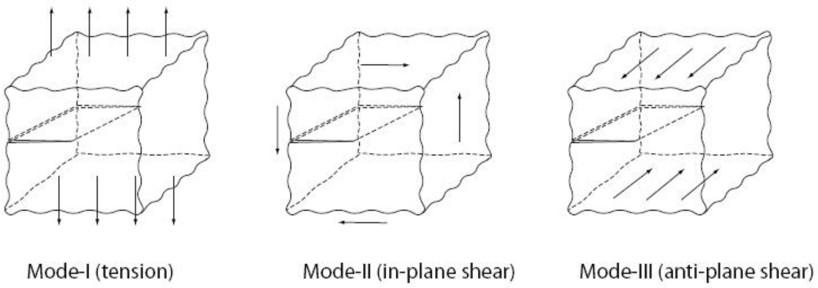

Figure 2.1: Modes of fracture [3].

As a crack front propagates through a material, it leaves behind two fracture surfaces. The crack fronts are not straight lines, but consists of many step lines. The actual fracture is thus an aggregation of crack segments. There is however little known about the fronts of well-developed cracks [4].

Tanaka et. al. [4] reported in a tree-dimensional, topological study on crack fronts in slow, brittle fracture of gels, that τ-shaped structures of step lines create an oblique pattern of lines on the fracture surfaces. These patterns are mirrored on the two surfaces created during crack propagation. The result of a fracture in a gel may therefore be a V-shaped line pattern. This was also observed by Tanaka et. al. in a later study [2]. These lines are similar to Wallner lines on fracture surfaces of glass, where the Wallner lines are locations “of the intersections between the crack front and fronts of stress pulses” [2]. The same study does however state that the lines in a gel can not be explained by the same mechanism as the Wallner lines. The cause of this step line pattern in gels is still not well understood.

2.2 Laponite

[2]Laponite is the most widely studied synthetic clay, and Laponite RD has a chemical formula $Si_8Mg_{5.45}Li_{0.4}H_4O_{24}Na_{0.7}$ [5]. This clay is a swelling 2 : 1 clay and like all other clays it has a layered silicate mesostructure. The general molecular structure of a swelling 2 : 1 clay is given in Figure 2.2. Swelling 2 : 1 clays consists of colloid particles with a thickness of $1nm$. These particles stack like a deck of cards and have a build up of electric charge on the surface. This charge is distributed so that there is a negative surface charge while the platelet edges have a smaller aggregation of positive charges. What makes Laponite a particularly interesting system to study compared with other clays is that the colloidal platelets of Laponite has a $25nm$ diameter mono dispersity. In most other clays, both synthetic and natural, there is usually a polydisperse distribution of platelets with a diameter of about a micrometer.

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[2] Unless otherwise specified, information provided in this section is from Fossum [1].

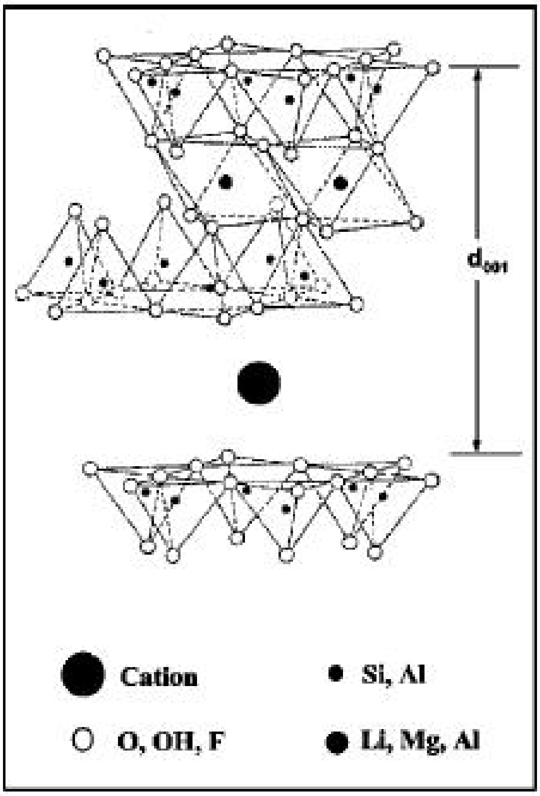

Figure 2.2: Idealized structure of a clay platelet. Exchangeable cations and water may occupy the interlayer region [6].

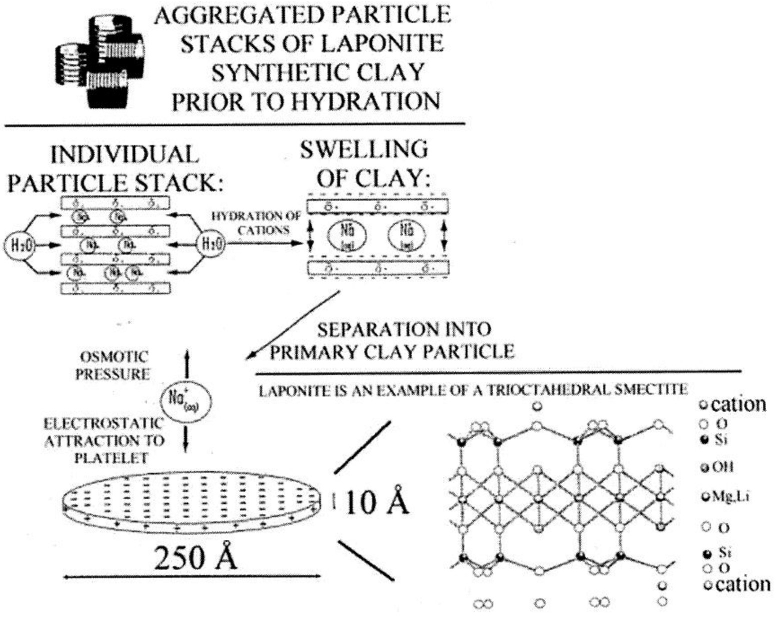

Figure 2.3: The structure of Laponite particles and the behaviour of Laponite platelets in an NaCl-H_2O concentration. Water seeps in between the individual Laponite particles in a particle stack and the ions in the water interact with the electrostatic charges on the surfaces of the Laponite particles [7].

2.2.1 The Phase Diagram for Laponite - Salt - Water Solutions

The pure form of Laponite is powder. This powder has the structure of a dehydrated mesoscopic layered system and when salt water is added to this system its behaviour will be dictated by a colloidal dispersion phase diagram, as shown in Figure 2.4. This diagram illustrates the behaviour of a system consisting of Laponite, salt and water. The behaviour of this solution is dependent on the concentrations of the respective components and may be split into four separate behavioural regions. These regions are the isotropic liquid (IL) region, isotropic gel (IG) region, nematic gel (NG) region and the flocculation (F) region. The reason for this behaviour is the competition between two different microscopic interactions between the individual clay platelets. One of the interactions is the van der Waals attraction. The other interaction is the repulsion between the negatively charged surfaces on the individual platelets and their interaction with the electrolytes in the salt in the water seeping between the platelets and larger particle aggregates, as shown in Figure 2.3. These interactions will then reach different local potential minima, which manifest themselves as different arrangements of the colloidal particles.

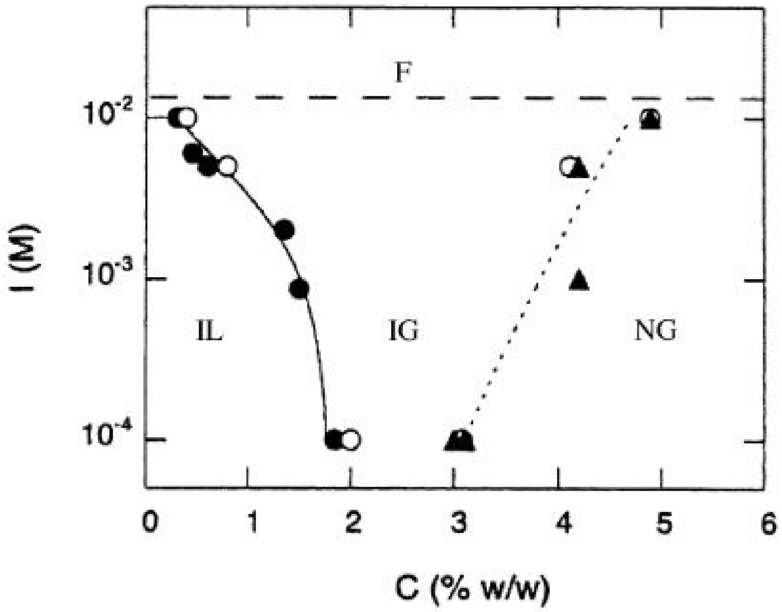

Figure 2.4: The phase diagram for Laponite and NaCl solutions. The respective concentrations of Laponite and NaCl determines the behaviour of the solution [1].

Isotropic Liquid

The IL region is a region in which smaller groups of platelets form Brownian aggregates, but these aggregates are not large enough to cover any significant portion of the system and thus the result is dispersed Brownian aggregates suspended in water. No gel is formed in this region, and there are no orientational order in the system as a whole. At the lowest concentrations the system behaves like a Newtonian fluid, but as the concentrations of either Laponite or salt approach the border toward the IG region the size of the aggregates increases and the behaviour of the fluid becomes more complex. As the combined salt and Laponite concentrations hit the line between the IL and the IG region, the aggregates become large enough for the aggregate structure to percolate. The nature of this transition is however not well known.

Isotropic Gel

The IG region of the phase diagram is the area in which the concentrations of clay and salt have become large enough for the mixture to set into a gel. There is still no global orientational preference for the clay platelets, but they are now locked in position in a larger system. According to some experiments the clay platelets form small stacks. These stacks form chains which bundle together and form a percolating structure.

When the concentration of salt or clay is large enough the system will start to order itself on a global scale. The individual platelets start to form more orientational uniform domains, which again will align themselves with the neighboring domains, creating a macroscopic orientational preference. There is however very little experimental data on this transition, and thus the current understanding of it is quite unclear.

Nematic Gel

After the transition from the IG region with increasing clay and salt concentrations the gel will move into the NG region of the phase diagram. The aggregates of platelets will arrange themselves in an ordered, nematic system in this region.

Flocculation Phase

In the F phase the salt concentration will be sufficiently high to break down the uniformity of the clay-salt-water concentration and cause the clay particles to sediment. In this phase the aggregates are no longer Brownian.

Chapter 3

Experiment

The objective of these experiments is to study fractures in Laponite gels. The experiment is carried out by placing a horizontal column of Laponite gel on a glass plate and making an initial notch at the desired starting point of the fracture in the manner described by Tanaka et. al. [2]. A strip of filter paper is attached to the top of the paste, and a line is attached to the filter paper. This line is attached to a VT-80 positioner placed approximately 1.6m above the clay. The positioner is controlled by computer through a Corvus control unit.

The positioner can pull the string upwards at constant velocity, which causes a fracture in the gel, as shown in Figure 3.1. The propagating fracture front and the resulting fracture surfaces may then be be studied. The fracture front may be studied by filming the propagation of the fracture with a video camera or a high speed camera, though a detailed study of the propagation of the fracture front is beyond the scope of this report. More importantly, the fracture surfaces may be studied by recording the fracture patterns in on the surface. For the duration of this project the recording of the fracture pattern was carried out by simple digital photography of the surfaces, though more elaborate recording techniques may be employed later, as described in Section 5.1.

3.1 Laponite Gel Samples

The Laponite gel samples are rectangular, transparent columns of gel. The base of a sample is $140mm$ x $20mm$ and the height is approximately $20mm$. The exact height may vary somewhat, as the sample solution is manually poured into the mould from the beaker it was mixed in. The final preparation of the sample before execution of the experiment involves removing the metal frame and placing a strip of filter paper on top of it. The removal of the metal frame involves careful cutting along the frame with a thin blade. This is done because the gel adheres slightly to the frame and thus removal of the frame will severely damage the sample unless the adhesion is neutralized. The cutting may however

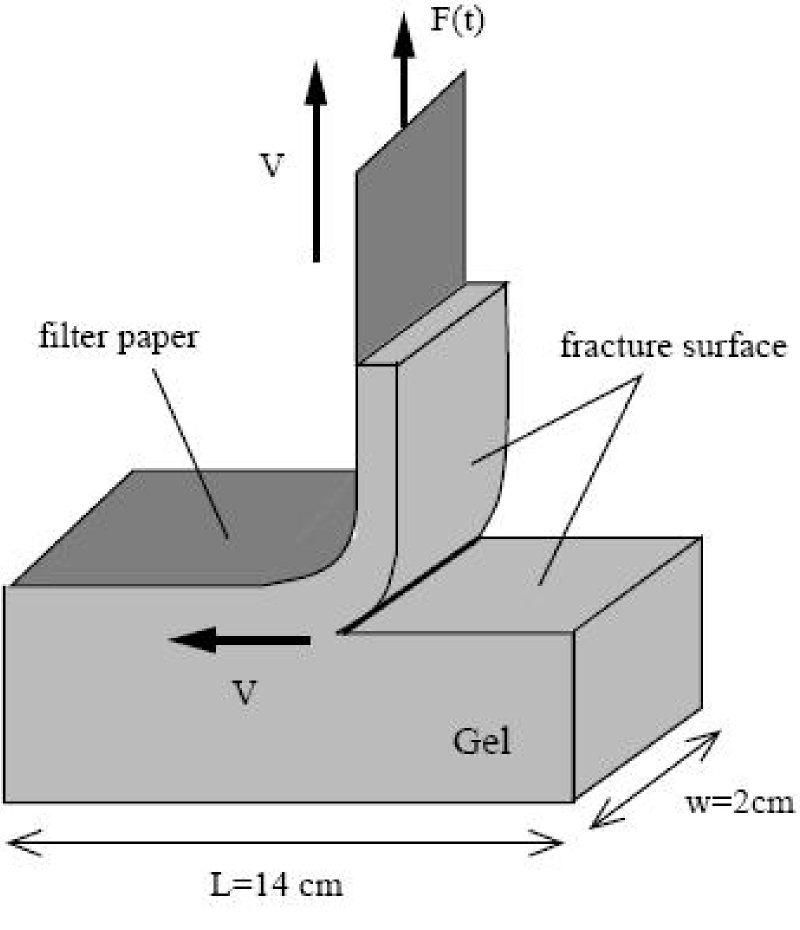

Figure 3.1: A schematic of a gel undergoing fracture. The filter paper is being pulled at a constant velocity v in the upward direction, causing the fracture to propagate at the same velocity v in the horizontal direction [2].

damage the edge of the gel slightly, and is thus not an optimal solution. Whether this has an impact on the fracturing of the sample remains to be seen.

Once the frame is removed from the sample, gravity will start deforming the sample. The speed at which this happens will however vary with the concentration of Laponite in the sample. It is therefore important to carry out the experiment immediately after the removal. The first step is to attach a strip of filter paper to the top of the gel. This is simply done by placing the strip on top of the gel to allow the adhesive process between the highly absorbent paper and the water rich gel to work for about 20 minutes. To speed up the process some slight pressure along the top of the gel may be applied, though this must be done extremely carefully to avoid causing damage to the fragile clay column.

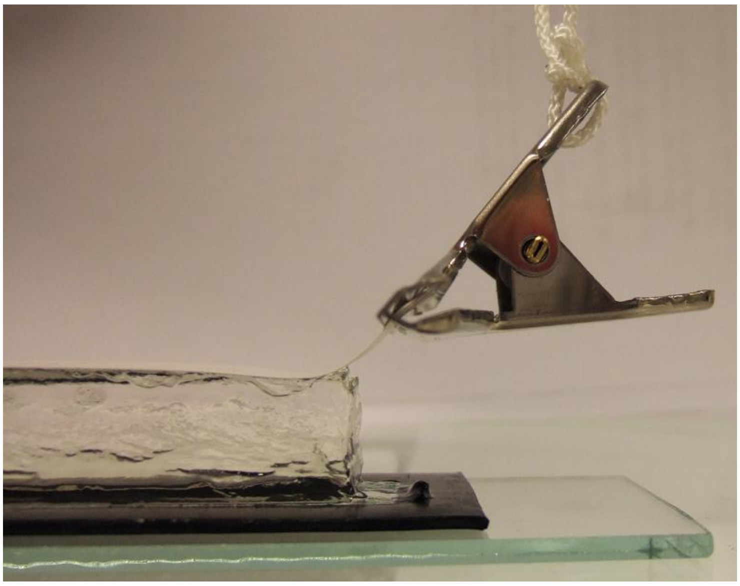

Figure 3.2: A sample clay column at the initiation of the experiment. The filter paper is attached to the top of the column and the end of the paper strip is held by a clamp attached to the positioner approximately $1.6m$ above. There are some slight traces of damage along the sides of the sample from the severing of the adhesion to the frame.

3.1.1 Sample Preparation

The preparation of samples is a process involving a few hours of work and approximately 10 days of waiting for the sample to rest properly, as done by Cousin et. al. [8]. The starting point for the process is mixing a 1% mixture of Laponite and distilled water. The first step of the mixing is carried out by pouring the desired amount of distilled water into a large beaker, dropping in a magnetic rod for the magnetic stirrer and placing the beaker on the stirrer. Thereafter the appropriate amount of Laponite powder is weighted on a Ohaus Navigator N30330 electronic balance and slowly poured into the beaker with distilled water as the water is being stirred. As a numerical example one might mention that the first set of samples prepared was made in a batch of $1000ml$. This required $10g$ of Laponite powder.

After the Laponite powder is poured into the beaker the mixture will at first be white and have a very low degree of transparency. This is because the powder is not yet dissolved and over time the mixture will become more transparent as the powder mixes better with the water. One to one and a half hour later the solution will be transparent and basically look like ordinary water. The solution is then ready for the next phase of the sample preparation, which is filtering.

The desired Laponite concentration will usually be higher than 1%, but it is an unfortunate complication that the samples have to be mixed at an initial concentration of 1%. This is because the Laponite solution needs to be filtered, and higher concentrations of Laponite causes the solution to be to thick to pass through the filter paper. The filtering is done to remove larger aggregations of Laponite particles to ensure uniformity in the solution, and is done with a filter unit attached to a suction pump to ensure flow through the filter. The filter is a filter paper with pores of diameter $0.8\mu m$ and is discarded after one use.

Once the filtering is complete the next step is to increase the Laponite concentration to the desired level. This is done by evaporation. The Laponite particles are too heavy to evaporate and thus only the water will evaporate from the solution. This naturally increases the Laponite concentration. It is important that the evaporation process takes a short amount of time, as the mixture will start to set when the concentration of clay increases from the isotropic liquid into the isotropic gel phase. The solution does however use some time to set, and thus a rapid evaporation process may allow the solution to gain concentrations well above the transition point. As an extra precaution, salt is not added to the mixture until after the evaporation process is complete, as the required concentration of clay for the transition into the isotropic clay phase decreases as the salt concentration is increased, according to Figure 2.4. An added bonus to postponing the addition of salt to the mixture is that this significantly simplifies the calculation of the amount of salt required in the mixture. This can now be calculated from the final volume of solution, and it is not necessary to compensate for an increase in the salt concentration as a result of the evaporation.

Evaporation is most efficiently carried out by pouring the solution into a container that gives the solution a large surface area and increasing the temperature significantly. Both steps will cause the water to evaporate faster. The container used for the evaporation process is a simple fire proof lasagna pan. The initial solution is poured into this pan to a depth of a few centimeter, with the depth depending on the desired amount of the final sample solution. It is however important that the depth of the solution never decreases down to a few millimeters, as this seems to cause the Laponite particles to aggregate locally and cause the solution to rapidly form local areas that starts to form gels. This was experienced several times during the first attempts at making samples.

The concentration of Laponite is monitored by weighing before, after and during the evaporation process. The weight of the pan is first recorded, and then the solution is poured in. After the initial weight of the solution has been determined by subtracting the weight of the pan from the total weight of the pan and solution, the mass the solution has to be reduced to in order to reach the desired concentration is calculated by (3.1).

$$V_{target} = \frac{\text{initial volume}}{\text{desired concentration}} = \frac{V_0}{C_{target}} \tag{3.1}$$

C_{target} is the desired concentration of Laponite and V_0 is the initial volume of the solution. The pan with the Laponite solution is then placed in a heating cabinet where the temperature is increased to $100°C$. The pan is left in the cabinet for a few hours to reach a concentration near the target concentration, and is occasionally taken out of the heating cabinet to be weighed. When the weight of the pan and solution is sufficiently near the desired weight, the pan is permanently removed from the cabinet and the contents are poured back in a large beaker. The beaker in then covered with cling film to prevent further evaporation and placed on a magnetic stirrer to prevent the solution from setting. The exact concentration of Laponite in the solution after the evaporation process is then determined by (3.2). The first successful attempt took approximately 2.5 hours and had a target density of 2.5%. It reached about 2.3%.

$$C = \frac{\text{initial volume}}{\text{final volume}} = \frac{V_0}{V_{final}} \tag{3.2}$$

The next step is to add salt to the mixture. NaCl was used in this experiment. The desired concentration of salt, I_{target} is used to calculate the mass of salt that should be added, according to (3.3), where V is the volume of the Laponite solution and M_{NaCl} is the molar mass of NaCl.

$$m_{target} = I_{target} V M_{NaCl} \tag{3.3}$$

This amount of NaCl is then weighted at a high precision balance like the Ohaus Analytical Plus AP-250D. The high precision is required because the amount of salt to be added is in the order of milligrams. This does however make it nearly impossible to get the exact target amount, and thus the actual acquired salt concentration I must be calculated. I is found by (3.4), where m is the actual amount of salt added to the solution. The salt is poured into the mixture while it is still on the magnetic stirrer. The new mixture is then stirred for another half hour to make sure the salt is mixed evenly into the solution.

$$I = \frac{m}{V M_{NaCl}} \tag{3.4}$$

The moulds are prepared while the salt is stirred into the solution. The complete mould consists of a metal frame, a rubber isolation seal and a glass plate. These individual parts are first washed in distilled water and then put together according to Figure 3.3. In order to keep pressure on the seal to keep the Laponite solution from seeping out, the frame is firmly pressed against the seal and glass plate by applying packing tape around the frame and glass plate. Finally the solution is poured into the moulds, which then are placed inside a plastic storage container. This container is firmly shut and kept at a higher level of humidity by placing an open container with a K_2SO_4 solution inside it. The high humidity keeps the water in the sample solution from evaporating and will thus conserve the prepared concentrations of Laponite and NaCl. Another reason for preserving the samples in the container is to keep them from breaking as they set. The water in the samples will continue to evaporate even after the samples have set properly if the samples are kept at the room humidity level. This would cause the sample gels to break as they set, as the clay will adhere to the frame, causing the center of the sample to be torn as the gel contracts because its mass is reduced due to the evaporation of water.

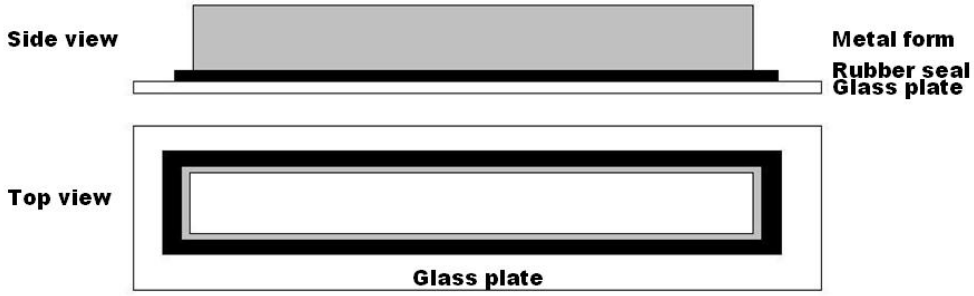

Figure 3.3: The complete mould for the setting of the Laponite gels for the experiment. A rubber seal (black) is placed between the glass plate and the metal frame (grey). The prepared experimental solution is then poured into the frame, and after about a week the samples are ready for the experiment.

The samples are then stored in the container for about ten days. This will allow the samples to set properly into proper gel columns. When the samples are ready for the experiment they will be removed from the storage container and placed on the experimental setup. The tape pressing the frame against the seal and glass plate will be removed, and a thin blade will be used to carefully sever the gel from the metal frame. When the gel no longer adheres to the frame, the frame will carefully be removed from the sample. Now only the gel column and seal will remain on the glass plate, and the sample is ready for the experiment. The experiment is however required to be initiated quite soon after the frame is removed from the sample, as the samples will start to loose their shape and float outwards soon after the frame is removed. This is particularly a risk with the samples with lower concentrations of salt and Laponite.

3.2 Method

The experiment is based on a peel-test-like method, based upon the method used by Tanaka et. al. [2]. The objective is to peel off a thin strip from the top column of Laponite gel in order to study the fracture between the gel column and the peeled layer. In the long term the objective is to study both the propagation of the fracture front and the fracture patterns on the fracture surfaces, but due to the limited scope of this initial project the particular short term objectives are to assemble a working experimental setup and acquire some initial data on the fracture surfaces.

The basic idea of the experimental setup is very simple. A Laponite gel is placed on transparent glass plate, after which a strip of absorbent, yet robust filter paper is put on top of the gel. After a short period of time this paper will be attached firmly to the top of the very aqueous gel. The paper is then attached very carefully to a line with the help of a clamp. The line is attached to a positioning unit (200mm VT-80) approximately 1.6m directly above the sample. When the positioner moves upwards it will generate a pull directly in the vertical direction. Due to the distance between the sample and the positoner, the pull on the filter paper will always be approximatively directly upwards. As the filter paper is pulled upwards, the angle between the part of the filter paper free from the clay gel and the part attached to it will be about 90°. Moving the positioner vertically will cause a fracture to propagate horizontally along the clay column. An initial notch in the end of the sample will give the fracture a good starting point.

Thereafter the positioner will be moved upwards at a constant velocity. This will cause the fracture to propagate horizontally through the clay column at a constant velocity.

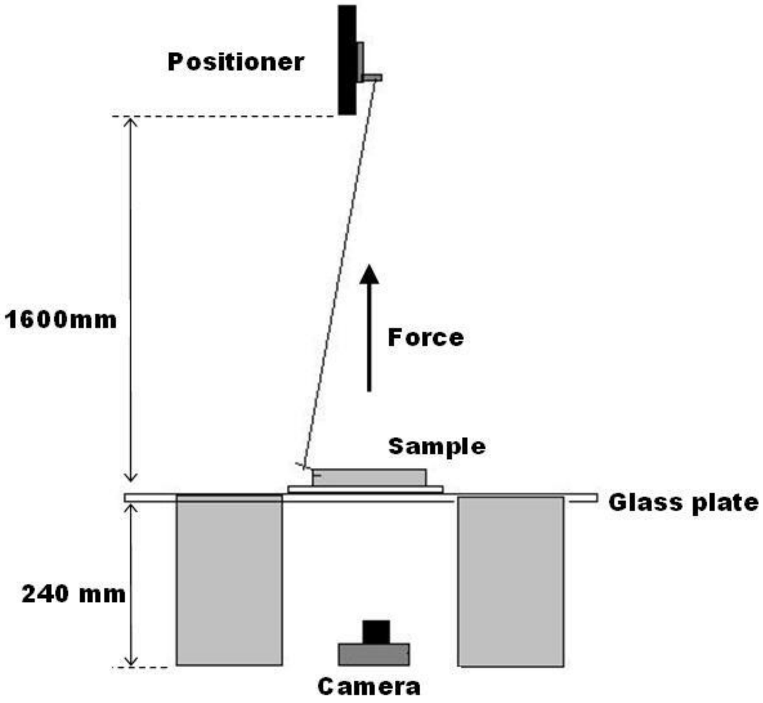

Figure 3.4: An illustration of the experimental setup. The VT-80 positioner at the top of the illustration pulls the line upwards, causing the strip of paper attached to the top of the sample to be pulled upwards, which again causes a fracture front to propagate horizontally along the length of the sample. As the fracture progresses through the sample it is filmed by a camera below the sample.

The fracture of the gel will be recorded as it progresses, as a video camera is situated directly below the sample. This camera films up through the gel, and this footage may hopefully eventually be used to analyze the propagation of the fracture front. The experiment is also being recorded using an ordinary digital camera, though this is first done after the experiment has been performed. Once the fracturing process is complete, the sample is moved to another surface and photographed. This is to record the fracture patterns in the surface. The pictures are taken both on a black and a white background with light sources at different positions around the sample. The pictures are taken directly above the sample from distances ranging from $10cm$ to $30cm$. The use of several backgrounds and light settings is due to the transparent nature of the gel. This transparency makes it very hard to get a good picture of any pattern that may have formed on the fracture surface, and therefore it is necessary to try many different approaches to get a good photo. However, this is not a good longterm solution, and it is difficult to obtain good, consistent and scientifically viable data on the fracture patterns this way. Thus an alternative solution should be sought at the first opportunity.

The pictures of the surfaces may be studied qualitatively after the experiment is done. However, these photos do not at the present time provide a satisfactory mean for a proper study of the fracture patterns, and further development of the techniques for recording and analysis of the surfaces is required.

3.3 Experimental Work

Due to time constraints, the work done this semester was primarily on the design, construction and assembly of the experimental setup. There was however time to carry out some tests and preliminary experiments in late November and early December. This work consisted of two initial tests of the system by using quick-setting gels to quickly produce samples of low scientific value due to the rough nature of their preparation, several attempts at producing scientifically satisfactory samples and one session of experiments on the one successfully produced batch of samples.

3.3.1 Sample Preparation Sessions

In total four attempts were made to create experimental samples. Only one was successful. The other attempts failed due to inexperience and flaws in the sample preparation technique, though they provided valuable experience with the process and yielded several ideas for improvement.

First Sample Preparation Session

The first unsuccessful attempt at creating samples failed in the evaporation phase of the process for preparing samples. In the first attempt the main problem was the trays that originally were intended for the evaporation process. These were simple fire-proof baking trays, which were thought to be suitable for the task due to their large surface area and their resistance to heat. These

Figure 3.5: A sample still in the mould. The tape presses the metal frame firmly against the rubber seal and glass plate underneath to prevent the solution from leaking out.

trays did however turn out to be quite unsuitable because of their low edges. The low edges created two very unfortunate complications. The first problem was simply that the trays were very difficult to carry without spilling some of their contents. This proved to be a serious problem, as the only way to monitor the concentration of Laponite in the solution during and after the evaporation process requires carrying the sample tray to an electronic balance and calculating the new concentration from the change in the weight of the solution. Thus even a spillage of a small percentage of the solution would make it impossible to calculate the exact concentration of the solution after the evaporation process. An additional problem would simply be that a loss of a portion of the sample could easily lead to that the process would not yield a sufficient quantity of sample material to fill all the moulds, thus decreasing the amount of complete samples created in a single batch and thus slowing the progression of the whole research project.

The second problem caused by the low edges was that only a limited amount of the initial solution could be poured into the trays to prevent spillage and thus the depth of the solution in each tray would have to be very low. This turned out to be a serious problem, as a significant percentage of the solution is supposed to evaporate. The low depth of the solution and the slightly uneven nature of the bottom of the trays then caused so much of the solution to evaporate at some points that the solution started to coagulate over most of the bottom of the trays. As the solution ended up setting before it was poured into the moulds this attempt at making samples was a failure, and the whole process had to be restarted. Due to the experiences from the evaporation process and the inadequacy of the the fire proof tray, a fire proof lasagna pan with higher edges was acquired as replacement for the trays.

Second Sample Preparation Session

The second attempt was however successful. In order to initiate the experiments with samples with concentrations from the centre of the phase diagram, illustrated in Figure 2.4, the intended concentrations for this batch of samples

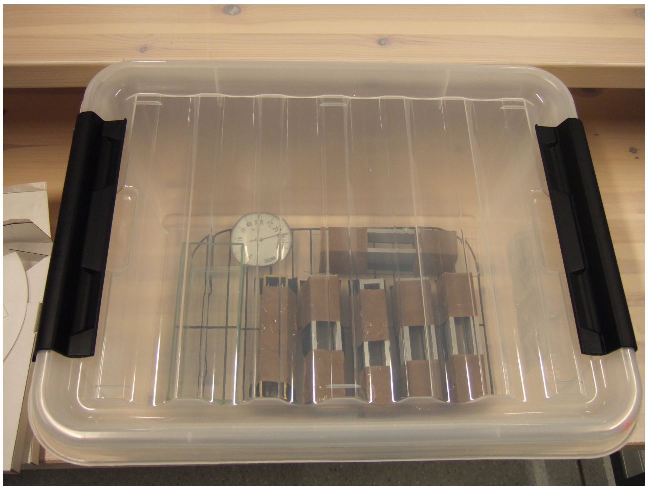

Figure 3.6: The storage box with sample moulds. The sauna hygrometer is visible on the left.

were $C = 2.5\%$ and $I = 10^{-3} mol/l$. The initial mixture with 1% Laponite consisted of $1l$ water and $10g$ Laponite. The mixture was stirred for for about an hour before it was filtrated and placed in the heating cabinet for 2hours and 20 minutes. It then had a Laponite concentration of 2.24%. According to (3.3), $2.637 \cdot 10^{-2}g$ of NaCl was to be added to reach $I = 10^{-3} mol/l$. However, the eventual amount of NaCl added was $2.664 \cdot 10^{-2}g$, which resulted in $I = 1.01 \cdot 10^{-3} mol/l$. The slight deviation was due to the difficulty with accurately preparing masses in the magnitude of 10^{-4} grams. After being stirred for about an hour on a magnetic stirrer, the solution was poured into the moulds and placed inside the sealed storage container. A sample mould is shown in Figure 3.5 and the storage container with samples inside is shown in Figure 3.6. The sauna hygrometer shown in the upper left corner of the container makes it possible to monitor the humidity inside the container.

Third Sample Preparation Session

Though the second attempt at creating a batch of experimental samples was highly successful, this was unfortunately not the case for the third attempt. The intended objective for this batch of samples was a Laponite concentration of $C = 3\%$. There were two reasons for this choice of concentrations. The Laponite concentration was to be increased in order to test whether samples of a Laponite concentration of this magnitude could be created with the current sample preparation technique. Furthermore, if the sample preparation was successful, the samples would be well suited for a test to determine whether a higher Laponite concentration would set into a Laponite gel that was more suitable than the last batch of gels for fracture experiments in the current setup. The salt concentration was to be kept at the same level as for the previous batch in order to limit variation of parameters and allow for an isolated investigation of the experimental impact of the Laponite concentration.

The mixing of the initial sample solution was done with $1.5l$ of water and $15g$ Laponite. The solution was mixed for about 2 hours in a beaker covered in cling film before the solution was filtrated. The filtration went smoothly and the solution was then poured into the large lasagna pan and weighted on the Sartorius balance. The pan was then placed into the heating cabinet, which was set to $100°C$. The pan was left in the cabinet for 2.25 hours before it was checked on. The mixture seemed to be fully liquid at this point, and by weighting the pan and applying (3.2), the concentration was determined to be about 1.7%. 45 minutes later the solution was at approximately 2.25% and the solution was still fully liquid. There were however some slight indications of coagulation within $5mm$ of the edges of the container, though no large scale setting. The solution was checked on again 35 minutes later. At this point the solution had at some points coagulated into large floating flakes of gel. The concentration was calculated to be 2.85%.

Because a large amount of the solution at this point had set into a gel-state the solution would not be usable in its current condition. As a final attempt to salvage the samples it was attempted to return the solution to a fully liquid state. The solution was immediately poured into a beaker and placed at a magnetic stirrer at maximum power, but the whole mixture set into a firm gel while being stirred, effectively locking the magnetic bar in place. Thus the third mixing session was unsuccessful in creating experimental samples. The session did however underline the most significant weakness of the sample preparation process, the evaporation process for increasing the concentration of Laponite in the sample. The complications experienced in the first unsuccessful session to prepare samples were also associated with this phase of the preparation process.

Fourth Sample Preparation Session

As the previous attempt at creating experimental samples was unsuccessful, it was thought best to try to duplicate the Laponite concentration from the successful sample preparation session. That batch of samples had $C = 2.24\%$. An initial mixture of 1% Laponite was made from $1l$ of water and $10g$ of Laponite. The solution was mixed for about an hour and then poured into the fire-proof tray and placed in the heating cabinet at $100°C$. The tray was left in the cabinet for 2hours and 20 minutes and the final concentration was $C = 2.31\%$. There were no indications that any of the solution had set into gel.

As the attempt at increasing the Laponite concentration was unsuccessful, it was decided to increase the salt concentration while keeping the Laponite concentration at approximately the same level. The intention was to see if a higher salt concentration would produce a gel which was easier to utilize for the experiment. It was decided to try a salt concentration near the phase boundary to the flocculation phase, and thus the concentration was set to $I = 10^{-2} mol/l$. This required mixing $m_{NaCl} = 0.2483g$ into the $0.4249l$ of solution, as specified by (3.3). The NaCl was mixed into the solution while it was being stirred on an magnetic stirrer.

Shortly after the salt had been poured into the solution there was marked increase in the viscosity of the solution. The power of the magnetic stirrer was turned to maximum, yet after 20 minutes the entire solution had become a solid gel with the magnetic stirring bar locked in the middle. It would appear that a concentration of 2.31% Laponite and $I = 10^{-2} mol/l$ creates a mixture that sets quickly into a very firm gel. Thus this session did not yield samples suitible for the intended experiments. It did however provide some useful insights, such as the potency of salt in the mixture.

From the experience of the four attempts at creating samples it would appear that evaporating water from the solution to increase the Laponite concentration works for creating concentrations of less than about 2.5%. For higher concentrations it would appear that either the high concentration of Laponite, the amount of time the mixture has to stand still in the heating cabinet or a combination of the two causes a rapid gelation process which renders the solution unsuitible for the intended experiments.

3.3.2 Initial Tests of the Experimental Setup

Two proper tests of the experimental setup were conducted to test the equipment before the main experiments were started[1].

Initial Test Samples

For both the test sessions a strong, unfiltered solution of 3.5% was used to produce test samples. As the filtering process requires a 1% concentration, a 4% solution would have to go through a long evaporation process in order to attain a concentration of this magnitude. Thus the solution was unfiltered because this allowed for the immediate production of strong Laponite concentrations. There may however be some slight differences in behaviour between filtered and unfiltered solutions. In the unfiltered solution some of the aggregates of particles from the original Laponite powder have not dissolved completely. A filtration will completely dissolve the aggregates, causing a change in the behaviour of the solution [9]. Whether this change of behaviour has an impact on the fracturing of the gel has yet to be determined.

These solutions set into a firm gel in a few hours and the gel samples were used for experiments within 24 hours of the mixing.

Testing the Experimental Setup

The first experiment on a test sample was carried out on a sample with a Laponite concentration of 3.5%. The sample was placed on the glass plate below the VT-80, and the metal frame was removed from around the clay column. Before the frame was removed, the clay column was severed from it by carefully cutting along the inside of the frame. In this case a scalpel was used. The blade

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[1] In addition several smaller tests were carried out for such purposes as finding the appropriate paper to attach to the top of the sample clay columns and finding finding the best design of the sample moulds. These small tests were carried out to solve small practical problems with the design of the experiment and will not be described.

of the scalpel was however slightly too short, and the hilt of the scalpel pushed the blade slightly away from the frame. This led to some light damage of the sides of the clay column, and later a longer blade would be used in order to avoid this sort of damage.

After the frame had been removed, a strip of Whatman Type 4 filter paper was placed on top of the sample and left there for 15 minutes to allow it to adhere properly to the gel. The end of the paper was then attached to the line to the positioner, a small incision was made at the desired starting point for the fracture, and then the positioner was moved upwards at a velocity of $1mm/s$. Unfortunately the strip of paper came loose from the top of the gel column and no fracture formed. The process was then performed again at the other end of the sample column. The process was speeded up by increasing the velocity to $5mm/s$. This time a fracture was created and started to propagate along the sample. Unfortunately the fracture seems to have propagated slightly upwards, and thus reached the top of the column after a few centimeters. Thereafter the paper was simply torn from the top of the column.

As a last attempt at attaining a good fracture in this sample, another experiment was made on the sample after cutting off some of the damaged material. Thus a new , clear and undamaged end was once again achieved, and the experiment was conducted again at $5mm/s$. This experiment went very well, and the fracture propagated all the way across the sample. The fracture surface also showed a clear pattern. This will be described in Section 4.1.

The second test of the experimental setup was also carried out on a sample with a Laponite concentration of 3.5% There was however a slight leak from the mould, so the gel column was nearly centimeter lower than it was supposed to be. The experiment was nevertheless carried out as planned, as the main purpose of this particular experiment was to test the experimental setup and to gain experience. The the gel was cut free from the frame without damage, as a longer blade was employed. Thereafter a strip of filter paper was put on top of the gel column. Some damage may have been caused to the structure of the sample as some pressure was applied to the top of the sample as the paper was placed on it. The samples are quite soft, and only a small amount of force needs to be applied before they start to lose their shape. The experiment was conducted twenty minutes after the paper was attached to the sample.

The fracture velocity for this execution of the experiment was set to $1mm/s$. The first attempt ended after only 3 centimeters when the fracture propagated to the top of the sample where the filter paper started to slip off the top of the column. The experiment was then repeated at the other end. This attempt was more successful, as the fracture propagated a distance of $7cm$ before the paper started to slip from the top of the sample. There were however no obvious patterns on the fracture surface.

3.3.3 The First Experimental Session

6 samples were prepared for the first proper experimental session, as described in Section 3.3.1. These samples all had a Laponite concentration of $C = 2.24\%$ and a NaCl concetration of $I = 1.01 \cdot 10^{-3} mol/l$. The objective of the tests was

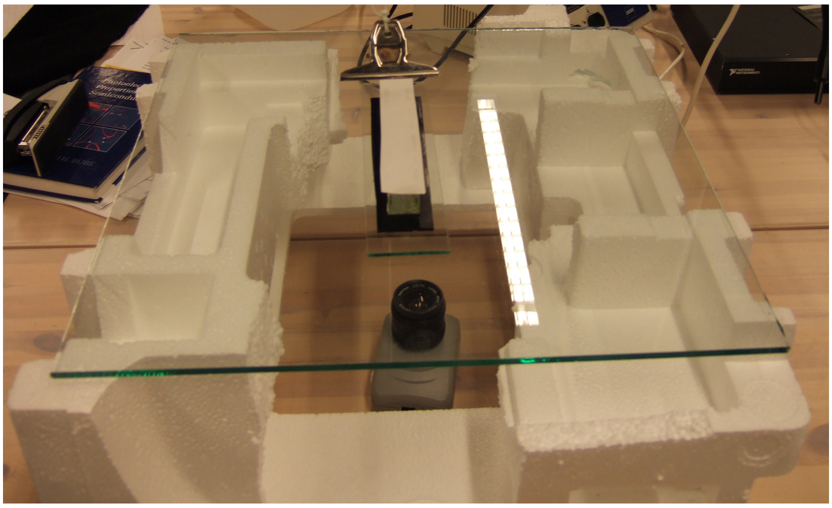


Figure 3.7: The experimental stand. The sample is placed directly above the PixeLink Firewire camera below the glass plate. The VT-80 positioner is 1.6m directly above the sample.

to investigate the behaviour of the identical samples for 3 different fracture velocities. Two samples would be tested at each velocity to test the reproducibility of the results. The velocities spans the orders of magnitude of velocities investigated by Tanaka et. al. [2].

All of the samples were prepared for the experiment in the same manner. They were placed at the experimental stand where the metal frames were removed and strips of filter paper were placed on top of them. An incision would be made at the desired starting points of the fractures approximately 20 minutes after the strips of filter paper had been placed on top of the samples. The experiments were conducted immediately after the incisions were made.

Sample 1 and 2

The first two samples were to be tested at velocity $v = 0.1mm/s$. Sample 1 did not yield any results, as the filter paper repeatedly came loose from the top of the gel column, and thus no clear fracture patterns appeared in the material. A similar effect was observed in the second sample. In the first sample it would appear that the initial incision at the end of the column simply was ignored and a slight fracture was created within a millimeter from the filter paper. For sample 2 a fracture started at the initial incision before moving in an upwards direction toward the filter paper and then following the pattern of sample 1, with the paper occasionally slipping from the surface in conjunction with creating a small fracture very close to the filter paper. Thus it is very hard to distinguish any patterns on the fracture surfaces, because the there is a piecemeal variation along the column between areas where a fracture occurred and areas where the paper slipped off the top of the gel. On sample 1 slipping seems to have been the most frequent effect. Sample 2 did however experience a fracture over most

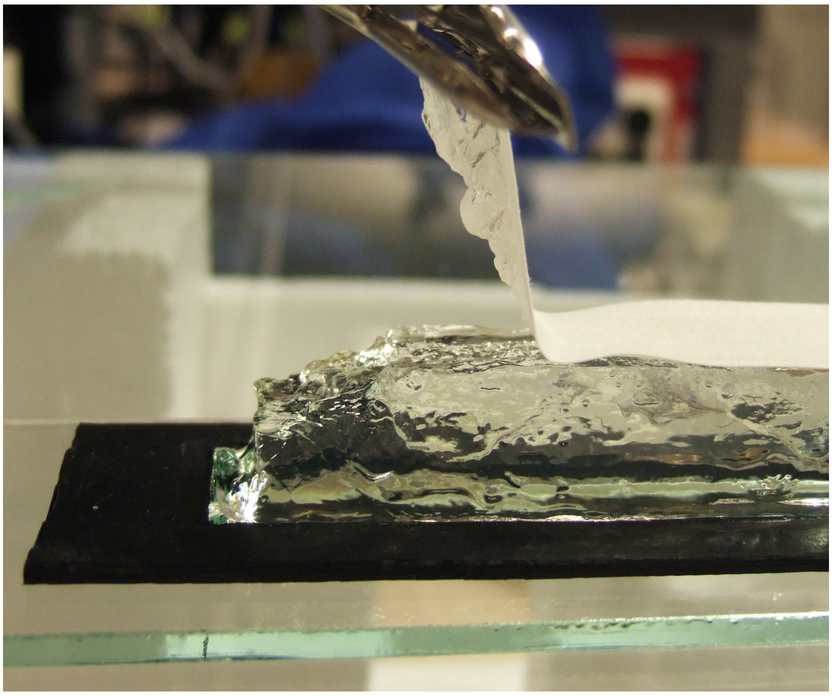


Figure 3.8: A sample where the fracture have moved toward the strip of paper directly after the experiment has started. The fracture moves somewhat upwards directly after the initial incition. In this case the paper varied between slipping on the sample surface and pulling off pieces of the sample surface.

of the sample, and thus it will be discussed in Section 4.

Sample 3, 4 and 5

The next two experiments were carried out at $v = 1mm/s$. Sample 3 behaved in about the same fashion as sample 2, though in this case there was no sign of the paper slipping from the top of the gel column.

Sample 4 was unfortunately ruined during the preparation of the experiment, so it was decided to use sample 5 for the second experiment at $v = 1mm/s$. This sample was unfortunately also damaged during the preparation of the experiment. The black rubber seal between the glass plate and the metal frame was so strongly attached to the metal frame that it was dragged off the sample together with the frame. This caused some damage to the sides of the gel column, though the experiment was nevertheless carried out. The value of the results may however be somewhat diminished as it is possible that the damage to the sample may have slightly changed its behaviour. As the experiment was carried out the fifth sample behaved in a manner similar to that of sample 3.

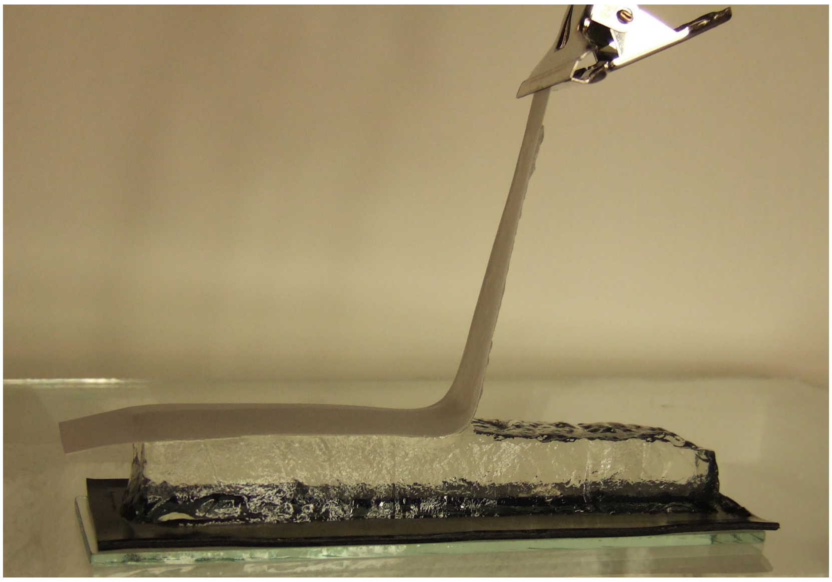


Figure 3.9: Sample 3 halfway through the peel-test experiment. The fracture started in the initial notch in the lower area of the gel column on the far right, before propagating slightly upwards toward the strip of filter paper. Near the filter paper the fracture once again started to propagate parallel to the strip of paper.

Sample 6

The last sample was tested at velocity $v = 5mm/s$. There was no major differeces in behaviour between this and the previous samples. The fracture started from the initial notch, propagated upwards until it was within $1 - 2mm$ from the strip of paper and then propagated along the paper. There were also some areas where the paper slipped from the top of the gel sample. This was clearly observed as some smaller areas of the strip of paper did not have any gel adhering to it after the experiment had been conducted, and thus these areas must have slipped from the gel surface.

3.4 Equipment

A wide range of equipment was used to carry out these experiments. Here is a short presentations of the individual parts.

VT-80 Positioner

The positioning device used for the experiment was the translation stage 200mm VT-80 from Micos Gmbh. The unit is based upon a back-lash compensated lead screw which produces quiet smooth movement through a DC-, 2-phase-micro-step (SMC-series) motor. The accuracy per 50 mm is $+/-100\mu m$, with a path flatness of $+/-20\mu m$ and a velocity range from 0.001 to $20mm/s$. This positioner can pull loads up to $5kg$ and is controlled by computer through a Corvus SMC controller [10].

Figure 3.10: Translation Stage VT-80 positioner [10].

Corvus SMC Controller

The VT-80 positioner is controlled through a Corvus SMC controller from Micos Gmbh. This unit can be controlled through a computer by the use of the ASCII command language Venus-1. This may be used through either Micos's software Winpos, Labview or Hyperterminal. In this case Winpos was used. The Corvus can control up to three positioners and has a 133 MHz RISC Processor, with Flash-Memory and several alternatives for computer interfacing [10]. In this case the RS-232 serial port was used.

Figure 3.11: Corvus SMC controller [10].

Cameras and Lenses

A PixeLink PL-A642 camera was used to film the fractures as they occur. The PL-A642 is a computer controlled megapixel colour camera and is based on a 2/3 inch CMOS Image Sensor. This camera connects to a computer through a single 6 pin IEEE-1394 Firewire interface connector and is fully controllable from the connected computer. The camera has a mount in front of the image sensor where either the camera may be mounted on a microscope or a lens may be mounted on the camera [11].

In this case a Navitar CCTV 8mm lens was mounted on the camera in order to film the fracture at close range. This lens may be focused from $0.2m$ to ∞ and allows for adjustment of the brightness.

The experiments were also photographed with an ordinary digital camera. A Fujifilm Finepix F11 6.3 Megapixel camera was used for this purpose.

Figure 3.12: PixeLink Firewire Camera and Navistar 8mm lens. The scale is in centimeters.

Equipment for sample preparation

The samples are mixed in a measuring cup which is placed on a magnetic stirrer. Several different stirrers were used for different samples, though it is unlikely that this has any effect on the prepared samples. The magnetic stirrers used were the Schott SLK3, the Heidolph MR1000 and the Heidolph MR3001 Magnetic Stirrer Hotplate.

Weighting was done also done at several different electronic scales. The Laponite for the samples were measured at a Ohaus Navigator N30330 with a division $d = 0.002g$. The measurement of NaCl for the Laponite samples required high accuracy, and thus a high accuracy scale was required. The balance used was a Ohaus Analytical Plus AP-250D, which has an accuracy of $0.01mg$ with a standard deviation of $\pm 0.02mg$. For measuring the trays used for water evaporation from the Laponite mixture a more sturdy weight was required. Here a Sartorius CP 6201 balance was used. This balance has an accuracy of $0.1g$ and a standard deviation of $\pm 0.1g$. The Sartorius CP 6201 was also used for measuring the K_2SO_4 used to keep a high constant humidity in the sample storage container.

After the sample mixture was prepared it was poured into smaller moulds. These rectangular moulds were made in aluminum at the precision engineering workshop at NTNU and had internal dimensions $140mm$ x $20mm$ x $30mm$. The moulds were open in the bottom and were placed on a glass plate to allow the Laponite mixture to bond with a glass surface while the mixture sets. The glass plates were ordinary glass and had dimensions $160mm$ x $40mm$.

The lack of a bottom did however frequently cause the mixture to leak between the aluminum mold and the glass and thus it was decided to use a rubber seal. This seal was a simple $2mm$ thick rubber mat cut after the shape of the mould. This arrangement worked well as long as sufficient pressure was applied on the rubber seal. This was most easily solved by using simple package tape around mould and glass before the mixture was poured in.

Figure 3.13: From the left: The Heidolph MR1000 magnetic stirrer and the Ohaus Navigator N30330 and Sartorius CP 6201 electronic balances.

Filtration Unit and Pump

The filtration unit used to remove large particles from the initial 1% laponite mixture was a Nalgene 75mm Filter Unit. This unit used filter paper with diameter of 75mm to filter the solution and allowed the attachment of a pump to create a lower pressure below the filter paper to ensure flow through the unit. The filterpaper utilized for this purpose was Nalgene filter paper with a pore size of 0.8μm and a diameter of 75mm.

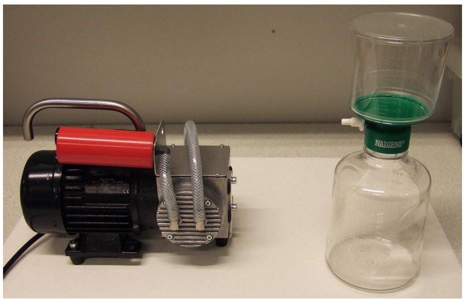


Figure 3.14: The filtration unit (without filter paper) and the pump used to pump the solution through the filter.

Equipment for the Evaporation of the Initial Laponite Solution

In order to ensure a rapid evaporation of the water in the Laponite solution to increase the Laponite concentration the solution was poured into trays and put into a heating cabinet. The used heating cabinet was a Memmert Modell 700, in which the solution was subjected to 100°C and a strong airflow to ensure rapid evaporation.

In the first attempts at evaporation the solution was poured into ordinary fireproof baking trays. Though these trays were suitable for the evaporation process due to their capacity of providing a large surface in comparison to a very shallow depth of solution, they caused certain other complications. Due to their low edges the trays were quite difficult to carry without spilling the Laponite solution. To compensate for this it was attempted to use a smaller amount of solution, but this resulted in a too rapid coagulation of the solution and rendered the mixture useless. It was thus decided to find a container with higher edges and a large fire-proof lasagna pan was a suitable replacement. The pan had the dimensions $400mm$ x $300mm$ x $60mm$ and the edges on this container proved tall enough to facilitate easy and spill-free transport of larger quantities of Laponite solution.

Sample Storage Container

The samples were stored in a large plastic container with dimensions $40cm$ x $30cm$ x $22cm$. This container is an ordinary commercial storage container with a loose top which may be attached by two clamps. These clamps press the top firmly against the box frame and thus make the container fairly airtight, although most likely not fully isolated. The box is however sufficiently airtight to keep a significantly higher air humidity inside than outside the container. The high humidity is ensured by placing an open container filled with a 8% solution of K_2SO_4 inside the container. The bottom of the container is covered by a metal grating to ensure a stable surface on which to store the samples and the solution of K_2SO_4 was kept in an open container with a large surface area to ensure maximum evaporation. The humidity was roughly monitored with a simple analog sauna hygrometer and was constant at about 80%. The container is shown in Figure 3.6.

Miscellaneous Equipment

There were many miscellaneous pieces of equipment used for this experiment. Some of these pieces were used to attach the VT-80 to the sample. The precision engineering workshop at NTNU were very helpful with designing and constructing both a roof mount and a line attachment for the VT-80. The roof mount was necessary to get a stable attachment for the main body of the VT-80 in the roof and the line attachment was used to connect the moving part of the VT-80 to the line going to the sample. The line was a simple Venetian blind cord, which is inelastic, light and robust. It was therefore suitable to connect the sample to the positioner as the inelasticity removed delay between the reaction in the sample and the movement of the positioner and prevented any distortion between the recorded and the actual velocity of the fracture propagation. Elasticity in the cord would also distort any measurement of the force required for a constant fracture propagation velocity. The other end of the line was attached to a simple clamp of the kind that is frequently used to attach paper to a notepad.

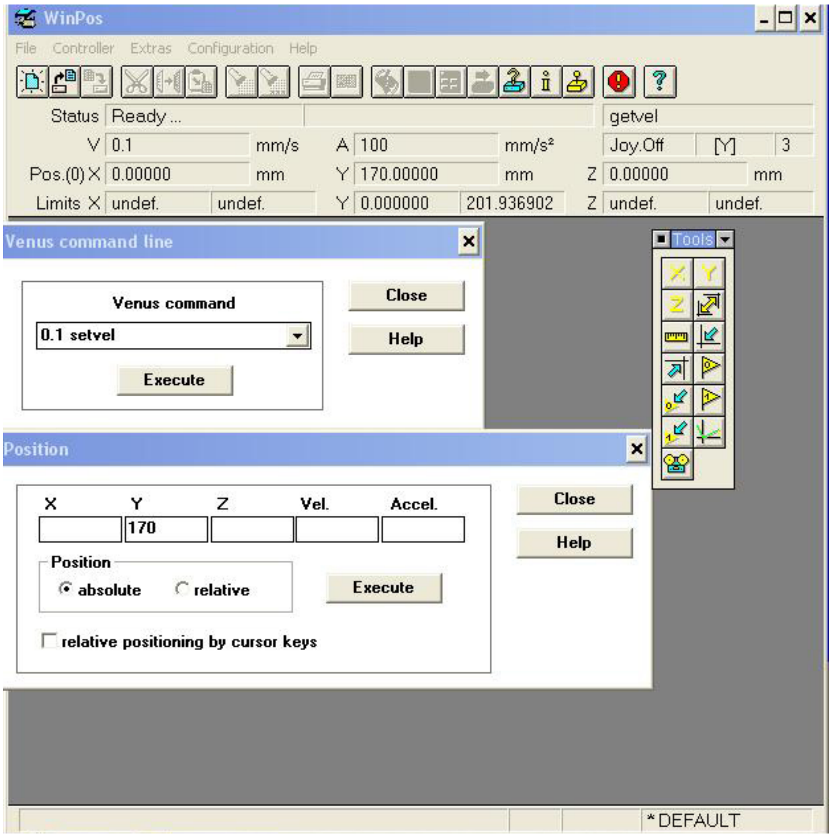
The filter paper that was attached to the top of the Laponite samples was circular sheets with a diameter of $240mm$ cut into rectangular strips of approximately $160mm$ x $25mm$. The paper was Whatman type 4 filter paper.

When the experiments were carried out the samples were placed upon a square $400mm$ by $400mm$ glass plate above a camera. This glass plate was placed on a simple construction of isopor, which stably elevated the glass plate $240mm$ above the surface of the table. This led to the camera lens being approximately $160mm$ from the sample, as the camera was laying on the table, with the lens pointing upwards. The whole setup was illuminated by a Eligent ELE-WL-ST-1 28W work lamp.

Software

The most important computer program for this experiment was the WinPos software. This program controlls the VT-80 positioner through the SMC Corvus. Winpos operates through the Venus-1 command language and allows the user to change position, velocity, acceleration and such for the positioner in a simple and efficient manner. The interface of the WinPos software is shown in Figure 3.15.6

Windows Movie Maker was used to record the experiment through the Pixelink camera. This simple software allows for easy recording and editing of video files and stores the recorded files in the *.wmv* format.



Status Ready ... getvel

V 0.1 mm/s A 100 mm/s² Joy.Off [Y] 3

Pos.(0) X 0.00000 mm Y 170.00000 mm Z 0.00000 mm

Limits X undef. undef. Y 0.000000 201.936902 Z undef. undef.

Venus command: 0.1 setvel

X | Y 170 | Z | Vel. | Accel.

Position ● absolute ○ relative

☐ relative positioning by cursor keys

*DEFAULT

Figure 3.15: The interface of the WinPos Software. This software may be used to control the position, velocity etc. of the VT-80 positioner. This screenshot was taken before the first test of the experimental setup, and the velocity of the positioner was set to $v = 0.1mm/s$

Chapter 4

Results and Discussion

Due to the limited amount of time available for experiments, the amount of experimental data attained was somewhat limited. The available data was collected from two tests of the experimental setup and one proper experimental session. It was unfortunately also very difficult to properly extract data from these experiments. The gel is transparent, and it is exceedingly difficult to photograph surface patterns on a transparent material. Possible solutions for this problem will briefly be discussed in Section 5.1.

The fracture patterns created on the experimental samples were photographed against black and white backgrounds with illumination from a variation of angles in order capture the patterns as well as possible. The resulting pictures were however not as good as hoped for, and it is therefore very hard to distinguish any clear fracture patterns from the photos. In all but one of the samples no clear fracture patterns were observed by neither visual inspection of the samples, nor by later inspection of the photos of the fracture surfaces. Whether this lack of results is due to a lack of a proper technique for recording fracture patterns from the transparent surfaces or because of an actual lack of proper fracture patterns is impossible to say without further experiments. The lack of proper means for spotting fracture patterns has thus severely limited any analysis of possible patterns on the fracture surfaces.

As for the nature of the fractures themselves, the fracture process is most certainly dominated by Mode I cracks. The fracture is however unlikely to be a completely pure Mode I crack, and there is possibly also a smaller degree of Mode II fracture. Mode I fractures probably dominate, as the force applied to the material is applied in the vertical direction, creating the appropriate conditions for a typical "opening" crack, a classic example of Mode I. As the crack is pulled along the horizontal gel column, there may however be some shear stress parallel to the direction of propagation. This Mode II effect will most likely have a much smaller effect than the Mode I crack, though it may still influence the growth of the fracture. There is also a possibility that there may be some local occurrence of Mode III cracks, though most likely only if there are any local impurities or dislocations in the gel around the crack front. There is no externally applied force in the horizontal direction perpendicular to the direction of propagation, and thus no externally caused Mode III fracture. Small inhomogeneities in the gel may however possibly shift the stresses locally so that a small horizontal shear stress arises in the direction parallel to the direction of propagation, thus causing a small degree of Mode III cracks. This effect is however most certainly negligible, and the overall fracture will be a Mode I crack, with the possibility of a some intermingling with a weaker Mode II crack.

4.1 Results from the Tests of the Setup

The first experimental test produced the most fascinating results. This experiment produced a clear large-scale V-pattern, as shown in Figures 4.1 and 4.2. These patterns may be a manifestation of the patterns observed by Tanaka et. al. [2]. That study showed that a peel-test experiment in acrylamide gels produced patterns of velocity dependent V-shapes. The V-shapes observed by Tanaka et. al. were observed as lines extending ±45° from the propagation direction. The lines observed in this experiment does however extend on an angle of ±23°, which is different from both the step lines observed in gels by Tanaka et. al. [2] [4] and the better known Wallner lines from fractures in glass.

There may also be alternative explanations of the V-shaped patterns. The slight damage caused when the gel was cut free from the frame may have changed the behaviour of the system. The patterns extend all the way from the edges to the centre of the gel, so the patterns might be the result of the the damage caused to the sides of the gel. The patterns have not been reproduced on any of the other experiments that have been conducted, though only a very limited number of experiments have been carried out so far. Furthermore, the gel used in the test experiments was different from the gel used in the later experimental session. Thus the behaviour could also be a result of the presence of larger aggregates in the gel made from an unfiltered solution. Due to the very limited number of conducted experiments it is however too early to come to a clear conclusion about the nature of this fracture pattern.

The second test produced somewhat different results than the first. The pattern from the previous test was not reproduced. The fracture surface on the second sample, shown in Figure 4.3, is very chaotic, and no obvious pattern of lines may be observed. It should however be noted that some problems were experienced during the testing of this sample and this may possibly have led to the lack of a well-defined pattern in this pattern. The complications are specified in Section 3.3.2.

The value of the result from the second test is somewhat dubious. As mentioned in Section 3.3.2, there were some problems with this this sample and the experiment. Some of the solution from the sample had leaked out before the sample had set, causing the sample to be lower than it was supposed to be. Furthermore some trouble was experienced during the experiment, both during the attachment of the filter paper and later as the strip of filter paper tended to slip off the top of the gel column. It is however at the present not possible to determine whether these complications had any impact on the development of the patterns of lack thereof on the fracture surface. Gaining further insight

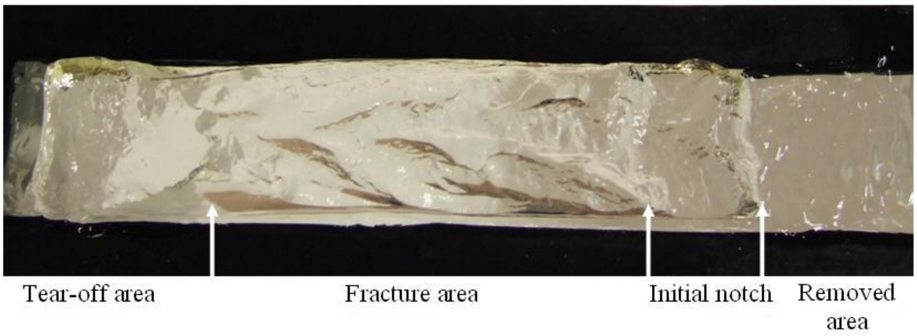


Figure 4.1: The sample from the first test of the experimental setup. The area on the far right part of the sample is a section of the sample that was cut away before the start of the experiment. The area next to it is the area of the initial notch in the end of the sample. The actual fracture starts at the left of the notch and continues until near the left end of the material, where the paper was torn off completely (the tear-off area). A V-shaped pattern is visible in the fracture area.

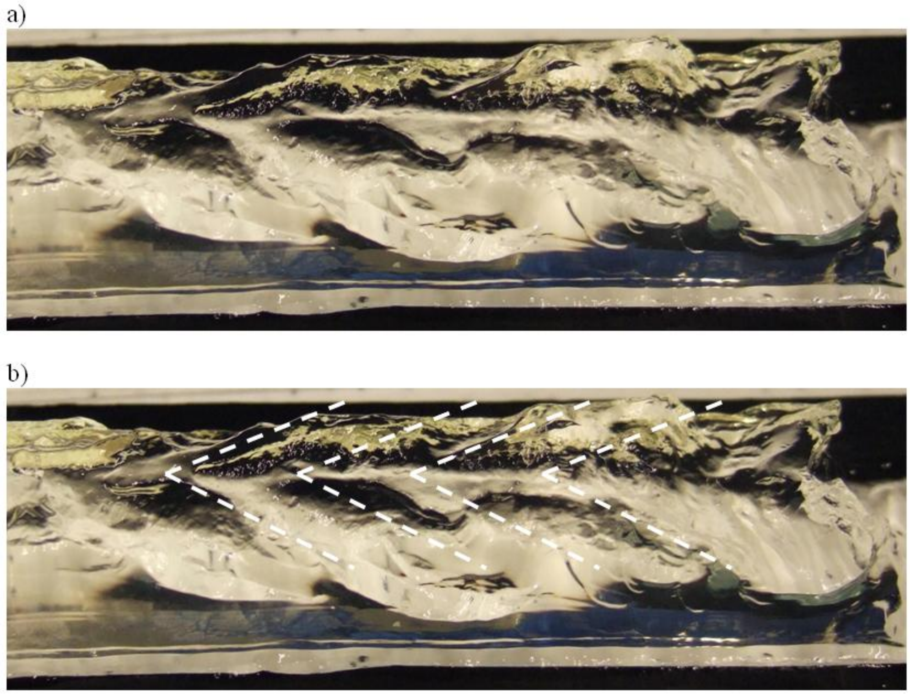


Figure 4.2: The fracture surface from the first test sample. a) The fracture surface has a V shaped pattern with lines extending at ±23° from the centre of the fracture surface. The pattern is illustrated on b).

Figure 4.3: The fracture surface after the second test of the experimental setup.

into the mechanisms of fractures in Laponite gels by continued experiments may possibly illuminate the subject sufficiently to reach a conclusion on this subject.

4.2 Results from the First Experimental Session

The execution of the first experimental session yielded 5 fracture surfaces produced at 3 different fracture velocities. Samples 1 and 2 were tested at the lowest velocity, $v = 0.1mm/s$. Samples 3 and 5 were tested at $v = 1mm/s$, and sample 6 at $v = 5mm/s$. No clear pattern was observed on the fracture surfaces, either because of the complications described above, or due to an actual lack of definite patterns. The first sample had to be discarded completely after the experiment, because the filter paper slipped from the top of the gel column. When a new attempt was made, the paper slipped from some areas of the top surface, but remained attached at other locations, thus tearing of some areas of the surface while leaving other areas intact. The sample could naturally not be used again.

Thankfully, this did not occur with sample 2. A very thin layer of this sample was torn off, though there may have been a few areas where the crack approached the paper sufficiently for the paper to slip locally. These areas of slipping were however quite rare. Unfortunately, there does not appear to be any clear pattern on the fracture surface of this sample. Neither for samples 3, 5 nor 6 was any clear pattern of step lines observed. The only observed difference between the samples tested at different fracture velocities is that the seemingly random distributions of lines seems to be further apart in the samples with the higher velocities. This would be consistent with the results by Tanaka et. al. [2], where it was observed that the structure of step lines became more complicated and occurred more frequently as the fracture velocity was reduced. However, the effect was a lot more noticeable in that study. Furthermore, only one successful test has at this point been conducted on the Laponite gels at each velocity, and thus there are not nearly enough experimental results to come to any definite conclusions. The effect of the possible increase in length between the lines on the fracture surfaces does unfortunately not appear to have become as discernible on the photos of the samples as it was on a direct observation of the samples.

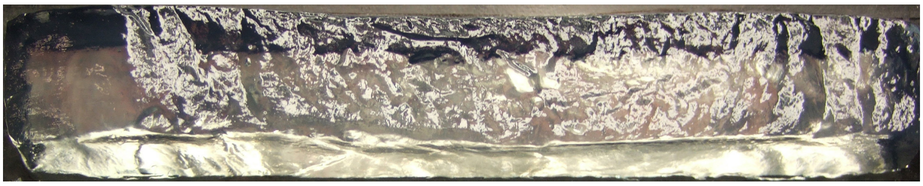


Figure 4.4: The fracture surface of sample 2 from the experimental session.

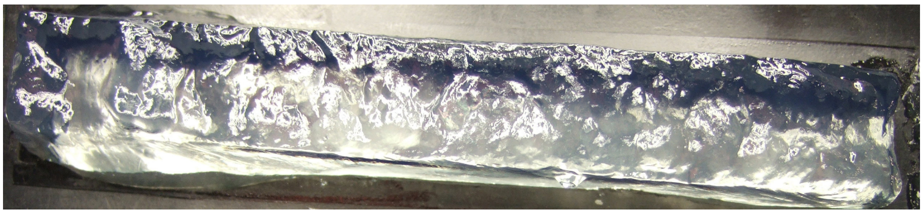


Figure 4.5: The fracture surface of sample 3 from the experimental session.

All the samples tested in this experimental session seemed to have a propensity to fracture very close to the strip of filter paper. Sample 1 started the fracture about $1mm$ from the paper, ignoring the initial incision altogether. Samples 2, 3, 5 and 6 started the fracture at the the designated starting point, but immediately moved in the direction of the paper. When the crack was within $1-2mm$ of the paper, it started propagating parallel to the paper. This happened within the first $30mm$ of the fracture propagation. This effect was not observed in the two test samples. Thus one possibility is that it is an effect of the concentration of Laponite. The concentration of Laponite was about 1.5 times stronger for the test samples than for the samples from the experimental session. The difference in behaviour may also be due to that the samples in the experimental session were filtrated, while the earlier samples were not. Remaining aggregates of clay platelets in the unfiltered samples may possibly in some way have held the test samples together more firmly than for the filtered samples without such aggregates, though there is most likely some other explanation. It may also be that samples of lower concentrations of Laponite require a very long time to set, and that the samples require more than 10 days to set fully into a gel.

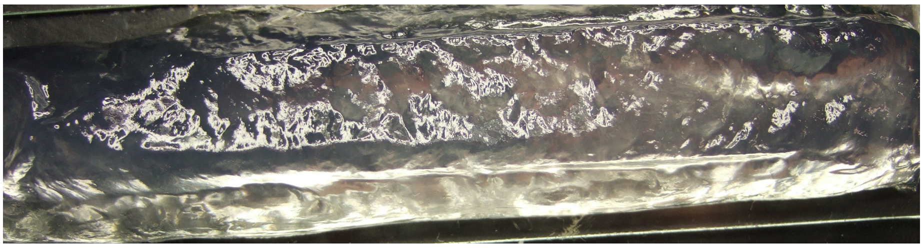


Figure 4.6: The fracture surface of sample 5 from the experimental session. Note that the structure of the sample is in the process of collapsing by floating outwards. This is the result of some damage caused to the sides of the sample when the frame was removed.

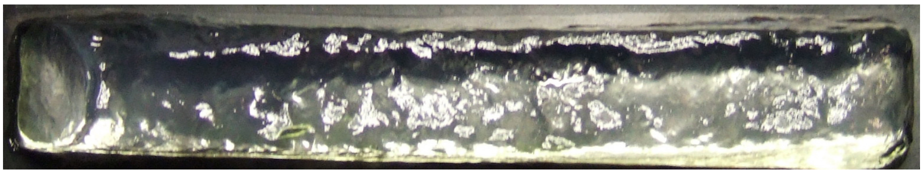


Figure 4.7: The fracture surface of sample 6 from the experimental session.

Chapter 5

Concluding Remarks

This project has been done as a preparation for a study on fractures in soft materials, with a focus on Laponite gels. To lay the ground for this study, a the objective for this project was designing and assembling an experimental setup according to the experiment done by of Tanaka et. al. [2]. Thus their peel-test like method for studying acrylamide gels might be applied on Laponite gels.

The project was started in mid September, 2006, with a deadline on December 20th. This short time frame was sufficient for the construction of the experimental setup, though there was only a very limited amount of time available for testing and running experiments after the assembly of the experimental setup. The design and assembly of the experiment involved finding and setting up a positioner, constructing a small experimental stand, and designing sample moulds and a general process for creating samples. There were some technical challenges in making the setup work properly, and the process for creating samples still need some improvements, but the setup is now working and well tested, even though further improvements certainly can be made.

The most time consuming work after the construction of the setup was complete, was the preparation of samples. A single session of sample preparation required nearly half a day, and carried a high risk for being unsuccessful. Thus only one session of experiments could be carried out, in addition to two smaller tests of the experimental setup. The tests of the setup were carried out on two gel samples with Laponite concentrations of 3.5%. The first of these produced an interesting fracture surface with a V-shaped pattern with possible step lines extending at ±23° from the crack propagation direction. There are several possible explanations for this pattern, of which the most interesting theory is that the pattern is caused by the same mechanisms as the patterns observed by Tanaka et. al. [2]. However, as was explained in Section 4.1, the source of the patterns may also be some damage to the sides of the sample. There is also a chance that the pattern is unrelated to the possibilities mentioned above, though this will only become clear with further experiments.

The second test sample showed no clear clear patterns on the fracture surfaces, and there was also a lack of clear fracture patterns on the samples from the experimental session. These samples, which had respective concentrations of

Laponite and NaCl of $C = 2.24\%$ and $I = 1.01 \cdot 10^{-3} mol/l$, were tested at 3 different fracture velocities, $0.1 mm/s$, $1 mm/s$ and $5 mm/s$. There might have been a slight increase of the distance between lines on the fracture surfaces with the increase in velocity, but further experments must be conducted to draw any conclusions about the actual impact of the fracture velocity.

The greatest remaining challenge is to improve the process for recording and analyzing the fracture surfaces. It has been quite challenging to do this so far due to the transparent nature of the Laponite gels. The experimental setup may also be used for studying the propagation of the fracture, though some more work is required before this may be carried out satisfactorily.

In conclusion, an experimental setup has been assembled to study fracture surfaces for Laponite gels. The setup was constructed according to the work done by Tanaka et. al. [2], and has been tested on a few samples. The setup is now ready for both further tests, though some improvements may be done to both the setup and the process for preparing samples. The most pressing concern is however the improvement of the methods for recording and analyzing patterns on the fracture surfaces.

5.1 Future Work

As the stated objective of this project is to lay the ground for future experimental work, there is naturally a great deal of follow-up activities that can and will be carried out. The experimental setup has so far only been used for a single proper experiment, yet conclusive data will require many iterations of the experiment with methodical testing of the impact of the various changeable variables. A proper parametric study should be carried out on the parameters fracture velocity, Laponite concentration and concentration of NaCl.

It is also possible to submerge the sample in silicon oil while performing the experiment. The oil will not destroy the water-based structure of the gel samples, but may seep in between the fracture surfaces while the experiment is being conducted, thus increasing the stability of the system and possibly allowing a purer fracture process. The oil has a slight colour and may provide some contrast for the filming of the fracture process. Hopefully this will increase the value of the film footage so that the captured movies may be used to analyze the propagation of the fracture front. On a related subject, alternative methods for studying the propagation of the fracture front should also be looked into. One possible method may be to use a high speed camera to continuously photograph the fracture propagation. Experimenting with the use of the PixeLink PL-A642 may also yield results.

A high priority will also be given to developing and improving methods for investigating the fracture surfaces of the gels. These highly transparent surfaces are notoriously difficult to photograph, though recording the information in the fracture patterns is crucial to the success of these experiments. There is no point in investigating fracture patterns if the patterns themselves cannot not be recorded. Several methods for recording the patterns may be tested. These methods may include casting with different types of epoxy, innovative use of light reflection and photography and possibly some other procedures.

There are also several improvements that should be done on the experimental setup itself. In order to greatly increase the value of the experiments, a load cell or equivalent device should be installed in order to allow for the measurement of the applied force required for fracture propagation at a constant velocity as a function of time. This data may be used to calculate the fracture energy, as done by Tanaka et. al. [2]. The fracture energy and force as functions of time may then be compared with the roughness and fracture patterns of the fracture surface. As a side note, the installation of a force measuring device should coincide with replacing the line connecting the positioner to the sample with a completely non-elastic line.

If the fracture energy is to be calculated, the Young's modulus for the Laponite gel will have to be determined. This may be done with the help of a rheometer. Another instrument which possibly may be used to obtain a greater understanding of the sample materials is an Atomic Force Microscope (AFM). This instrument has previously not been particularly suited for investigating soft materials, though recent progress in the field may change this[1]. Using an AFM would in such a case make it possible to investigate the microscopic structure of the Laponite gel. A greater understanding of the microscopic structure, together with the macroscopic fracture experiments may grant a deeper understanding of the nature of fractures in aqueous gels.

The process for preparing samples should also be improved. Having to use an evaporation technique to increase the concentration of Laponite above 1% is clearly unsatisfactory. The the best solution for eliminating the evaporation phase of the process is to use a filtering tecnique that allows for filtering solutions with higher concentrations of Laponite. If such a replacement for the filtering process could be found, the the evaporaton phase would be redundant. Therefore a high priority will be given to finding and acquiring a filtering system capable of filtering solutions with a higher concentration of Laponite.

Finally, a literature study on oblique step lines on fracture surfaces should be conducted together with repetitions of the first proper test of the system. The patterns observed in the first test seems similar to the step lines observed in other studies, yet the angle between the lines and the crack propagation direction is much smaller than the ±45° observed by Tanaka et. al. [2] [4]. If the pattern and the angle is duplicated in new tests and no record of other observations or possible explanations are found in the literature, this could be a very interesting discovery.

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