Despite the large number of experiments and simulations still several basic questions regarding the mechanical strength of highly porous materials can not be answered with full confidence. Nevertheless we shall make an attempt in this concluding chapter to outline our present view on the subject. This chapter is organized as follows: First the main question of this thesis: **How does the mechanical strength depend on macroscopic size and microscopic morphology** is restated and discussed. Then the main results of this thesis are discussed and placed in perspective. This includes a discussion of the limitations of the present work.

Besides the properties of the basic material five separate aspects influence the mechanical strength of highly porous materials:

- sample thickness
- sample width
- porosity
- pore shape
- pore size

where we defined sample thickness as the sample size in the direction of the applied force, sample width as the sample size perpendicular to the applied force, porosity as the volume percentage of the pores, pore size as a characteristic measure of the inhomogeneities in the material and the pore shape as a measure of the roughness of the inhomogeneities (related to the magnitude of local stress field).

Our discussion is guided by figures that illustrate the different aspects. In these figures deformation stress is applied in the vertical direction. The pores are drawn at random positions to mimic the non-cellular porosity of real samples. All arguments used are also valid, if length scale of pore size and sample size differ by more than one order of magnitude. Note that in every figure only one of the above aspects is altered.
Mechanical strength of highly porous materials

Figure 125. Mechanical strength: influence of sample thickness.

*Thin samples are stronger than thick samples.* The chance that fracture is initiated in a large specimen is bigger than in a small specimen. This can be validated by the Weibull argument, the minimum of two values is smaller than the average, or by the Duxbury-Leath argument, a larger volume has a larger chance of a large critical defect. In both cases it is essential that load is not transmitted if somewhere in the structure a (thin) layer has failed. Some damage accumulation may occur, but it will not be proportional to the thickness of the sample. If height is increased onset of ultimate failure takes place earlier because the number of possible critical crack paths is increased: the weakest link will be weaker. Therefore the strength decreases with increasing thickness. This will ultimately lead to a vanishing strength at infinite thickness.

In chapter 4 we observed that for the spring network simulations this size effect can be described adequately by:

\[ \sigma \sim \left( \ln \frac{\xi}{\zeta} \right)^{-1/\mu}, \]

where \( \zeta \) is a measure of the typical size of the heterogeneities within the structure.

Figure 126. Mechanical strength: influence of sample width.

*Narrow samples are stronger than broad samples.* Again, the chance that fracture is initiated in a large sample is bigger than in a small sample, but now the load-bearing capacity increases with the sample width, resulting in a finite strength for an infinitely broad sample, as already argued by Daniels "2.5 Classical fibre bundle" on page 12.
If porosity increases the effective elastic moduli decrease and in addition fracture will often be initiated at smaller strain. The chance that fracture initiation will immediately lead to ultimate failure can diminish (or enhance, details of which depend, among other things, on the basic material used). The overall effect is likely to be a decrease in strength if porosity increases.

The observation that samples with large pores are weaker than samples with small pores is often explained by the fact that the stress intensity at the tip of an ellipsoid increases if its long axis is enlarged (2.18), but this argument can not be used if the eccentricity of the ellipse is kept fixed (2.18bis). Without damage accumulation the stress fields for the two structures on the left in Figure 129 are similar at equal strain.

In many practical applications samples of identical macroscopic size but with different pore sizes are compared (the two outer samples in Figure 129). Theoretically the strength difference here is caused by a combination of the size effects explained in Figure 125 and Figure 126. Since the two samples on the left are similar, the large sample with the small pores is effectively larger than the large sample with the large...
Mechanical strength of highly porous materials

Figure 129. Mechanical strength: influence of pore size. Here, we will first compare the sample on the left with the large pores with the sample in the middle with the small pores. Comparison of the two outer samples (same macroscopic size and porosity but with different pore size) is discussed thereafter as a decrease in pore size combined with an increment in width and thickness of the sample.

This implies that in a strain controlled deformation experiment, samples with large pores are stronger than samples with small pores. This trend has been observed in three point bending tests on rods of random porous gold [28], but in experiments on less ideal materials the opposite is often found. This opposite effect may originate from several causes, of which we found the following three to be the most likely: possible differences in pore shape (small pores being rounder than large pores); a failure mechanism that is dominated by toughness (this is discussed later); and apparent differences on a length scale smaller than the pore sizes.

The latter can be illustrated with a spring network where heterogeneities on two different length scales are introduced; in addition to randomly omitting springs from the initial configuration, an extra percentage of springs is omitted by randomly placing small hexagons (for the 2-D spring model) on the network. If the size of the small hexagons is proportional to the macroscopic size of the network and all springs that are covered by the small hexagon are omitted, a model system is introduced that exhibits heterogeneities on two different length scales. We will denote the hexagonal pores the size of which are proportional to the system size as macropores, while the pores on the smaller length scale are called micropores. Although we have not carried out this kind of simulations, it is obvious that now a small structure with small macropores is stronger than a large structure with large macropores. On the length scale of the macropores the stress field of the two examples is similar. On the micropore length scale the (macroscopic) size effect discussed earlier causes the largest region to fail at a lower stress, thus a large network with large macropores will be weaker than a small network with small macropores.

If samples with identical macroscopic size and equivalent heterogeneity on the micropore scale, but with macropore sizes that differ are compared, then we expect the fluctuations on the smallest length scale to dominate the overall behaviour.
regions of micropores are large for large macropores. A large structure with large macropores will be weaker than a large structure with small macropores.

Apart from the explanation given above, a size effect in fracture toughness (see [30] or [31] for an introduction) can also explain why thin samples are stronger than thick samples. Close to a crack the local stress field diverges as

$$\sigma_{\text{local}} = \frac{K}{\sqrt{2\pi r}}$$

where $K$ is the stress intensity factor. This stress intensity factor depends on the macroscopic stress applied, the size and eccentricity of the crack and the macroscopic size and shape of the sample. A crack starts to propagate if the stress intensity factor attains a critical value, also known as the fracture toughness $K_c$. Fracture toughness is a material property that depends on temperature and strain rate.

The stress intensity factor for a crack of length $2l$ in the middle of finite plate of thickness $2z$ and width $2w$ is given by [32]

$$K = \sigma \sqrt{\pi l} F \left( \frac{l}{w}, \frac{z}{w} \right)$$

**Figure 130.** Stress intensity factor for a center crack in a finite plate. The stress intensity factor of a crack of length $2l$ in an infinite plate is $\sigma \sqrt{nl}$. For a plate of finite width $2w$ and height $2h$ this has to be multiplied with the corresponding factor $F$. (Figure copied from [32])
Mechanical strength of highly porous materials

Figure 131. Stress field ahead of crack. The analytical stress field diverges close to a crack opening. The flat plateau is caused by the fact that locally the material is not able to carry a load that is higher than some critical value $\sigma_{\text{yield}}$.

where $F$ has been estimated numerically, see Figure 130. This example shows that the stress intensity factor increases when a crack grows. In consequence a plate with a large crack will fail at a lower stress level. If the $l/w$ ratio and $z/w$ ratio are kept fixed, then (5.3) implies

$$\sigma \sim l^{-1/2} \sim z^{-1/2}$$

which is one of the results of Linear Elastic Fracture Mechanics.

Locally the material is not able to carry a stress greater than some critical value $\sigma_{\text{yield}}$. Therefore the local stress field ahead of a crack looks like Figure 131. Beside the stress intensity $K$ and the critical value $\sigma_{\text{yield}}$ this figure also shows the length scale $a$ below which the stress field is cut off. For large non-interacting cracks this cut-off length scale $a$ will be proportional to the size of the crack, but for small cracks $a$ will not vanish, here the size of the process zone will be independent of $a$. As a consequence the macroscopic stress at which the sample fails scales differently in the two regimes.

$$\sigma = \frac{\sigma_{\text{yield}}}{F\left(\frac{l}{w}, \frac{z}{w}\right)} \sqrt{\frac{a}{l}}$$

If two samples with cracks proportional to the macroscopic size are compared, and these cracks can be considered as large, both samples will fail at the same stress ($a \rightarrow \lambda a$; $b \rightarrow \lambda b$) for $a$ small that is when $h \rightarrow h \lambda$; $l \rightarrow \lambda l$; and $z \rightarrow \lambda z$. The macroscopic stress then scales with $\lambda$.

Equation (5.4) shows that for small cracks the 'large' crack effect is included in the scaling. For $a \rightarrow a$; $h \rightarrow h\lambda$; $l \rightarrow \lambda l$; $z \rightarrow \lambda z$ this effect is excluded.

In our case both these scaling relations are significant. By comparing the behavour of the mechanical strength of the material with the same size of the process zone, a more accurate result can be compared.
Conclusion

The stress field diverges close to the fact that locally the stress field diminishes outside a zone whose size is independent of the macroscopic size of the sample, then thin samples will be stronger than thick samples (\(h \rightarrow l/\lambda h; w \rightarrow l/\lambda w; z \rightarrow l/\lambda z; \sigma_{yield} \rightarrow \sigma_{yield} \Rightarrow \sigma \rightarrow \sigma/(\lambda^2)\)). If however the cracks are so small that the stress field only diminishes outside a zone whose size is independent of the macroscopic size of the sample, then thin samples will be stronger than thick samples (\(a \rightarrow \lambda a; h \rightarrow \lambda h; l \rightarrow \lambda l; w \rightarrow \lambda w; z \rightarrow \lambda z; \sigma_{yield} \rightarrow \sigma_{yield} \Rightarrow \sigma \rightarrow \sigma/(\lambda)\)).

Equation (5.5) also teaches that structures of fixed size with large cracks (where 'large' refers to the regime where the cut-off length scale \(a\) is proportional to the crack length \(2l\)) weaken when the crack size is increased. For structures with small cracks this effect is even stronger.

In our discussion above the size of the process zone is put in more or less empirically. Since the physical size of the process zone is one of the parameters that determines the scaling behaviour of the strength, it is by itself already an interesting subject of investigation. Even in model systems as discussed in chapter 4 long range interaction may play a significant role.

Bazant has modified (5.4) in order to explain deviations in the experimental observations. By introducing a transition between a unit strength for thin samples to the scaling behaviour of (5.4) he is also able to fit the experimental data on the effect of the thickness of the sample on the strength. We claim that the approach that results in (5.1) is a more natural way to explain the observed trend. In appendix B both descriptions are compared.

Figure 132. Silica Extrudates: Median strength as a function of direct density. Median strengths are calculated for a standardized length \(L = 10\, mm\). Labels refer to the diameter of the die-holes.
Two problems arise when attention is focussed on experimental data. All strength data exhibit an intrinsic size dependent scatter and manufacturing conditions often have different impacts on samples of different size. This complicates the drawing of decisive conclusions on the size effect. The mechanical strength of silica extrudates discussed in chapter 3 is dominated by interrelated differences in direct density and pore sizes. We chose to normalize these strength data to a standardized length that is independent of the diameter. Alternatively also a standardized length proportional to the diameter could have been chosen, resulting in a slightly different diametrical size effect. In addition our standardisation procedure is based on the \( \mu = 1 \) Duxbury-Leath distribution. Although the residual analyses confirmed that the \( \mu = 1 \) Duxbury-Leath distribution provides a good quantitative description (within error bars) of the effect of the length of the extrudate on its mechanical strength, it hampers decisive conclusions on (the absence of) a diametrical size effect. On the other hand, if the strength scales according to the theoretical considerations and the typical size of the heterogeneities \( \zeta \) in (5.1) is of the order of magnitude of the median pore size then a diametrical size effect is hard to distinguish (a few percent only).

By changing the sintering conditions we succeeded in increasing the strength of the silica extrudates by a factor of 5. What physical change caused this strength increase is partly an open question. Figure 132 shows that the strength of the extrudates increases with the density, but changes in pore size and morphology have been shown to be very interrelated for all batches. Therefore no separate conclusions on the influence of the pore diameter, principal particle size and density can be drawn.

We expect that the strength of samples that have a density far below the density of a random loose packing is mainly caused by the quality of the connectivity between the principal particles. This quality can only improve if the principal particles come closer together locally. This normally results in a macroscopic densification. If two principal particles come together, the distance towards other principal particles will enlarge, i.e. the pore diameter increases.

Recent experimental work of Rong Li and Sieradzki [28] concentrates on the influence of the pore diameter on the mechanical strength. First impression from the published Scanning Electron micrographs is that they succeeded in changing the characteristic length scale within the random porous gold rods without altering other characteristics of the morphology (average porosity and ratio between pore size and principal particle size). Their observations show a clear deviation from (5.1) and (5.4). One of the explanations they propose is that the ligaments comprising the porous gold display elastic-plastic behaviour. Besides this explanation we suggest that it would be useful to investigate whether residual stresses are present within the samples. It might also be very useful to perform a deformation experiment in which the length scale of the effective stress field within the sample is better defined as in a three point bending test. Now the data are analyzed taking the thickness of the rod as the characteristic size of the stress field, but actual thickness of the area of interest might have been a few orders of magnitude smaller.

Apart from the above considerations it should be mentioned that the load-bearing capacity does not only depend on the size of the area of interest, but also on the direction of the local stress magnitude law. This is that codependence of the largest tensile stress and the largest shear stress. The latter is the only effective explanation for the observed diametrical size effect of the loaded silica extrudates.

In paragraph 2 of this chapter we discussed that the Duxbury-Leath macroscopic model because the observed diametrical size effect. A closer inspection of the experimental data shows that the macroscopic size effect is associated with the size scale at which the stress spots within the sample takes place. Although the local stress magnitude law is independent of the structure size, the average stress magnitude law is not. This is because the rigidity of the random porous network has changed due to the local densification. This again to end up at a smaller stress magnitude law. This does not seem to be the case for the tensile stresses they observed because they observed diametrical size effect. The deformation experiment could provide insight into the derivation of the effective stress magnitude law distribution.

Apart from the above considerations it should be mentioned that the load-bearing capacity does not only depend on the size of the area of interest, but also on the direction of the local stress magnitude law. This is that codependence of the largest tensile stress and the largest shear stress. The latter is the only effective explanation for the observed diametrical size effect of the loaded silica extrudates.
All strength testing conditions often have the drawing of decisive extrudates discussed in the literature. We found that is independent of the strength that is plotted on the particular size of the extrudates discussed in the previous paragraph. We recognize that the strength of the largest defect determines the tensile strength. Although this gives a good qualitative explanation of the strength differences observed, we want to stress that tensile strength of large samples will also be influenced by the crack arrest phenomenon.

In paragraph 3.3.2 we applied Duxbury-Leath (μ = 1) analyses, with L instead of V because the ln C term was shown to depend on the diameter. In paragraph 4.3 the Duxbury-Leath distribution was not able to describe the strength of the 3-D spring model because ln C was shown to depend on the height of the beam. This can be linked to the observations made in paragraph 2.5. There we have seen that the accumulation of damage which is coupled to a load bearing capacity results in a shift of the failure distribution. We have seen that this is described by a rotation in a Gaussian analysis plot. In a Duxbury-Leath analysis this results in an increment of the slope when the system size is increased, while in addition the lines become more curved. This brings us to the general question: How does the mechanical strength distribution depend on macroscopic size and microscopic morphology? The chance of a large defect as well as the chance that the growth of this defect leads to ultimate failure is affected by the macroscopic size of the system. Estimation of the first effect is hampered by the distinction of what a large defect is. In fact we are only interested in the macroscopic stress at which the defect will extend. Hereby we are not interested in the removal of weak spots within the sample, but in the stress at which a defect starts to grow sub-critically. Although the simple considerations on the stress intensity at the tip of a defect in a spring network model explained the observed size effect for this kind of ideal system, closer examination of cluster size distributions seems to be essential to get more grip on the observed porosity effect. If a crack starts to grow sub-critically this does not imply that the global structure fails immediately. In the neighbourhood of a growing defect rigidity of the network will be lost. The consequence is that first the remainder of the network has to be damaged before the stress on the semi-broken part rises high enough again to encourage a further growing of the defect. Different parts of the network does not seem to have much interaction at this stage of the fracture process (except for the load they share); in other words: the entire network seems to be built from almost independent columns. The strength distribution of the network can be approximated by calculating (2.31) and (2.32) for the failure distribution of a single column. This describes the influence of the sample width on the failure distribution. Increasing the height affects the failure distribution of a single column, this has to be investigated along the lines of the derivation of the Duxbury-Leath distribution, i.e. by investigating the cluster size distribution in more detail.

The consequence of the above considerations is that we may conclude that the observed diameter dependence of ln C in paragraph 3.3.2 is caused by load-bearing that took place only in the axial direction of the extrudate. This implies that at least part of the theoretical foundation of the Duxbury-Leath distribution is not valid for the extrudates. On the other hand it seems that the axial size effect in the experimental data.
Mechanical strength of highly porous materials

is described accurately enough by the \((\mu = 1)\) Duxbury-Leath distribution. An alternative description based on a Gaussian distribution with size (and porosity) dependent parameters might have a sounder physical basis, but will be more difficult to apply. In a Gaussian analysis (see paragraph 2.5) a fixed point has to be found, which requires more data than a Duxbury-Leath analysis. Therefore we recommend to apply \((\mu = 1)\) Duxbury-Leath analyses to quantify the effect of the length of cylindrical samples. For short extrudates however we expect the Duxbury-Leath analysis to give unreliable results; short cylinders are expected to break like the HCP-cubes, so are spherical samples.

The observed \(L^{d-1} \log z\) scaling behaviour of the amount of damage in the spring network simulations also indicates that the network should be considered as almost independent columns. To what extent this is valid in experimental situations can be investigated by applying ultrasonic methods during a deformation experiment. We have not applied such techniques because the high porosity of the silica extrudates hampers wave propagation, but for less porous samples this technique is a valuable tool to visualize location and intensity of the damage process during a deformation experiment.

Although the number of series of simulations with the 2-dimensional spring model with prestress was rather limited we can draw some important conclusions from them. Structures with prestress are weaker than structures without prestress as was expected. Also we were not surprised that small structures experience the prestress procedure differently from the larger structures. What does surprise, is that the obtained failure distributions show that large structures seem to fail in a similar way to structures without the prestress, whereas the small structures in addition also seem to fail according to a totally different (second) failure mode. This implies that one should be very careful in extrapolating the results of experimental tests on small samples to large scale engineering applications. If inhomogeneities (like the random porosity combined with a non-uniform shrinkage) affect the fracture mechanism, then the load-bearing capacity of small samples can diminish more strongly than that of large samples. In practice it will be very difficult to predict whether and when the scaling behaviour will change. On the other hand, this also reconfirms the engineering practice that a material whose strength shows a small scatter (high Weibull modulus or whatever criterion is used) is seen as being a 'perfect' material.

Finally we would like to mention the applicability of this work in the context of several related fields of interest. Clearly a similar scaling behaviour should be found whenever brittle fracture occurs that is dominated by extreme events intrinsically connected to the heterogeneity of the material. Details however can be very specific, therefore we will mention only some typical examples: porous rocks, bricks, pharmaceutical tablets and composites.

$$\text{Analyzing}$$

Here, questions arise concerning the reproducibility of the observed scaling behaviour from these kinds of experiments. As explained in Section 2.5,

$$F_r \sim \frac{\log \left(\frac{r}{\sigma}ight)}{c}$$  

but now \(F_r\) is unknown. For the Weibull distribution

$$F_r = 1 - e^{-\left(\frac{r}{\sigma}\right)^{\theta}}$$