Mechanical properties dependence
on microstructure in aerogel-like
Quartzene®

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Abstract

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In this project the relation between pore size/porosity and the mechanical properties has been studied in the aerogel-like material Quartzene®. Quartzene® is a patented material produced by Svenska Aerogel AB. Density measurements were made on three different types of Svenska Aerogels ABs Quartzene® in the shape of pellets. These three types of Quartzene® is called CMS, ND and E9. The mechanical properties were studied by doing diametrical crush-tests on the pellets. Afterwards the samples were examined through SEM in order to study the structural properties like porosity and microstructure. By examining the materials in this order the group hoped to find a correlation between the mechanical properties and the pore size/porosity. Other microscopic analyses such as TEM and FIB was considered, but due to time limitation these methods were not used.

Rough density measurements resulted in an estimated density of 0.82-0.88 g/cm³ for CMS, 0.28-0.30 g/cm³ for E9 and 0.21-0.22 g/cm³ for ND. The crush-tests resulted in a mean fracture stress of 0.81-0.89 MPa for CMS, 0.30 MPa for E9 and 0.20-0.21 MPa for ND. Studying the materials in SEM resulted in an observed mean pore size of 59-73 nm for CMS, 264-362 nm for E9 and 690-710 nm for ND in the mesoporous domain.

A subtle relationship between density/pore size and fracture was obtained, with a higher density and smaller pores leading to a higher fracture stress. Due to the lack of data in this study, it is recommended though that this is something that should be examined further before any conclusions can be made.

In general Quartzene® has shown to be a brittle material, but this study indicates that the mechanical properties could be controlled in somehow through the microstructure of the material, focusing on controlling the pore sizes. Further investigations in sintering of Quartzene® are also recommended in this study because of its promising effects on the mechanical properties shown in other studies.
Acknowledgements

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1 Introduction

Svenska Aerogel AB is interested in how their aerogel-like material Quartzene® as beads, pellets and granules is affected by mechanical strain. With this study Svenska Aerogel AB wants to develop a standardized methodology to classify their different products regarding mechanical properties. As for today the company does not have a standardised method to decide the mechanical properties of their products. It is important for Svenska Aerogel AB to determine the mechanical strength of Quartzene® since this will help them to meet their clients’ requirements of their products. Svenska Aerogel AB also wants to compile a chart/table where the properties of their products are listed. A compilation regarding the properties of their products could give Svenska Aerogel AB and their customers a better overview of Svenska Aerogels ABs products.

1.1 Goals

The main goal with this project was to investigate the connection between the mechanical strength, geometric microstructure and the pore size/porosity of Quartzene®.

Another goal was to provide a compilation of data and properties of the materials that were investigated to Svenska Aerogel AB. This would give Svenska Aerogel AB a better overview of their materials and their properties in order to advise their customers more easily in their choice of material.

1.2 Delimitations

Studies of Quartzene® were not performed under different environmental conditions (such as humidity, temperature, pressure) since it would be too extensive in proportion to the time span for this project. These parameters for the actual conditions during the experiments have been well documented though, so that the experiments can be reproduced or used as references for any further studies by Svenska Aerogel AB.

The samples were only studied with SEM and tabletop SEM to investigate the pore size and the porosity. This was because of the time limitation and the fact that the technical consultants gave the advice that this would be a good delimitation.

The three different types of Quartzene® CMS, E9 and ND were studied in this project and only as pellets. This was because beads and granules would have too much of an unsymmetrical geometry to use for mechanical strength tests. Together with Svenska Aerogel AB the conclusion was made that this would be a good limitation for this project.

1.3 Project design

The first step in this project was to do pre studies on the area and the available methods. The pre studies consisted of literature studies followed by consulting and advising from the technical consultants available at Uppsala University. The technical consultants were of much help in order to set the project on the right track and evaluate whether the ideas would actually work and be applicable. Along with the technical consultants and Svenska Aerogel AB a goal was set up for the project and what methods that would be used were chosen.

After the pre studies were finished it was decided that the mechanical tests would be performed prior to the microscopy. This order was preferred partly because there was a long analysing time of the data from the mechanical tests. It was also preferred because the samples that had gone through mechanical tests wanted to be analysed with microscopy tests. The mechanical tests that were performed were crush tests.
After the mechanical tests it was time for the microscopy tests. The microscopy tests consisted of tabletop SEM (Scanning Electron Microscopy) and high resolution SEM.

When the laboratory moments were completed the only thing remaining was to analyse the test results and to compile these results to give Svenska Aerogel AB a better overview of their different variants of Quartzene®.

2 Background

2.1 Aerogels

Aerogels are low density materials that are built up by a porous net structure with a large inner surface area. As indicated by the name, aerogels commonly consist of about 95% air which gives these materials unique properties and applications such as low thermal conductivity and a high specific surface area.

A gel is generally built up by a solid network structure surrounded by a liquid. Aerogels are materials where the liquid has been removed and replaced with a gas (often air).

There are many different types of aerogels with different chemical compounds building up the gel network. The compounds can be anything from metal oxides and silica oxides to inorganic-organic hybrids as well as organic compounds. These chemical compounds form particles and agglomerates that build up the gel network. In figure 1 these particles are illustrated as circles. The network structure is very complex and is similar to that of foam. Aerogels can either be made as intact bodies, monoliths, when dried from the wet gel or as smaller agglomerates suspended in a solution. [1]

A model to describe aerogels is to picture them as cellular materials where the structure and shape of the network is approximated as cells. These cells have a certain geometry that repeatedly builds up the structure. The aim of a model like this is to get a well-defined and uniform geometry of the network structure in order to better explain the mechanical behaviour of the material. Ashby used the model of a hexagonal cell network in order to describe the mechanical behaviour of a cellular structure under compression (figures 2a-2e). Ashby described and explained the resulting stress-strain curve during compression and the result is shown in figure 3. [2][3]
As with models in general, it is of interest to describe aerogels as cellular materials because of the predictions that can be made from the model. The model of a cellular material is a good way to predict mechanical properties and behaviour for a material (if the material can be described as a cellular material of course).

2.2 Quartzene®
Quartzene® is a hydrophilic aerogel-like material with about 98% porosity and density varying from about 40 to 400 kg per m³. Svenska Aerogel AB produces Quartzene® for many
different applications, such as air and water purification, insulating building materials and cosmetics. Depending on the application, Quartzene® is produced in different chemical compositions and by different methods. The different production methods result in Quartzene® in different shapes and geometries of the final product, such as pellets (figure 4), beads and granules. And the different chemical compositions result in different microstructural properties. [4]

Figure 4: Quartzene® in the form of pellets.

The three different types of Quartzene® that were examined in this study were CMS, E9 and ND, all in the shape of pellets. Two different pellet types were examined called 4 mm and 6 mm. These are simply the diameters of the casks were the pellets are casted. E9 and ND are quite similar and there are just small differences in the chemical composition. Both E9 and ND consists essentially of Si(OH)$_2$ in some kind of hierarchical structure in two or three different stages. In this structure it is probably possible to identify particles in different sizes. From small particles that are only a few-nanometres in size to agglomerates that are in the 10ths’ micrometres range. [5]

CMS is a stoichiometric compound who could be written as $(\text{Ca}_{0.35}, \text{Mg}_{0.65})\text{O} \times 3.35 \text{SiO}_2$. CMS is produced according to the principle Svenska Aerogel AB has stated in their patent. A balanced salt solution containing Ca and Mg typically in compound with chlorides is mixed with a diluted water glass solution and the compound of CMS is formed. It is possible to say that two Na-atoms in the water glass are replaced with 0.35 Ca and 0.65 Mg. Ca and Mg are divalent and that is why only half of the Na-atoms are replaced.

The slurry which is created contains Na-ions as a residue from the water glass and Cl-ions from the original Ca- and Mg-salts. These ions are removed and then the paste is dewatered to a cake that despite its rigid rubbery texture still has about 85% weight percent of water in it. After this step there are various methods to process the cake to desired shape and it is also possible to impregnate the paste with chemicals if wanted. [5]

3 Theory
The strength of a material is commonly measured by tensile test, shear test and/or compressive test. The hardness can also give some relevant information and the most common test methods are Brinells hardness test, Vickers hardness test and nanoindentation.

Common methods for studying the microstructure are transmission electron microscope (TEM), scanning electron microscope (SEM), computed tomography (CT) and focused ion beam (FIB).

3.1 Compressive strength with crush-test
The compressive strength is examined in a crush-test, also called diametrical compression test, which is a method where a cylinder/disc exhibits a load in radial direction, as shown in
figure 5. By measuring the compression force and the strain in radial direction a stress-strain curve can be obtained.

\[ F \] is the applied force, \( D \) is the diameter and \( \sigma \) is the stress.

The purpose of the compression was to study the mechanical properties of the three different types of Quartzene® CMS, ND and E9 that Svenska Aerogel AB manufactures. The stress-strain curves make it possible to compare the three different types to see how they differ in strength since crush-testing is a standardised and comparable method. The stress-strain curves may also tell if Quartzene® in the form of pellets behaves like a cellular material.

3.2 Brinell hardness test
In the Brinell hardness test a carbide ball is forced into the material that is examined. The loads can vary much and depends on the hardness of the investigated material. Harder materials require higher applied loads. During a test the load is held constant for a certain amount of time and is then removed. Then the compressed area is measured. It is then possible to calculate the hardness, \( HB \), which is a function of the size of the load and the diameter of the resulting compressed area. The fact that Brinell hardness test is a standardized and comparable method makes it an interesting way to qualitatively say something about the hardness of the three types of Quartzene®. The hardness test represents a part of the characterization of a material.

3.3 Vickers hardness test
Vickers hardness test is almost the same as the Brinell test, but with a pyramidal geometry used instead of a ball. Otherwise the principle is the same where the diagonals of the indentation is measured and then translated into a hardness value, called \( HV \) in this case.

3.4 Nanoindentation
Brinell and Vickers hardness tests gives good results but sometimes the area you want to investigate with an indentation is too small. The procedure of nanoindentation is similar to that of Brinell and Vickers hardness test. The difference is that the tip that is pressed into the material and the applied load is much smaller. Also, the imprint is not measured manually since it is too small. It is instead automatically registered by the device.
3.5 Scanning electron microscope
The SEM is the most common method of electron microscopy. In the SEM an image is created by scanning the surface of a sample with a focused conical electron beam and detecting the back-scattered electrons and secondary electrons.

The SEM uses vacuum which allows the electrons to move unhindered. This prevents the electrons from colliding with any gas molecules, which would scatter the electrons and prevent them from reaching the sample. The sample to be studied in the SEM therefore has to be able withstand low pressures and must not contain any volatile fluid. The size of a sample is limited by the size of the sample holder and the chamber in the SEM. The thickness of the sample has no theoretical upper limit (in contrast to TEM as seen below), since it is the back-scattered electrons and not the transmitted electrons that are detected in SEM. The sample has to be electrical conductive to make the electron-atom interaction possible and to avoid building up charge on the sample surface. Surface charge could repel incoming electrons in the beam and give a signal in the detector which does not represent the sample. If the sample is insulating it therefore has to be prepared before studying it with SEM. This preparation could for example be sputter coating.

The incoming conical electron beam (about 1 nm tip) usually scans the sample point by point in a raster scan pattern. The electrons in the incoming beam interact with atoms at the surface of the sample. Surfaces of the sample pointing in the direction of the detector give a higher amount of detected back-scattered electron than surfaces which does not. The detector output signal is proportional to the amount of detected electrons, so more detected electrons (surfaces pointing towards the detector) result in a higher detector signal. The position of the incoming beam is synced with the detector signal received at the monitor. This results in real time imaging of the scanning procedure on the monitor.

The detectors used in ordinary SEM are secondary electron detectors, which detects secondary electrons (SE) and backscattered electrons (BSE). Yet, the SEM can be equipped with additional detectors to detect a variety of other signals, such as characteristic X-rays, visible light (CL), transmitted electrons and specimen current. This can give information about the composition and other properties of samples studied with SEM. Samples studied in a SEM can be magnified to up to about 500,000 times. The magnification is very easy to control since SEM uses electromagnets instead of lenses as a regular optical microscope. The images are two-dimensional and despite the high magnification very distinct and with high resolution. SEM does not tear up the samples so the same sample can be studied repeatedly.

3.6 Transmission electron microscope
The transmission electron microscope (TEM) is analogous to a regular optical microscope. Instead of transmitting a beam of light in the visible spectra through the sample, as in an optical microscope, a very thin beam of electrons is used. The beam is of high energy (several hundreds of kV) which results in electrons with wavelengths much shorter than that of visible light.

TEM uses vacuum for the same reason as for SEM. This requires the samples to be viewed in the TEM to be able to withstand low pressure and cannot contain any volatile fluid. Additionally the sample has to be extremely thin (about a 1,000 Å at most) so that the sample has “electron transparency”. This means that the electrons must be able to pass through the sample so that they can be detected on the other side of the sample. Thus samples thicker than this must be prepared with more or less advanced techniques to fit the maximum thickness. Materials of higher dense elements need to be thinner than those of lighter elements to prevent the material from absorbing the electrons. Further preparation of the sample is sometimes
required depending on the properties of the actual sample. For example non-conductive samples need to be prepared by sputter coating to make it conductive, as in SEM.

The electron beam is focused by electromagnetic lenses onto the sample and interacts with the sample when it is transmitted through. Different structures in the sample scatter the electrons unequally which results in information of the overall structure of the sample. The resulting beam, i.e. the electrons that have passed through the sample and have not been scattered away, is projected by a second set of electromagnetic lenses onto a screen and an image of the samples inner structure is received. (Compare with optical lenses in an optical microscope).

The information received from TEM gives information about surface, shape, size and structure. As in SEM the resulting images are two-dimensional, but TEM is able to give a higher resolution (in the nanoscale) than SEM. However, the resolution in TEM is limited by the intrinsic aberrations in the electromagnetic lenses and so the resolution is limited to about 0.2 nm. [6][7][9][10][11]

3.7 Focused ion beam

A focused ion beam (usually gallium ions) is accelerated against a sample. When the beam hits the sample some of the material in the sample is sputtered and how much of the sample that is sputtered depends on the intensity of the beam. If a low beam current is used it is possible to get a good image of the sample that is investigated even though some material is sputtered. With FIB an image with good resolution down to 5 nm is possible to achieve. With a high beam current it is possible to sputter out some of the material where it is wanted. This sputtering can be very useful if an image of the bulk is wanted. The sputtering from FIB is extremely precise and this leads to that basically any area and size of the sputtering can be chosen down to nanometres.

A signal is sent from the sputtered ions or the secondary electrons and this signal give an image of the sample. FIB is often used together with SEM to be able to look inside the sample (sputtering from FIB followed by observation by SEM) but to get a good image with FIB is also possible. The resolution in FIB is however not as good as in SEM. [6][7][12]

3.8 Computed tomography

The principal for CT is that instead of sending X-ray beams from one angle the CT rotates and send X-ray beams from different angels. The X-ray also reaches different depths in the studied material depending on the intensity of different layers in the material. Because of these different depths it is possible to achieve two dimensional pictures of “slices” of the material. With these pictures and the rotation of the X-ray a three dimensional picture of the studied sample can be received. This can be used to study voids and cracks in materials. It is a non-destructive analysis method, which means that a sample can be analysed without destroying it. [13]

4 Method

It is very difficult to determine tensile strength and shear strength through standard methods because Quartzene® is so brittle. Neither a standard shear test nor a tensile test would work because the sample would break before any useful information could be obtained. After considering these facts and after consulting the technical consultants it was determined that a crush-test would be basically the only test that would work. From this test it is possible to calculate and estimate the tensile strength through assumptions and mathematical formulas. A material with higher tensile strength will also have higher shear strength. [14]
Brinell hardness tests was also planned to be performed. To perform a Brinell test on a material this brittle is however very challenging. It is challenging because the sample is likely to break during the test and then no relevant results would be given from the test. Considering this and the lack of time the Brinell hardness tests were excluded.

Brinell was considered rather than Vickers due to the difficulty in performing a Vickers test on a material that is likely to crack, which is the case with Quartzene®. The geometry of the load in the Vickers test could enhance the formation of cracks in the material and would possibly (more possible than the load in Brinell) lead to breaking the material. [14]

After literature studies and after consulting the technical consultants the conclusion was made that the mechanical properties most likely depends on the pores in the mesoporous domain. The mesoporous domain is in the magnitude between the nano- and the microscale structure. [7]

TEM was an alternative at first for the project but considering the amount of time it would take and the fact that the samples needed to be so extremely thin led to exclusion of the method. The structure that was of interest was also better and easier investigated by SEM.

The resolution from FIB is not as good as in SEM. FIB is also very time-consuming. Due to this, FIB was excluded as a method. [7]

CT was also considered as an alternative at first because in this method it is possible to look at a cross section of the materials in two and three dimensions. But when SEM and CT were compared the conclusion was made that SEM would be better. This because of the higher resolution and greater magnification in SEM. CT also emit a lot of radiation. Considering these facts as well as the lack of time, the conclusion was made that other methods like SEM were better and CT was excluded. [14]

4.1 Density Measurements
Density is one variable that Svenska Aerogel AB easily can measure. Therefore a decision was made to measure the density of each of the three types of Quartzene®. To really be able to take any measurement of the densities seriously the statistical sample needed to be large. Therefore measurements were made on 100 pellets of each batch.

At first, the hundred pellets with the most representative geometry were chosen from each batch of the different types of Quartzene®. All of those pellets geometry were measured, diameter and length, with a digital calliper. The calliper had an accuracy of 0.001 mm. Their weight was also measured with a digital scale that had an accuracy of 0.1 mg. The shape was approximated as a cylinder. When the data of the diameter, length and weight were found, the density was calculated for each pellet. A mean density was then calculated for each batch. After that 24 of these samples were picked out for further studies. These 24 were chosen because of their symmetrical and nearly cylindrical shape.

4.2 Crush-test
The tests were made with a machine called Shimadzu. The basic of this test was that two metal plates were pushed together with the sample in between, as shown in figure 6. The machine was linked up with a computer where the test could be controlled. The compression rate could be set and in the experiment a rate of 0.5 mm/min were used. The machine compressed the sample in the chosen rate and computed the force needed to compress the material in the constant selected rate. While the compression was made the computer plotted the force against the compressed length. The resulting curve was linear and very steep and when the curve deviated from this linear behaviour the test was stopped manually. The deviation from this behaviour indicated that some kind of crack or damage had occurred in the
sample and therefore a change in needed force. The sample was then removed and all the pieces of it were saved for the SEM tests. [15]

First some test runs were made so the behaviour of samples from each batch was detected. Then the real test was made on an average of 10 samples from every batch, so the total number of runs was 60. The temperature and relative humidity during the test were measured to 22°C respectively 51%.

Figure 6: A pellet of Quartzene® in the Shimadzu during compression.

4.3 Tabletop SEM

Some initial scanning electron microscopy (SEM) was carried out using a Hitachi TM-1000 tabletop microscope, prior to the studies with the high resolution SEM.

The samples did not needed to be prepared before they were analysed in the tabletop SEM. The reason why the samples did not need any preparation(as is needed in the high resolution SEM, in form of sputtering to make an insulating sample conductive) is because the vacuum is not as high in tabletop SEM as in the high resolution SEM. The electron gun that is used in tabletop SEM provides with a wider irradiation, resulting in a poorer resolution. [16]

The tabletop microscope could only handle very thin samples (about a millimetre thick). Therefore the crush tested pellets could not be analysed without destroying them further, which was unwanted since it was desirable to study the actual resulting fracture surfaces from the crush tests. The crush tested pellets were therefore not used in the tabletop SEM, but were instead saved for the high resolution SEM which could handle larger samples.

The samples for the tabletop SEM were prepared one at a time, by crushing the pellet into very small pieces (millimetre size at most) and attach them to the sample holder with a carbon tape.

Once the sample had been prepared the sample holder was placed in the tabletop SEM, vacuum was established and the sample could be viewed on a computer monitor by using custom software.

Only one sample from each batch (i.e. a total of six samples) was examined in the tabletop SEM.

4.4 High resolution SEM

The crush tested samples were prioritized when choosing samples for the SEM. One sample from each batch, i.e. a total of six samples, were chosen. The samples were attached with a carbon tape to each sample holder. The most porous samples needed to be “glued” to the sample holders with silver paste to prevent them from falling off. (Since the SEM uses vacuum the samples were required to be properly fixed to the sample holders). The silver paste also increased the conductivity between the sample and the sample holder. [7]
The samples were placed in the sputter chamber. When the vacuum pump was turned on, one of the samples (ND 4 mm) was lost since it apparently was not fastened enough to the holder. The samples were then sputtered with a gold palladium alloy at 2.2 kW for 60 seconds. The aim was to keep the sputtering at 20 mA, which according to a graph in the sputter manual should give a 8-10 nm coating, but it was hard to keep it stable. It fluctuated a lot between 10 and 30 mA. [7]

Then the sample holders were placed in a sample holder tray and placed in the SEM. The tray got stuck in the door when it was loaded into the SEM. When trying to get the tray loose, one more sample (ND 6 mm) was lost and only four samples could be studied in this run. In a second run, samples from the ND 4 mm and the ND 6 mm batches were prepared again (as above) and could finally be studied.

The SEM used in this project was from the manufacturer Carl Zeiss and the model is called Merlin, and had a Gemini electron gun.

5 Results

5.1 Density measurements

The resulting densities were calculated using equation (1) and (2), and can be seen in table 1.

\[
V = \frac{\pi d^2 l}{4} \quad (1)
\]

\[
\rho = \frac{m}{V} \quad (2)
\]

<table>
<thead>
<tr>
<th>Mean value of Density (g/cm³)</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>CMS (6mm)</td>
<td>0.87911</td>
</tr>
<tr>
<td>CMS (4mm)</td>
<td>0.82151</td>
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<tr>
<td>E9 (6mm)</td>
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</tbody>
</table>

The main difficulty with this particular part was the measurements of the geometry. The measurements were made by a digital calliper. This calliper was adjusted by hand and therefore some errors occurred. Another problem was encountered when doing the measurements and that was that the pellets easily fell apart. Because of that the callipers could not be put as close as wanted to the sample. This was more of a problem with the ND samples than with any other sample. Problems also arose when selecting representative cylindrical
pellets since none of them had an even cylindrical geometry. A needed approximation was made.

5.2 Crush test
From the experiment a graph of the force against compressed length was received for every sample that was tested. An example how this graph looked like can be seen in figure 7.

![Figure 7: Representative behaviour of the analysed pellets of Quartzene® during compression, with CMS 6mm as an example. At first the curve is steep and a somewhat elastic behaviour is shown. When a crack in the pellet occur the curve drops.](image)

The stress ($\sigma$) in a cylinder during diametrical compression is calculated by equation (3).

\[ \sigma = \frac{2F}{\pi Dt} \]  

(3)

For the strain equation (4) was used. In equation (3) & (4) F is the applied force, D is the pellets diameter, t is the length of the pellet and U is the diametrical compression.

\[ \varepsilon = \frac{U}{D} \]  

(4)

Because of the fact that the density was calculated for all the samples, A plot of the fracture stress, the stress where the cracks occurred, against the density could be plotted. This is shown in figure 8.
Figure 8: The fracture stress against the density for the three types of Quartzene®.

An interesting behaviour was noticed when the fracture stress-density graph was plotted with logarithmic axis, see figure 9. It shows that the logarithm of the fracture stress is linear with the logarithm of the density, a trendline was calculated and plotted in the stress-density graph with logarithmic scales. If then the value for SiO$_2$’s fracture strain and density in crystal form (quartz) [19] was plotted in the same graph the point for quartz falls perfectly aligned with the trendline.[18] The trendline follows equation (5), shown below.

$$y = 3.59 \times x^{3.24} \quad (5)$$

The values for SiO$_2$ were used because that is the main structural compound in many types of aerogel as well as for the three types of Quartzene® in this study.
Figure 9: Fracture stress as a function of density. The trendline shows that the relationship between fracture stress and density can be explained by a power law with the exponent of 3.24.

The way the samples cracked was studied to see if it affected the values of the strength. Three different “crack-modes” were defined as shown in figure 10. One where the samples cracked along the length (1) and one where it cracked over the cross section (2). The last mode was when the crack was undefined, like a corner piece fell off or the sample fell into many pieces (3). No sign of “crack-mode” dependence was found in the data. One “crack-mode” could have both high and low fracture stress values in one batch.

Figure 10: Illustration of the three different "crack-modes" identified from the crush tests. (1): cracked along the length. (2): cracked over the cross section. (3): no sign of “crack-mode” dependence.
5.3 Tabletop SEM
The tabletop SEM uses a less focused electron beam and it is not possible to get as high resolution and the same magnification as in the SEM. This led to images that were quite blurry and somewhat distorted by charging effects and this can be seen in figure 11. Due to this none of the images from the tabletop SEM were of any greater use.

Figure 11: Image from the tabletop SEM showing an edge in the material E9 4mm.

5.4 SEM
The SEM was used to create the images needed to see the pore size and analyses the pores. Images were taken of one of each type of pellet, each of these pellets had been broken along the length of the pellets. These pellets were chosen because of their open bulk. In figure 12 and figure 13 the three different types of Quartzene® at two different magnifications can be seen.
Figure 12: The three different types of Quartzene® at the same magnification (150,000 X) in the SEM. a) CMS 4mm. b) E9 4mm. c) ND 4mm.

Figure 13: The three different types of Quartzene® at the same magnification (20,000 X) in the SEM. a) CMS 6mm. b) E9 6mm. c) ND 6mm.

5.5 Pore Size
The pore size was really hard to establish and no exact figure could be produced. However an approximated value could be established and is showed in the diagram below.

The pore size was measured using a free for all SEM analysis program from JEDL called SemAfore 5.21. In this program the contrast, lighting and histogram in general could be changed. This helped when trying to measure the pores and how the program worked can be seen in figure 14. In table 2 the results from the measurements with this program can be seen. In figure 15 the pore size against the density is plotted and in figure 16 the fracture stress is plotted against the pore size.
Figure 14: Measurements of the pore sizes in the materials, made with the program SemAfore 5.21. The indicated lengths in the image were drawn by hand, not automatically by the program. The length was then given by the program.

Table 2 below shows the measured mean pore sizes and the standard deviation. These measurements were used for the diagram in figure 15 and 16.

Table 2: Measured mean pore sizes and standard deviations for the three different types of Quartzene®.

<table>
<thead>
<tr>
<th></th>
<th>Mean value of Pore Size (µm)</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>CMS (6mm)</td>
<td>0.05890</td>
<td>0.02034</td>
</tr>
<tr>
<td>CMS (4mm)</td>
<td>0.07323</td>
<td>0.02265</td>
</tr>
<tr>
<td>E9 (6mm)</td>
<td>0.26403</td>
<td>0.06315</td>
</tr>
<tr>
<td>E9 (4mm)</td>
<td>0.36170</td>
<td>0.09289</td>
</tr>
<tr>
<td>ND (6mm)</td>
<td>0.68977</td>
<td>0.29666</td>
</tr>
<tr>
<td>ND (4mm)</td>
<td>0.70955</td>
<td>0.21038</td>
</tr>
</tbody>
</table>
Figure 15: The diagram above shows how the density and pore size varies for each pellet. The cross is build out of the standard deviations of each measurement of density and pore size. This means that each type of Quartzene® has a specific range that the pore size and the density should be within.

Figure 16: Shown above is a diagram over the logarithm of the pore size and the logarithm of the fracture stress. The diagram is in a logarithmic scale for the simple reason it looked a lot better.
6 Conclusions and further studies

From the crush-tests and the density/pore size measurements it is possible to see some sort of relationship where a higher density and smaller pores leads to a higher fracture stress. Due to the insufficient data in this study no real conclusions can be made, but the results indicate that there is an interesting connection that can be further studied. In table 3 a compilation of the examined properties for the three types of Quartzene® is presented.

Table 3: Compilation of the investigated properties of the three aerogel-like, Quartzene®-based materials CMS, E9 and ND.

<table>
<thead>
<tr>
<th>Type of Quartzene®</th>
<th>Mean density (g/cm$^3$)</th>
<th>Mean pore size in the mesoporous domain (µm)</th>
<th>Fracture stress compression (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CMS (6mm)</td>
<td>0.879</td>
<td>0.059</td>
<td>2.2905</td>
</tr>
<tr>
<td>CMS (4mm)</td>
<td>0.822</td>
<td>0.073</td>
<td>2.6411</td>
</tr>
<tr>
<td>E9 (6mm)</td>
<td>0.285</td>
<td>0.264</td>
<td>0.0598</td>
</tr>
<tr>
<td>E9 (4mm)</td>
<td>0.295</td>
<td>0.362</td>
<td>0.0711</td>
</tr>
<tr>
<td>ND (6mm)</td>
<td>0.216</td>
<td>0.690</td>
<td>0.0220</td>
</tr>
<tr>
<td>ND (4mm)</td>
<td>0.209</td>
<td>0.710</td>
<td>0.0243</td>
</tr>
</tbody>
</table>

In a previous study of silica aerogels similar compression strengths were obtained. The examined aerogels with a density ranging from 0.10-0.40 g/cm$^3$ had a measured fracture stress ranging between $10^{-2}$-1 MPa. An increase in strength with an increase in density was shown. [20]

When the SEM images were studied a clear difference between the three different types of Quartzene® could be seen. ND had the biggest pores, E9 had the second biggest pores and CMS had the smallest. This can clearly be seen if the images are studied next to each other. The results from SemAfore also pointed to this fact as can be seen in table 3.

In general Quartzene® has shown to be a very brittle material with quite poor mechanical properties. The products that Svenska Aerogel AB manufactures are made from the powder form of Quartzene®. Since these products are formed by casting the powder they do not have the same structure and properties as an intact monolith of Quartzene® would have. The three dimensional structure of the pellet is held up by the mechanical adhesion between agglomerates in a more random way than what could be described in a cellular structure. The way of describing Quartzene® as a cellular structure through a mechanical perspective is no longer valid due to this. As a result, when casted to pellets, the mechanical properties of the material likely depend most on how the casting process has been carried out. Things like cracks in the pellet and the shape of the pellet should contribute to what mechanical properties will be obtained.

The different types of the aerogel-like material Quartzene® that appear in this study are made through casting Quartzene® powder. Therefore it is hard to describe their mechanical behaviour through an existing model, like the model of a cellular material.

This study has been greatly affected by the short time period that it has been carried out through. There are still things that can be done and studied with this interesting material. Due to lack of time there is still room for more testing in order to obtain more statistics and high
quality data. This study gives an indication of the properties of Quartzene® at least and all results should be considered with the lack of both time and sufficient data in mind.

A suggested path for Svenska Aerogel AB is to further study the effect of sintering of Quartzene®. Available studies indicate that sintering will greatly affect the mechanical properties of aerogel-like materials, making them stronger. Sintering could help reaching the structure of the monolith form of the material, of course with a loss in porosity and a more dense material as a result. For applications where a stronger material is required, sintering could be considered as an option. Especially in situations where there is a risk that the pellet will break and turn into powder. Perhaps, for an example, the thermal properties of the sintered pellet (which will not break) are worse than an un-sintered pellet, but better than the powder form of Quartzene®. Therefore in the case when the pellet risk breaking into powder the sintered pellet could be considered an option. However, this is something that came across during this project and even though no further investigations were made in this project it was thought worth mentioning. [21]
7 References


[4] Prof. Christer Sjöström, Research & Development, Chairman of the Board, Svenska Aerogel AB.

[5] Peter Norberg, Chief Technology Officer, Svenska Aerogel AB.


