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Failure mechanism in porous materials under compression: crackling noise in mesoporous SiO_2

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The failure mechanism of porous materials under uniaxial stress has been investigated experimentally. Microporous SiO_2, Vycor, has been subjected to slowly increasing compressive uniaxial stress with stress rates between 0.2 and 12.2 kPa/s. With increasing stress the strain changes stepwise with acoustic emission correlated with each volume collapse. The acoustic emission followed the characteristics of 'crackling noise' with a power law distribution over an exceptionally large interval of 6 decades at the slowest stress rate. The power law exponent is -1.39. Possible applications in mining industry and others are discussed.

Keywords: crackling noise; cracking; mechanical testing; porous media; silicates

1. Introduction

Porous materials are widely used in filtering, separation, medical transplants and others [1–7]. One very widely used class of materials is metal foam, which has found several well-established industrial applications [8–10]. In contrast, understanding porous materials remains one of the great challenges in mining, building industry and geology. Mining is often done in environments containing porous mineral assemblies [11,12], including goethite, pyrite and coal, which may give rise to serious accidents when landslides occur in open mining or when mining shafts collapse. Understanding the relevant failure mechanisms and, what is even more important, to find some structural criteria that are useful for predicting or preventing mining accidents has not been achieved. The common approach is to consider static failure mechanisms. Failure of dense and slightly porous materials is usually induced by shearing, buckling and yielding while highly porous materials collapse internally which can often been understood in simple strut-and-node modes which reduce the geometry of the porous assembly to simple polyhedra as characteristic units [13,14]. The elasticity and failure behaviour of man-made, novel materials has been widely

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investigated [15–18] and applications to building stones and oil bearing geological strata has been well established: most historic building stones are porous and damage is affected when they are mechanically sheared or compressed [19,20], and many rocks such as shales and volcanic extrusions also display high porosity which is important for their use in, say, CO_2 adsorption or as host rock for oil and other liquids [21–28].

A plausible strategy to obtain failure stresses is to determine the elastic moduli as function of porosity and to determine the failure stress as function of this modulus [29,30]. This approach contains significant approximations, such as the fact that a simple relationship between porosity and moduli cannot exist without further quantification of what is exactly meant by 'porosity', i.e. the topology of the holes and cross-linking of the bridges between holes [31]. Nevertheless, surprisingly robust scaling relations seem to exist for most natural and artificial materials. For porosities $\Phi < 0.5$ one finds that the modulus follows a linear relationship with porosity $\kappa/\kappa_{(\Phi=0)} = (1-\Phi/0.5)$ while for greater Φ one finds a power law dependence [31] $\kappa/\kappa_{(\Phi=0)} = (1-\Phi)^{\mathrm{m}}$ with $\mathrm{m} \sim 3.2$ for most natural materials. The failure bending strength is then either proportional to κ or scales with an exponent near unity. The prefactor is some 10^{-3} so that a rough determination for failure shear stress is $\mathrm{f} = 0.6\ 10^{-3}\ \kappa_{\mathrm{shear}}$ [31].

A different approach is taken in this article: mechanical failure is often accompanied by dynamical features which constitute acoustic precursors of the collapse as some 'crackling noise' [32,33]. The typical failure under shear stress is that porous material 'snaps' when exposed to the critical shear stress. Crack propagation is fast and few intermediate states are observed at low temperatures. Snapping becomes more viscous for torsion pendulum experiments at high temperatures near the melting point where grain boundary sliding and dislocation creep become dominant [e.g. 34,35]. Uniaxial compression leads to a completely different picture, however. The collapse of the sample is gradual and progresses by avalanches which can be detected by acoustic emission (AE) experiments. It is the purpose of this article to show that these avalanches follow power law statistics with characteristic exponents similar to those measured in mechanical instabilities in martensites and ferroelastic materials [36–39], critical dynamics in microfracturing [40] and spontaneous AE in volcanic rocks [41].

2. Experimental

We performed slow compression tests on mesoporous silica ceramics. Four prismatic samples of Vycor with a height of 5 mm and areas of 18.23, 29.49, 16.99 and 13.17 mm^2 have been tested at constant stress rates of 12.2 kPa/s, 6.5 kPa/s, 1.6 kPa/s and 0.2 kPa/s, respectively.

The four specimens have a porosity of 40%. Vycor is synthesised via phase separation of a Na_2O-B_2O_3-SiO_2 melt, followed by leaching [42] which leaves a 98% pure SiO_2 skeleton containing interconnected pores of random length, direction and density. An average ratio of pore length l over pore diameter d was reported by Levitz et al. [43] as $\mathrm{l}/d \sim 4.35$. Pores of our samples show a diameter of 7.5 nm and a rather narrow pore-size distribution (from N2-adsorption experiments and

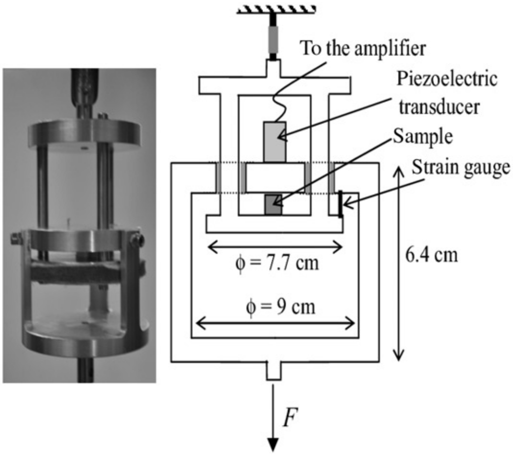

Figure 1. Photograph and schematic representation of the compression arrangement.

BET/BJH analysis). After cutting and sanding, cleaning of these samples was done with a 30% solution of H_2O_2 for 24 h. Drying was done under vacuum at 400 K for another 24 h. The porosity was determined via weighing (accuracy 0.0001 g) samples and measuring dimensions (accuracy 1 μm). The bulk modulus of the sample was 8.1 GPa and the shear modulus was 6.7 GPa as determined by RUS [31,44].

Samples were placed between two aluminium plates as shown in Figure 1. The lower plate is attached to the load cell hanging from the ceiling. The compression force is applied by supplying water at a constant rate to a container hanging from the upper plate, that can move vertically through three Teflon-covered holes that act as guides. By this method we can reach a good control of the stress rate applied to the sample.

The average shrinkage of the sample was estimated by measuring the separation between the two plates using a capacitive strain-gauge. A piezoelectric AE transducer (micro-80) was attached to the upper plate. The electric signals from the transducer were pre-amplified (60 dB) and input in a PCI2 acquisition system from Europhysical acoustic working at 1MSPS. The setup allows for a direct measurement of the energies of the AE events, which are obtained by performing a fast integration of the square voltage of signals detected above a given threshold (26 dB). A more detailed description of this AE setup can be found in [23,27]. The AE activity (counts/MPa) was computed as the number of AE events recorded in an interval of 20 s divided by the stress rate.

3. Results and discussion

An example showing the sample shrinkage and the corresponding AE activity during a compression test is depicted in Figure 2.

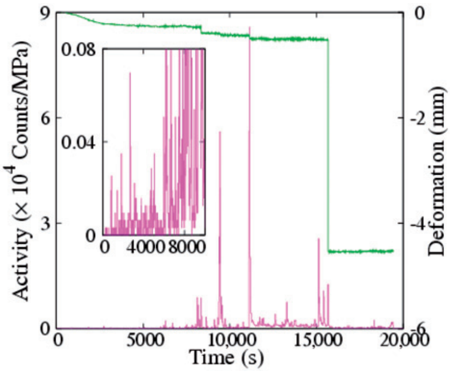

Figure 2. AE activity and sample shrinkage as a function of time, corresponding to the experiment driven at 1.6 kPa/s. The total number of recorded signals in this case is $N = 28{,}652$. The inset shows a detail of the initial part of the experiment revealing low AE activity. The large strain relaxation at 15,670 s corresponds to the big sample crash.

Despite the fact that the AE activity shown in Figure 2 is smoothed by integration over periods of 20 s, the curve still shows a very inhomogenous behaviour. Experiments start with a short period of low activity (as shown in the inset), which is followed by a random intermittent sequence of spikes of high activity separated by periods with low activity. Typically, the activity decreases again to very low values after a big crash involving a change of the average length of more than 50%. This occurs for values of stress about 30 MPa (almost independent of the compression stress rate). Moreover, it is worth noting the existence of a good correlation between high activity peaks and the big deformation drops.

Figure 3 shows the distribution of energies of the individual AE events recorded along the whole test. The log–log plot reveals a linear behaviour for the four studied rates. The total number of recorded events, N, strongly depends on the acoustic coupling between the sample and the plate, as well as the plate and the transducer. Therefore, this number is difficult to be compared from experiment to experiment. In any case, it seems to show a tendency to increase with decreasing the compression rate.

For the experiments in which the number of recorded signals is high enough, the power-law behaviour extends over more than six decades of energies. This result clearly demonstrates that the failure process under compression shows avalanche criticality. The power-law exponent characterising criticality has been estimated using a Maximum Likelihood method appropriate for the cases with a high enough number of recorded events, following the numerical techniques proposed in [45]. Figure 4 shows the behaviour of the estimated exponents as a function of the lower fitting cut-off. As pointed out by the theoretical analysis in [46], this curve should display a kink followed by a plateau extending several decades. Our data obtained at stress rates lower than 6 kPa/s conform this picture. The method renders a very well defined exponent of -1.39 ± 0.02.

Crackling noise in Vycor under slow compression rates is almost equally distributed over 6 decades of stress. For very slow stress rates we find in the initial

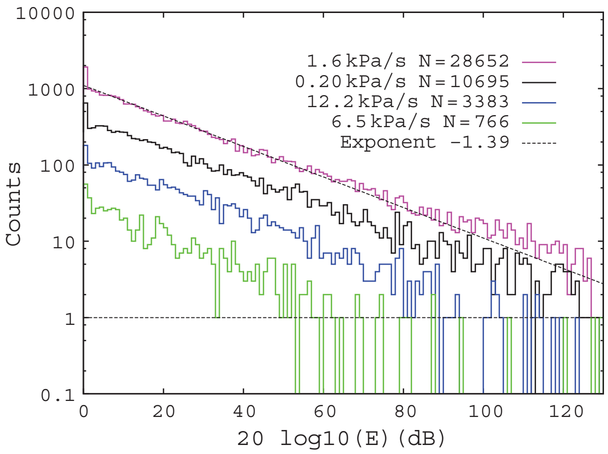

Figure 3. Log–log plot of the energy distribution of the AE events corresponding to the four runs performed at different compression rates. The order of the curves is the same as in the key above. The dashed line indicates the power law with exponent −1.39.

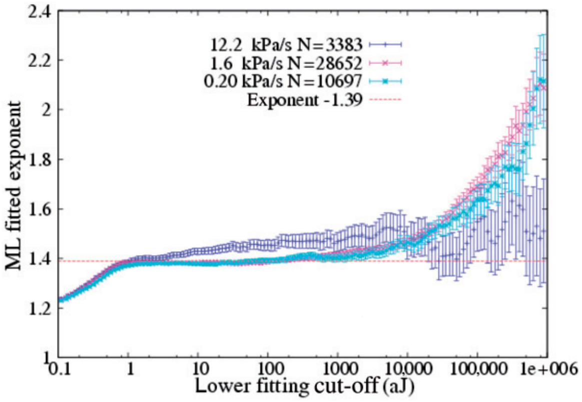

Figure 4. Fitted exponent as a function of the lower fitting cut-off. The dashed line defines the value of the critical exponent.

part of the experiment a lower level of crackling noise intensities. High activities occur both before and after a stress collapses but the level of crackling noise appears not to be correlated with the size of the collapse. The data in Figure 2 show four major events: near 9000 s, 10,000 s, 11,000 s and 15,000 s. Only the latter leads to a complete collapse of the sample while the AE signal is not the biggest for this event. The largest signal near 11,000 s relates to a minor deformation and not to catastrophic failure. Crackling noise does indicate imminent danger of collapse, therefore, but it is more widely distributed and happens also whenever smaller, localised collapses occur. We have not observed collapses without crackling noise either just before or after the event; so we can conclude that acoustic emission is indeed useful as a danger indicator without being able to distinguish between collapses of different size. The open question is then whether this latter result is a feature of Vycor or, possibly, a matter of the finite size of the sample. Further work on other minerals and larger assemblies are planned to clarify this issue.

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