Quartzene— A promising thermal insulator
Abstract

Quartzene – A promising thermal insulator

Aerogel 1

The purpose of this project was to study Svenska Aerogel AB’s product Quartzene®, and develop its capacity as a thermal insulator. Quartzene® is a silica based mesoporous material developed by Svenska Aerogel AB, with properties similar to aerogels produced by the sol-gel process. In this report, the correlation between pore structure and thermal conductivity in the material has been studied using techniques, such as scanning electron microscopy, focused ion beam, finite element simulations and transient plane source. Its properties are interesting because of the expanding market of insulated vacuum panels; in which Svenska Aerogel AB wish to expand to. It was found that the pore sizes of M21-BU increased after compression, and the pore sizes of M4-0-2 decreased. The pore sizes of M21-BU became so large that the Knudsen effect is no longer of interest, and that could explain the different behaviors in thermal conductivity.
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Notations and Abbreviations

Notations

\( \lambda_{\text{eff}} \) [W/(m·K)] Total thermal conductivity
\( \lambda_s \) [W/(m·K)] Thermal conductivity of the solid
\( \lambda_g \) [W/(m·K)] Thermal conductivity of the gas
\( \lambda_r \) [W/(m·K)] Thermal conductivity of the radiation
\( \lambda_{g0} \) [W/(m·K)] Thermal conductivity of the gas when moving freely
\( \beta \) [1/K] Thermal expansion coefficient for air
\( K_n \) Knudsen number
\( l_{\text{mean}} \) [m] Mean free path
\( \delta \) [m] Pore size
\( k_B \) [J/K] Boltzmann’s constant
\( T \) [°C] or [K] Temperature
\( \sigma \) [J/(K^4·m^2·s)] Stefan-Boltzmann constant
\( P_g \) [Pa] or [bar] Gas pressure

Abbreviations

GDM Gatan Digital Micrograph
FEM Finite Element Method
FIB Focused Ion Beam
PCD Powder Compressing Device
SEM Scanning Electron Microscopy
St. Dev. Standard Deviation
TPS Transient Plane Source
1. Introduction

1.1. Background

Quartzene® is a mesoporous material similar to aerogels. An aerogel is a material with a very low density and a very high porosity. Quartzene®'s structure, and properties, is comparable to other aerogels. Both have a skeletal structure made of porous silica, a very low density, and a very low thermal conductivity. The largest difference is that Quartzene® is produced as a powder, not a gel from the sol-gel process like other aerogels.

Quartzene® was designed by Svenska Aerogel AB with the intention of it being competitive in the filtration industry and then, due to market expectations, developed to be a thermal insulator. Unlike the traditional aerogels, Quartzene® is significantly cheaper to produce, and its chemical properties, in terms of hydrophilicity/phobicity, can be tailored to fit the application. As a thermal insulator, due to it being a powder, Quartzene®’s efficiency is closely related to the degree of compression. Both the intra grain porosity and the inter particle distance affect its thermal conductivity. Therefore the correlation between its thermal conductivity and its degree of compression are of great interest to Svenska Aerogel AB. [1]

1.1.1. Purpose

This project is executed in order to assist Svenska Aerogel AB in further developing Quartzene® as a thermal insulator. This assistance will be given in the form of a study regarding thermal conductivity, compression, as well as a simplified model of the thermal conductivity in the mesostructure of the pore system and its grains.

1.1.2. Goals

Our goal is to present a base to explain the thermal conductivities dependence of density/compression for Svenska Aerogel AB. We hope to deliver traceable and relevant results from several methods of measurement. Svenska Aerogel AB expressed concern regarding the issue of the Knudsen effect. Another goal is to deliver data that is as functional as possible to Svenska Aerogel AB as well as granting a deeper understanding of Quartzene®. We also intend to provide Svenska Aerogel AB with basic research for future studies.
1.1.3. Material choices

In this project two samples were studied: M21-BU and M4-0-2. They were chosen due to the knowledge that their thermal conductivity behaved differently when compressed. It was of interest to find a difference between their pore structures.

1.1.3. Project boundaries

The scope of this project does not include the development of a theoretical model describing the correlation between thermal conductivity and density of Quartzene®. The process of producing powder for analysis is also not a group assignment. The responsibility of supplying the group with a sufficient amount of powder for the analysis falls upon Svenska Aerogel AB. Other limitations are that the group is not able to book the instruments directly and the lab time will depend on how much time the supervisors assigned to us can secure.

1.1.4. Project design

In order to reach our final goal the project has been separated into five different blocks. Each block stretched for approximately two weeks with individual milestones. The first block included research and literature studies to learn about different techniques and about Quartzene®.

The second block was named “Knowledge Base” and included a visit to the offices of Svenska Aerogel AB where we met their specialists and discussed project goals. We were also introduced to the various experts liaisoned to our project. The powder compression problem became a primary concern. In order to prevent further delay during the laboratory work in block three we decided to construct a so called Powder Compressing Device (PCD). Preparing the halftime report was also a part of block two.

The third block was the laboratory block and this block was followed by block four, which contained the analysis of the results from block three. The final report and presentation were created in the fifth and final block.

1.2. Theory

1.2.1. Thermal conductivity in porous materials

To describe the thermal conductivity in porous silica, three different mechanisms of heat transfer must be considered, which all contribute to the total thermal
conductivity \( \lambda_{eff} \). It can be described by Eq. 1: 
\[ \lambda_{eff} = \lambda_s + \lambda_g + \lambda_r. \]
These contributions have been evaluated in previous studies by Simmler et al. (2005) shown in Fig. 1. [2]

Figure 1: Thermal conductivity as a function of compression (density). [2]

In this study, the interest lies upon minimizing the thermal conductivity by compression. As seen in Fig. 1 above, the heat transfer by radiating heat will decrease with compression, while the solid backbone will contribute more and more as the density increases. The gaseous phase, as seen in Fig. 1, is constant. This will be true in most cases, although, if the pore size is sufficiently small, the Knudsen effect must be taken into consideration. For brief information of the Knudsen effect, see chapter 1.2.4.

1.2.2. Finite element method

For this specific project, the module *Heat Transfer in Porous Media* was used in COMSOL Multiphysics. The main equation solved in this module is:

\[
(\rho C_p)_{eq} \frac{\partial T}{\partial t} + \rho C_p \mathbf{u} \cdot \nabla T = \nabla \cdot (k_{eq} \nabla T) + Q \quad \text{Eq. 2}
\]

To be able to solve Eq. 2, the program also solves:

\[
(\rho C_p)_{eq} = \theta_p \rho_p C_{p,p} + (1 - \theta_p) \rho C_p \quad \text{Eq. 3}
\]

\[
k_{eq} = k_p \theta_p + (1 - \theta_p) k \quad \text{Eq. 4}
\]
1.2.3. Former use of a syringe as a compression device

The method of using a syringe to compress aerogels has been performed before by Neugebauer, A. et al. [3]. Instead of compressing aerogel powder they used granules, which resulted in a decrease in thermal conductivity from about 24 mW·m⁻¹·K⁻¹ to approximately 13 mW·m⁻¹·K⁻¹. The uncompressed granules had a density of 50 kg/m³ and the minimum of the thermal conductivity was reached at 140 kg/m³ [3]. This is a large reduction in thermal conductivity therefore using granules instead of powder could be of interest. Unfortunately there is no time for us to ascertain if this actually is a fact for the different samples of Quartzene® that were studied in this report.

1.2.4. The Knudsen effect

The Knudsen effect is a phenomenon that arises when the characteristic pore size in a system is significantly smaller than the mean free path of the gas molecules occupying the pores. The limit of interest for Quartzene® is 70 nm [1]. When that is the case the molecules have a high probability of colliding with the pore walls and the resulting thermal conductivity contribution is proportional to the number of gas molecules, i.e. the gas pressure. This will result in lower gas conductivity compared to the case with larger pore size.

In order to calculate the gas conductivity in an aerogel, this effect will have to be considered since the characteristic pore size generally is well below the micron range. The gas conductivity is calculated by Eq. 5.

\[ \lambda_g = \frac{\lambda_{g0}}{1 + 2\beta K_n} \quad \text{Eq. 5} \]

where \( \lambda_{g0} \) is the conductivity of the gas when moving freely, and \( \beta \) is a constant describing the energy transfer between the colliding gas molecules and the pore walls. The Knudsen number (\( K_n \)), described by Eq. 6, is defined as the ratio between the pore size \( \delta \) and the mean free path \( l_{\text{mean}} \). The mean free path is calculated by Eq. 7.

\[ K_n = \frac{l_{\text{mean}}}{\delta} \quad \text{Eq. 6} \]

\[ l_{\text{mean}} = \frac{k_B T}{\sqrt{2} \sigma P_g} \quad \text{Eq. 7} \]

Where \( k_B \) is Boltzmann’s constant, \( T \) is the temperature, \( \sigma \) is the molecular cross sectional area and \( P_g \) is the pressure. [4]
2. Methods

The behavior of M21-BU and M4-0-2 was thought to depend on changes in the macro-/mesoporous region of the aerogel powder. Consequently, the nanoporous region was not studied. The macroporous region required a scanning electron microscope (SEM) to analyze. To see the mesoporous region inside the agglomerates a focused ion beam (FIB) was used.

2.1. Focused Ion Beam

The goal with this method was to, as accurately as possible, describe the mesoporous domain of Quartzene® by analyzing it in two different states, uncompressed and compressed. Another goal was to correlate the pore size to the change in thermal conductivity.

The focused ion beam system that has been used to make cross sections of the samples is based on an ion cannon with a controllable focal point. This focused ion beam is also coupled with a SEM. Combined with a SEM it is called a two beam system. The two beams are focused at an optimized position called the coincident point, and the angles between the beams are usually range between 45-52°. A focused ion beam system produces and directs a stream of high energy ionized atoms of a relatively massive element. Thus focusing them onto the sample both for the purpose of milling into the sample and as a method of imaging. Milling means to bombard a small area of the sample with high energy of positively charged gallium ions. These ions carry a substantial amount of kinetic energy, and therefore they dislodge atoms on impact with the sample. [5]

The ion beam is created from a reservoir of Gallium atoms. The gallium atoms are first heated close to evaporation and afterwards the now gaseous Gallium flows to the tip of a tungsten needle. The Gallium atoms are then efficiently ionized by field evaporating then accelerated by a potential difference down a column and finally the atoms hit the sample. The result of this is a controlled cutting beam. In the milling process, this beam is used to mill sections out of the sample to make the bulk portion available for SEM imaging. [5]

Using the FIB, a cross section of the material on a mesoporous domain (scale 50 μm) was made. The FIB incorporated a SEM, which was used for the imaging of the FIB-milled Quartzene®. The received data was used to image a 2D segment of the Quartzene® material. The images were analyzed in image processing programs which quantified the pore size, shape and spatial distribution. The decision to use FIB as a method for analyzing
Quartzene® was based partially on a recommendation by Sjöström C. and Leifer K., and partially on the availability of the method [1].

Compressing the powder in a reproducible way was the only problematic point so far. This problem was solved by compressing the powder samples using a modified syringe. The desired density was known to us as (0.14 g/cm³).

A focal point in the final results will be the traceability, and all methods will be carried out in accordance with current standards in the cases were standards actually exist.

The data from the FIB-labs was meant to be used in the Finite Element Method (FEM) calculations, but the complexity of the COMSOL software made success to apply the experimental data for the FEM model a lot harder to achieve.

Before the labs research of the FIB/SEM apparatus and the surrounding factors such as sample preparation and handling was done. Two days of FIB-labs were performed and after these two labs analysis and interpretation of the data became our primary concern.

2.2. Scanning Electron Microscopy

A well known method to study the surface morphologies of a material is SEM. This method is appropriate for this project due to the rapid scan rate and the easy usage compared to e.g. the transmission electron microscopy. The advantage of SEM is also that the untrained student will achieve a sharp image and will not have any difficulty in interpreting the SEM images [6]. Previous image analysis of Quartzene® has been successfully carried out with SEM [7].

The surface of the sample is scanned with an electron beam. When the beam collides with the surface, backscattered electrons, secondary electrons, Auger electrons, etc. are emitted. The information from these electrons are collected and analyzed to generate an image. [7]

2.3. Transient Plane Source

Our goal in using the transient plane source (TPS) method, for analyzing the thermal conductivity of Quartzene® is to use a stable and reproducible method of analysis. TPS will perform fast, accurate measurements of samples and as such can be used to take several measurements in a short amount of time.

The TPS will however not be used by us. Svenska Aerogel AB has a TPS in their office and they have supplied us with the data we required.
The TPS method consists of a device in which a TPS element is embedded in two equally thick layers of compressed powder sample. The embedded TPS element works as both a heat source and as a temperature sensor [8].

The TPS was chosen to analyze the thermal conductivity of the samples as described by Gustafsson S. E. [8]. This, due to recommendation by Sjöström C. [1], was substantiated in a comparative study of methods for analyzing nanoporous silica materials, which shows that the method is indeed well suited for macro-/mesoporous powders [9].

2.4. Finite Element Method

FEM is a method used to solve differential equations by dividing a system into a large number of small geometries, so called finite elements. The equation is then solved for each individual element and the results are added up to receive an approximated solution for the system, thus, an exact solution is not possible to create. The calculations can be carried out using different software.

In this project, COMSOL Multiphysics 4.3b was used. COMSOL can give a solution to a problem even if the problem is wrongly formulated. This can make it difficult for the user to evaluate the plausibility of the solution.

A major challenge with FEM was the knowledge background, the theory behind COMSOL and the time limit in concern.

The goal with using FEM is to achieve a model that can be used to calculate the thermal conductivity of Quartzene® under different circumstances.

Because of the flexibility of the method and the software it is possible to do calculations in a large variety of settings in COMSOL. In our case it would be possible to, e.g. modulate a change of the dimension and properties of Quartzene®, or, change the properties of the surroundings. This is useful when trying out different settings without having to produce them in an experimental environment.

2.5. Powder Compressing Device

The PCD is a device designed by Aerogel 1. The device is essentially a modified syringe. An instruction for the PCD is located in Appendix D. The syringe was modified in three steps:

1. The end of the syringe was cut off. This procedure was carried out attempting to alter the final volume of the syringe as little as possible.
2. A “cap” was created by lathing a piece of plastic to fit the cut end of the syringe. The idea here was that the final capped syringe can hold exactly as much volume as the unmodified syringe, thus enabling us to use the depicted scale to determine the volume.

After the powder was compressed to the desired degree, the cap was carefully removed. At this point the compressed powder could be pushed out gently with the plunger. Since all materials used in the device were commonly found in a laboratory, we felt that there should not be any problems cleaning and maintaining the device.

![Figure 2: The powder compressing device.](image)

Fig. 2 illustrates the PCD and its cap. This device was used to compress the Quartzene® during the FIB- and SEM-labs.

In this case, we knew the desired density (0.14 g/cm³), and therefore, all we needed to do was to compress a predefined weight of powder to a desired volume. We feel at this time that the PCD should meet the requirements for reproducibility.
3. Results

3.1. Focused Ion Beam

The samples were coated with an Au/Pd alloy. An instruction of the FIB-lab appears in Appendix A1.

Table 1: Data of the density and the tapped density.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Compressed</th>
<th>Density [g/cm³]</th>
<th>Tapped density [g/cm³]</th>
</tr>
</thead>
<tbody>
<tr>
<td>M21-BU</td>
<td>Yes</td>
<td>0.14</td>
<td>0.077</td>
</tr>
<tr>
<td>M21-BU</td>
<td>No</td>
<td>0.06</td>
<td>0.077</td>
</tr>
<tr>
<td>M4-0-2</td>
<td>Yes</td>
<td>0.14</td>
<td>0.128</td>
</tr>
<tr>
<td>M4-0-2</td>
<td>No</td>
<td>0.11</td>
<td>0.128</td>
</tr>
</tbody>
</table>

Tab. 1 shows the data of the density and the tapped density for the different samples. The density of the uncompressed samples and tapped density was received from Svenska Aerogel AB. The density of the compressed samples was calculated.

Table 2: Data obtained from Gatan Digital Micrograph.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Compressed</th>
<th>Average pore size [10⁻¹⁵ m²]</th>
<th>St. Dev. Pore size [10⁻¹⁵ m²]</th>
<th>Average amount of pores</th>
<th>St. Dev. pores</th>
</tr>
</thead>
<tbody>
<tr>
<td>M21-BU</td>
<td>Yes</td>
<td>5.07</td>
<td>1.11</td>
<td>136</td>
<td>24.0</td>
</tr>
<tr>
<td>M21-BU</td>
<td>No</td>
<td>3.21</td>
<td>1.26</td>
<td>90.1</td>
<td>42.0</td>
</tr>
<tr>
<td>M4-0-2</td>
<td>Yes</td>
<td>2.66</td>
<td>0.41</td>
<td>135</td>
<td>44.7</td>
</tr>
<tr>
<td>M4-0-2</td>
<td>No</td>
<td>3.00</td>
<td>0.42</td>
<td>274</td>
<td>75.0</td>
</tr>
</tbody>
</table>

The data in Tab. 2 concerns average pore sizes, average amount of pores, as well as standard deviation of them both. M21-BU and M4-0-2 are two different kinds of Quartzene®, see Fig. 10 in 3.3. These were calculated from the data in Appendices E1-E4.

A diagram was constructed with error bars for a clearer illustration of the average pore size and the standard deviations from Tab. 2. This diagram is illustrated in Fig. 3, on the next page.
In Tab. 3 we have used the data from Gatan Digital Micrograph (GDM) to calculate porosity. It has been carried out by multiplying the average pore size with the average number of pores. Pores per volume were calculated with: \( \frac{\text{porosity}}{\text{area}} \), where the area is the total size of the images analyzed. The density in this case has two different sources. The density 0.14 g/cm\(^3\) was calculated by us when compressing the powders. The densities 0.06 g/cm\(^3\) and 0.11 g/cm\(^3\) are both taken from the graph in Fig. 10, supplied by Twumasi, E. from Svenska Aerogel AB [7].

Table 3: Data calculated from contents of Tab. 2 and Tab. 4.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Compressed</th>
<th>Density [g/cm(^3)]</th>
<th>Porosity ([10^{-21} \text{ m}^3])</th>
<th>Pores/volume [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>M21-BU</td>
<td>Yes</td>
<td>0.14</td>
<td>5.81</td>
<td>10.9</td>
</tr>
<tr>
<td>M21-BU</td>
<td>No</td>
<td>0.06</td>
<td>2.43</td>
<td>4.55</td>
</tr>
<tr>
<td>M4-0-2</td>
<td>Yes</td>
<td>0.14</td>
<td>3.02</td>
<td>5.65</td>
</tr>
<tr>
<td>M4-0-3</td>
<td>No</td>
<td>0.11</td>
<td>6.96</td>
<td>12.7</td>
</tr>
</tbody>
</table>
Fig. 4 is constructed from average pore size from Tab. 2 and density from Tab. 3.

Table 4: Data obtained from Gatan Digital Micrograph.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Compressed</th>
<th>Average shape</th>
<th>St. Dev. shape</th>
</tr>
</thead>
<tbody>
<tr>
<td>M21-BU</td>
<td>Yes</td>
<td>0.78</td>
<td>0.03</td>
</tr>
<tr>
<td>M21-BU</td>
<td>No</td>
<td>0.85</td>
<td>0.04</td>
</tr>
<tr>
<td>M4-0-2</td>
<td>Yes</td>
<td>0.87</td>
<td>0.04</td>
</tr>
<tr>
<td>M4-0-2</td>
<td>No</td>
<td>0.83</td>
<td>0.03</td>
</tr>
</tbody>
</table>

The data in Tab. 4 includes average shapes and standard deviations of compressed and uncompressed Quartzene®. Shapes in this case are illustrated by a number. The closer 1 the number is, the more circular it is. Numbers around 0.8 suggests a more square like shape, and shape numbers above 1 suggest a circle forming a bit of a waist.
The diameter in Tab. 5, on the previous page, was calculated from the average pore size in Tab. 2 according to Eq. 5:

\[ D = 2r = 2 \sqrt{\frac{\text{Area}}{\pi}} \quad \text{Eq. 5} \]

These diameters were calculated in order to compare the samples with each other to find out if the Knudsen effect was of concern or not. A complication of all data from the FIB-labs, see Appendices E1-E4.

![Graph showing change in porosity for uncompressed and compressed samples](image)

**Figure 5: Diagram illustrating the change in porosity for the uncompressed and the compressed samples.**

To illustrate the behavior of the two different powders a diagram was constructed, see Fig. 5. The porosity is shown in percentage of the powder, and the compression just if it was compressed or not. The graph is used to provide the difference between compressed and uncompressed M21-BU and M4-0-2. It was evaluated by adapting a straight line between two measurements. It is noteworthy to mention that the values between “Yes” and “No” does not necessarily behave linearly.
Fig. 6 illustrates two compressed agglomerates of the compressed sample of M4-0-2. In this image, we can see with the naked eye that the interface between the two conjoined agglomerates contains a larger average pore size than the surrounding volumes. For more information about image processing, see Appendix A2.
3.2. Scanning Electron Microscopy

The images were taken using a Zeiss Merlin SEM. The samples were uncompressed powders coated with a Au/Pd alloy. An instruction of the SEM-lab appears in Appendix B1.

Figure 7: Mag. 1,000x. M4-0-2 is to the left and M21-BU to the right.

The images in Fig. 7 illustrate the agglomerates in the two aerogel powders with a magnification of 1,000x for the uncompressed samples. For larger images from Fig. 7, see Appendix B2.

Figure 8: Mag. 20,000x. M4-0-2 is to the left and M21-BU to the right.

The images in Fig. 8 illustrate the agglomerates in the two aerogel powders with a magnification of 20,000x for the uncompressed samples.
Figure 9: Mag. 25,000x. M4-0-2 is to the left and M21-BU to the right.

The images in Fig. 9 illustrate the agglomerates in the two aerogel powders with a magnification of 25,000x for the uncompressed samples.

3.3. Transient plane source
The following results, in Fig. 10, were received by Svenska Aerogel AB.

![Diagram](image)

Figure 10: Diagram constructed from data supplied by Svenska Aerogel AB. [7]

3.4. Finite Element Method
The figures 11 and 12 have been created by following the instructions in Appendix C.
Figure 11: This diagram illustrates the relation between the effective thermal conductivity and the volume fraction of bulk material. The relation is described in Eq. 4.

Figure 12: Temperature gradient of the model material for volume fraction with 0.02 of bulk material.

Fig. 12 shows the temperature gradient of the model material for volume fraction 0.02. This is the main result from using the module Heat Transfer in Porous media in COMSOL.
4. Discussion

4.1. Focused Ion Beam

Our results seem to suggest that M21-BU’s porosity increases with compression. And M4-0-2’s porosity seems to decrease with compression. This, in addition to the calculated pore diameters in Tab. 4, seem to suggest that M21-BU not only increases in porosity, but that the pore size increases to the point where the Knudsen effect is no longer of concern due to it being larger than 70 nm. This limit regards where the Knudsen effect no longer is of interest. In total, these results could explain why empirical studies of M21-BU show that the thermal conductivity becomes smaller when compressed.

One comment regarding the standard deviation of average pore size is that we have not researched whether the size of pores vary as a function of their position in the volume. The large standard deviation in this case is not necessarily a sign of error, but rather that the sizes of the pores have a broad distribution. This distribution was not researched in this project. If the pores do vary upon that, or perhaps depending on where in the volume they are, this could be an explanation as to why we have a relatively large standard deviation. The reason we have not researched this is due to lack of time and general experience of image analysis.

Furthermore, we have not researched whether the pore sizes vary with different agglomerate sizes. The agglomerates in the powder are of a rather broad distribution in terms of size. As we have not analyzed more than one agglomerate per sample, these differences are not recorded or handled in any way. In the case of uncompressed M4-0-2, see Appendix A4, it is clearly visible that the agglomerate that was analyzed is substantially smaller than the other ones analyzed. If agglomerate size causes a change in the pore size distribution it could be a future area of interest for further research in attempting to create an accurate model of Quartzene®.

The standard deviation issues discussed above also apply in the case of average amount of pores. In this case a much larger number of agglomerates would have to be analyzed in order to cast any light on how representable these standard deviations as well as for the reliability of the analysis.

All together, the issue with these standard deviations is overshadowed by the fact that our methods have not been as extensive as they would need to be in order to yield
statistically significant, or otherwise, reliable data. In addition to this we are hopelessly under qualified for this analysis or even practical use of a FIB.

Average shape displayed here has a very low standard deviation which of course is due to the pores having a rather uniform shape. The shape concept basically means that the closer to 1 a pore is, the more circular it is in shape and a more circular shape means a more spherical pore. At approximately 0.8, the shape of the agglomerates was square like. This square like shape is believed to be the result of the pores in the images being so small in the image itself, that the square nature of the actual pixels affects the outcome of this measurement. When looking at an image we can clearly see that the pores are not squares.

The porosity and pores per volume calculations here are results we have calculated from the results yielded in GDM. In other words they are not something the software has calculated. As a project group we simply felt that we needed a concept more commonly used in the industry.

The behavior displayed in Fig. 4 is very similar to the trend of the measurements supplied by Ebenezer in Fig. 10. This can be a result of the pore size being proportional to the thermal conductivity.

4.1.1. Sources of errors

Doubt is cast upon the studies by the methods used in applying the powders to the sample holders. Since our studies concern the compression of the powder, the actual force of applying the powder to the adhesive upon the conductive tape. It is likely to cause changes in the mesoporous region of the powders. This uncontrolled factor of cause reduces the repeatability of our research. In order to obtain standardized and repeatable measurements this would surely be a focal point in future projects.

The problem of compression is very hard to remedy. The powder is nonconductive, and therefore a very thin layer of a Au/Pd alloy is necessary to sputter on the samples. Otherwise, the FIB apparatus will not work on Quartzene®. A thin layer of Au/Pd alloy means that the sample is compressed even more. The powder was mounted from the adhesive when sputtered with Au/Pd and would not be likely to stay unmoved by the decompression inside the FIB or indeed by the force of the ion beam itself. When the samples was mounted on the adhesive and sputtered with Au/Pd.

Results from uncompressed M4-0-2 are not as reliable as the results from the other powders. We have fewer images from this material due to curtaining effects and tilt errors.
On the second day of the FIB-labs these problems were solved but no further time was available to remake the images of the uncompressed M4-0-2.

Since the results are based on data from GDM, which provides a simplified picture of reality, any errors from GDM affect all of our calculations and conclusions. And since we lack knowledge in image analysis, errors in this step are likely to be substantial. In further analysis of Quartzene® we recommend the involvement of an expert in the area of image analysis.

In addition to this step in the GDM analysis, is that the remove edge particle step in the analysis literally removes particles around the edge of the image, and therefore, removing a small amount of pores in each image. These removed pores are not a steady number and vary from image to image.

The density of the uncompressed powders was taken from raw data from Fig. 10. This is probably a density near to our uncompressed powder, but still an estimated value. To make more precise conclusions from Fig. 4, you would need to have measured values of the density and not approximated data. In addition to measured values of the density it would be interesting to have more data from additional images to get a more reliable result from Fig. 4 and Fig. 5.

As the time given to these FIB-studies was limited we could only analyze a few grains of each powder. This leads us to believe that the statistical significance of the studies is not necessarily representable of what is actually happening inside the agglomerates.

Future improvements to our research or additions could be analyzing a greater number of grains. Perhaps a better way of increasing the conductivity of the powders could be devised granting the option of analyzing a larger volume of powder. More cuts with the FIB could also help increase accuracy but the main concern here would be the possibility of analyzing a sample compressed in a completely repeatable manner.

Another field of interest could be researching the differences between pores located near the surface of the grains versus pores located in the bulk, or perhaps the interface occurring between two or more grains when forced together. One of the grains we studied in this project suggests this interface between two grains. It could be an essential factor to the increase in porosity for the compressed M21-BU.
4.2. Scanning Electron Microscopy

In Fig. 7, the agglomerates appear to lie closer to each other in M4-0-2 than in M21-BU. As a result of this, M4-0-2 seems to be denser than M21-BU, which corresponds to the given data. M4-0-2 has a tapped density of 0.128 g/cm$^3$ and M21-BU has a tapped density of 0.077 g/cm$^3$. The main problem with the image analysis is the difficulty to verify the depth of a 2D picture. Larger images are found in Appendix B2.

The images in Fig. 8 and Fig. 9 show that M4-0-2 is smoother on the surface than M21-BU. The M21-BU indicates a more porous structure due to the jagged surface. This statement is questionable because of the few images analyzed. A quantitative image analysis was not possible to do due to time- and knowledge issues. Further image analysis could be done by using fractal analysis.

The agglomerate size is ranged from approximately 1 μm to 30 μm for both samples and with a quick look at the images in Fig. 7, regarding the magnification of 1,000x, the M4-0-2 seems to have more small agglomerates, relative to M21-BU. This is a very rough assumption and not very valid due to the non-quantitative analysis. An interesting point is to verify if and how the porosity relates to the agglomerate size.

The reason of the doubtful SEM-analysis is because of the complexities of practical issues in the lab and difficulty to achieve relevant images.

4.2.1. Sources of errors

The sample preparation with our PCD was successful, and considering the small amount of powder lost during compression, due to powder sticking to the wall, the compression should be easily repeatable. Some problems occurred when the compressed sample was transferred to the sample holder. The sample fell apart to smaller pieces due to the low compression. If this affects the pore structure is unknown for us. Another difficulty was to get the sample to attach to the sample holder, two different types of adhesives was tried out, but with limited success.

The aerogel powder is nonconductive, which means that it was necessary to cover the sample with a thin layer of electrical conductive material, Au/Pd. The Au/Pd alloy was applied using sputtering but during sputtering a vacuum is necessary. Unfortunately, due to the poor attachment, the vacuum sucked out the aerogel samples.

Since the sample did not attach well to the sample holder, a small amount of force was used to make the sample attach better. This means that the structure and compression
changed so repeatability, at this stage, was lost. How much impact the applied force had is unknown to us.

During the SEM lab, more difficulties occurred. One of those was that the sample got overcharged really quickly and another phenomenon was that the sample vibrated. These two difficulties affected the quality of the images.

4.3. Transient plane source

M4-0-2’s thermal conductivity increased linearly with increased compression. While M21-BU displayed a different relation, with increased compression, its thermal conductivity decreased to a density of 0.14 g/cm³, as displayed in the diagram in chapter 3.3, in Fig. 10. This behavior makes M21-BU to a material of interest for use in thermally isolative materials.

4.4. Finite Element Method

When using COMSOL, one is limited to using existing modules. Experienced users may modify these modules, making them fit their specific needs. Unfortunately, we do not possess the skills needed to do this. Because of this, our work in COMSOL is based entirely on the default module Heat Transfer in Porous Media.

In this module a linear equation is used to calculate the effective thermal conductivity, $k_e$, see Eq. 4. In Quartzene® and other types of aerogels, the equations for thermal conductivity are much more complex than the one used in this module [10]. Thus, calculating $k_e$ for Quartzene® with this equation will not provide an insight in how pore size and distribution affects $k_e$. Fig. 11 demonstrates COMSOL using Eq. 4.

The main result from the calculations in the heat transfer module is instead the temperature gradient in the material, see Fig. 12. This result is interesting but not quite what we were looking for in this project.

Even though we did not manage to create a realistic model of Quartzene®, which can be used to calculate its thermal conductivity, we still think that it is plausible to achieve such a model. We have spent much of our project time on learning the software, and we still feel like we have just been scratching the surface. Given a more experienced user with more times at their hands, we are confident that a proper model can be created.

Creating a model for a specific custom material is rather complex and time consuming in COMSOL. Because of this, it might be more efficient to use other methods of
comparison to find the version of Quartzene® best suited for thermal insulation. FEM-calculations can then be used at the end of the optimization process to try the material out in different settings rather than using it early on to compare different versions of Quartzene®.
5. Conclusions

In conclusion our data indicates that M21-BU’s average pore size increases when compressed to the density of 0.14 g/cm³, and M4-0-2’s average pore size decreases with compression to the density of 0.14 g/cm³.

Calculations based on this data show that the diameter of M21-BU’s compression induced pore size is so large that it no longer falls within the range of interest for the Knudsen effect.

More time, research and competence would be required in order to make any of these conclusions statistically significant and in any way reliable.

No real conclusions regarding the correlation between pore structure and thermal conductivity could be drawn from the SEM-analysis due to the fact that the compressed samples could not be analyzed thoroughly. Further studies regarding this subject are possible if the problem with sample preparation could be fixed e.g. by using a better conducting adhesive to hold the samples in place during the vacuum procedure.

Trying to calculate the thermal conductivity and material properties of different versions of Quartzene® in COMSOL would not be time efficient using the Heat Transfer Module in Porous Media. However there may be other more suitable modules in COMSOL for calculating these properties.

Using the Heat Transfer Module in Porous Media at a final step of the development will give a way to evaluate the temperature gradient in the material under different boundary conditions for the complete product. This is demonstrated in Fig. 12.
6. References


7. Appendices

7.1. Appendix A

7.1.1. Appendix A1. Instructions for remaking the Focused Ion Beam lab:

The samples were prepared in two ways, uncompressed and compressed. Both ways resulting in a sample mounted on conductive tape on top of a small sample holder.

The uncompressed sample was prepared by gently smearing a small quantity uncompressed powder upon the conductive tape. After, the surplus powder was removed using compressed air.

The compressed powder was first compressed using the PCD, described in chapter 2.5. The powders were compressed to 0.14 g/cm³. After compression the cap was removed and the formed column of powder was ejected. This column was then gently broken in half using a scalpel. The conductive tape was gently dabbed against what was the central volume of the column. Excess powder was again removed using compressed air.

Both uncompressed and compressed powders were then sputtered with an Au/Pd alloy coating. This process was set at 60 s, 2.2 kV, and 20 mA, which resulted in an approximately 9 nm thick layer deposit. This thickness according to a table located in the manual of the device used.

After mounting and preparation, the samples were loaded into the FIB. The actual process of milling and imaging the samples were carried out by Thom Thersleff, as operating the FIB is not within the scope of our knowledge [11].

The images yielded from the FIB-labs were processed in Adobe Photoshop CS6 and analyzed in GDM. The data captured with GDM was exported to Microsoft Excel for processing, in order to calculate average pore size, shape, and standard deviation for the samples.

7.1.2. Appendix A2. Image processing

Adobe Photoshop CS6 was used as image processing software. After the initial processing, further analyses of the images were conducted using GDM suites.

All images were, throughout the process, kept in a lossless .tif format.

Adobe Photoshop CS6 was initially used to modify the images supplied from the FIB; the images were altered from black and white with a 256 color setting to a black and white
with a grayscale setting. The images at this point contained a large amount of high energy noise, which consists of high concentration bright pixels evenly distributed over the image. The problem with these pixels is that they disturb the image processing step in image micrograph. The high energy noise was removed by adding a filter called a Gaussian blur, which essentially blurs out the effect of noise. The Gaussian blur filter was added to an effect of one pixel on all images processed in Adobe Photoshop CS6.

After these steps a square of 300x300 pixels large square of the image was cut out as a separate image. The idea to use a square of 300x300 pixels large images from each of the images analyzed was based on making the analysis in itself repeatable by others. In theory we could have simply cut out the entire surface represented in the image. This however, would lead to different sizes of analyzed surface per image, due to the different size of the particles. On the samples M21-BU (uncompressed), M21-BU (compressed) and M4-0-2 (compressed), four such images fit inside one single surface. The position where this square was taken from was marked and saved in the original picture.

This process was repeated for every image that was analyzed. The amount of 300x300 pixel images differed from sample to sample, due to the size of the objects in the images.

A noteworthy point was that the uncompressed sample of M4-0-2 was analyzed during the first day of the FIB labs, and it had trouble with a so called curtaining effect. A curtaining effect is a vertical, linear distortion, which causes the image to appear horizontally thinner than it is. In short, parts of the image are missing. The curtaining effect, in this case, was thought to have arisen due to the low conductivity of Quartzene®. A problem well anticipated in this study. The curtaining effect was solved, but as a result of it, all the images achieved during day one were not useable. In fact only one of them was initially thought as acceptable according to Thom Thersleff. In addition to this, the images from the uncompressed M4-0-2 were gathered from a very small particle. All images used were taken at 35,000x zoom, but due to the size of the particle in the uncompressed M4-0-2, the imaged surface was smaller than in the other images. This resulted in a lower possible number of 300x300 pixels images from the sample; often as few as one single image was possible.

This produces the inevitable result that calculations of the uncompressed M4-0-2 are of lower significance than those calculations based on the other samples.

The end results are that we have twelve 300x300 pixel images from each of the uncompressed M21-BU and the compressed M21-BU and M4-0-2, but only six from the uncompressed M4-0-2.
In addition to this, three of the images of the uncompressed M4-0-2 were deemed too dark to give any reliable results at all. These images were therefore altered by T. Thersleff using Adobe Photoshop CS6 in order to yield usable data [11].

The darkness in these images was, due to a narrow histogram, where most scales of gray represented were sloping to the darker parts of the histogram. This was altered by cutting away the lightest parts of the histogram (representing the conductive Au/Pd alloy layer on top of the agglomerate), as well as the darkest parts of the histogram (representing the darkest parts in the background), the histogram was then redistributed yielding a more even curve, and so a more evenly scaled hue. After these images were altered, they were analyzed in the same matter as the other images, using GDM.

7.1.3. Appendix A3. Image analysis with GDM

GDM was used to analyze the images from the FIB, mainly to gain information about the porosity of the samples. To ensure the repeatability of the method, the following steps were executed in the same way for every image.

1. Thresholding

GDM was used to separate the solid backbone from pores. The process of separating the pores from the solid backbone was called thresholding and was done by using a histogram. The histogram ideally contains two major color peaks: one for the solid backbone and one for the pores. The idea was to highlight, in the histogram, a color frequency that was representative for the pores. In order to make the images as simple as possibly to threshold, the images were processed in Adobe Photoshop CS6, see Appendix A2.

2. Fill Holes

This process recognizes inconsistencies in pores and evens them out, effectively "filling in" the pores.

3. Remove Edge Particles

This was used to remove the particles that touched the edge of the image. This function is used to minimize the standard deviation of the roundness and geometry of the particle, since edge particles are not representative for the sample.

4. Open

Opens up gaps between pores connected with a thin bridge of pixels.
5. *Find particles*

Used to identify and index the different pores.

6. *Analyze particles*

This function calculates desired values of different properties of the particles found. In our case values for area, perimeter, length, CenterX, CenterY and roundness was calculated.

7.1.4. *Appendix A4. Images examples from the FIB-labs:*

![Image of FIB-labs example](image_url)

<table>
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<tr>
<th>E-Beam</th>
<th>Mag</th>
<th>Det</th>
<th>FWD</th>
<th>05/09/14</th>
<th>Spot</th>
<th>HFW</th>
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<td>15.0 kX</td>
<td>TLD-B</td>
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<td>14:58:57</td>
<td>3</td>
<td>20.3 μm</td>
</tr>
</tbody>
</table>
7.2. Appendix B

7.2.1. Appendix B1. Instructions for remaking the Scanning Electron Microscopy lab:

The powders observed in the SEM were M4-0-2 and M21-BU. Two samples of each powder were planned to analyze, one uncompressed, and one compressed. Compression was done by the PCD to a density of 0.14 g/cm$^3$. This density was chosen because of previous measurements of thermal conductivity performed by Svenska Aerogel AB. Their measurement indicates that different trends occurred in the powders at this density.

The uncompressed aerogel powders were prepared by putting a small amount of powder on the sample holder, which had been fitted with a bit of conducting, carbon based tape. The compressed sample was prepared by using the PCD, as described in chapter 2.5., and was formed to pellets. The aerogel powder is nonconductive, and therefore, the four samples were sputtered in a vacuum chamber to form a coating of an Au/Pd alloy. The sputtering layer was approximately 9 nm thick.

At this point one problem arose. The compressed pellet flew away while preparing the vacuum. This was because they did not stick well enough to the adhesive. A second attempt was made for the compressed pellets by brushing a conducting silver paste between the sample holder and the pellet, although this was also found not very effective.

We therefore chose to look only at the uncompressed samples. Those two samples were put into the SEM. The instrument used was a Zeiss Merlin, but some problems arose in the SEM as well. Charging effects and artifacts were observed on the sample surface due to the coating. This was solved by our supervisor, Thersleff T., who is familiar with the software [11]. Another problem was that the agglomerates had a tendency to vibrate, or even drift away causing blurry images, due to the light weight of the agglomerates.

6 images were taken per sample at different zoom levels to best represent the structure of the samples, and to compare them.
7.2.2. Appendix B2. Larger images from the SEM-labs:
7.3 Appendix C

7.3.1. Instructions for remaking the FEM-model in COMSOL 4.3b:

1. Choose 2D-model
2. Choose module Heat Transfer in Porous Media
3. Choose Time-dependent study

Through this, we can create a desired geometry, and by following the instructions below the same square builds that was used during the project.

1. Create a square with measurements 1x1 cm
2. Add the material SiC that can be found under AC/DC and set the Ratio of specific heat to 1
3. Create boundary conditions Temperature, 273.15 K and General inward heat flux, 50W/m$^2$ for two opposite sides
4. Let the initial temperature of the bulk be 293.15 K
5. Create a global parameter volumefraction and put the value to 0.01
6. Choose the parameter $\theta_p$ as user defined and assign it the value volumefraction
7. Choose the parameter $k_p$ as user defined and assign it the value 0.03
8. Choose the parameter $\rho_p$ as user defined and assign it the value 128
9. To make the pores exist of air choose:
   - Under "Heat Conduction, Fluid" choose the thermal conductivity as user defined and set the value to 0.0257 W/m*K
   - Under Thermodynamics, Fluid choose the density as user defined with the value 1.2 kg/m$^3$, the Heat capacity at constant pressure as user defined with the value 1000 J/kg*K and the Ratio of specific heats as user defined with the value 1
10. Choose extremely fine under Mesh
11. Choose parametric sweep under Study. Choose the parameter volumefraction and choose range from 0.02 to 1 in ten steps
12. Run compute under Study
Many results are now available. Below follows instructions for finding a plot of the thermal conductivity versus the expression volumefraction.

1. Right click at \textit{Results} and choose \textit{1D Plot}
2. In the \textit{Model Builder} a \textit{1D-plot} comes up, right click and choose \textit{Point Graph}
3. Under \textit{Point Graph}:
   - Choose \textit{Solution 2} under \textit{Data set}
   - Choose \textit{All solutions} under \textit{Selection}
   - For data on the y-axis choose \textit{effective thermal conductivity} under \textit{Expression}
   - For data on the x-axis choose \textit{volumefraction} under \textit{Axis source data} and \textit{Parameter value} under \textit{Parameter}
   - Click \textit{Plot}
7.4. Appendix D

7.4.1. Instructions and practical use of the PCD

The practical use of the PCD was as follows:

- The syringe (including the cap) was put in a small Erlenmeyer flask that was put on a scale
- The scale was tarred and the flask with syringe was removed
- The syringe was filled with powder and put back on the scale
- The mass of the powder was measured
- The syringe was removed from the Erlenmeyer flask
- The final compressed volume was calculated based on desired density
- Using this volume, a height of the compressed pellet was then calculated based on the ratio of volume per mm of the syringe
- This height was marked on the syringe with the aid of calipers
- The powder was compressed by pushing the plunger to the marked height
- The cap was removed
- The powder was pressed out from the syringe on a filter paper using the plunger
7.6. Appendix E

7.6.1. Appendix E1. Data for the uncompressed M21-BU.

<table>
<thead>
<tr>
<th>Image</th>
<th>Position</th>
<th>Average pore size $[10^{-15} \text{ m}^2]$</th>
<th>St. Dev. pore size $[10^{-15} \text{ m}^2]$</th>
<th>Average shape</th>
<th>St. Dev. shape</th>
<th>Number of pores</th>
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<td>Total average</td>
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<tr>
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7.6.2. Appendix E2. Data for the compressed M21-BU.

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<th>St. Dev. pore size $[10^{-15} \text{ m}^2]$</th>
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<td>44.67</td>
<td>45.26</td>
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<td>0.02</td>
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### 7.6.3. Appendix E3. Data for the uncompressed M4-0-2.

<table>
<thead>
<tr>
<th>Image</th>
<th>Position</th>
<th>Average pore size [10^{-15} m^2]</th>
<th>St. Dev. pore size [10^{-15} m^2]</th>
<th>Average shape</th>
<th>St. Dev. shape</th>
<th>Number of pores</th>
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<td>45.51</td>
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<td>0.31</td>
<td>347</td>
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<tr>
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<td>42.46</td>
<td>43.94</td>
<td>0.82</td>
<td>0.29</td>
<td>319</td>
</tr>
<tr>
<td>58</td>
<td>1</td>
<td>36.98</td>
<td>37.88</td>
<td>0.83</td>
<td>0.30</td>
<td>319</td>
</tr>
<tr>
<td>58</td>
<td>2</td>
<td>44.67</td>
<td>45.36</td>
<td>0.83</td>
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<td>0.81</td>
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### 7.6.4. Appendix E4. Data for the compressed M4-0-2.

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<th>St. Dev. pore size [10^{-15} m^2]</th>
<th>Average shape</th>
<th>St. Dev. shape</th>
<th>Number of pores</th>
</tr>
</thead>
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<td><strong>11.85</strong></td>
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