Predicting failure: acoustic emission of berlinite under compression

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Abstract

Acoustic emission has been measured and statistical characteristics analyzed during the stress-induced collapse of porous berlinite, AlPO\textsubscript{4}, containing up to 50 vol% porosity. Stress collapse occurs in a series of individual events (avalanches), and each avalanche leads to a jerk in sample compression with corresponding acoustic emission (AE) signals. The distribution of AE avalanche energies can be approximately described by a power law $p(E)\,dE = E^{-\beta}\,dE$ ($\beta \approx 1.8$) over a large stress interval. We observed several collapse mechanisms whereby less porous minerals show the superposition of independent jerks, which were not related to the major collapse at the failure stress. In highly porous berlinite (40\% and 50\%) an increase of energy emission occurred near the failure point. In contrast, the less porous samples did not show such an increase in energy emission. Instead, in the near vicinity of the main failure point they showed a reduction in the energy exponent to $\beta \approx 1.4$, which is consistent with the value reported for compressed porous systems displaying critical behavior. This suggests that a critical avalanche regime with a lack of precursor events occurs. In this case, all preceding large events were “false alarms” and unrelated to the main failure event. Our results identify a method to use pico-seismicity detection of foreshocks to warn of mine collapse before the main failure (the collapse) occurs, which can be applied to highly porous materials only.

Keywords: acoustic emission, AlPO\textsubscript{4}, porous material, failure

(Some figures may appear in colour only in the online journal)
but is distributed over a multitude of smaller events which combine to fracture the sample. Partial collapse events are named ‘jerks’ and, within the context of out-of-equilibrium dynamics, have been classified as avalanche phenomena. Their statistical features are similar to crackling noise as reviewed by Sethna et al [7]. Jerks are also observed under shear deformations where the microstructure of the sample changes in sudden movements rather than continuously [11, 12]. Salje et al [13] and Baró et al [10] used a porous glass material (Vycor) to show that avalanches under compression follow almost perfect power law statistics (‘crackling noise’) with a characteristic critical exponent similar to those measured in mechanical instabilities in martensites and ferroelastic materials [14–17], critical dynamics in micro fracturing [18], crack growth in heterogeneous materials [19], and spontaneous acoustic emission in volcanic rocks [20]. These results have put the problem of understanding the failure of porous materials under compression firmly within the scenario of crackling noise and avalanche criticality.

The key question in the analysis of compression avalanches in porous materials relates to the existence of precursor effects: is it possible to predict a main event from pre-shocks before the failure event occurs? At first glance one may assume that this problem is similar to the prediction of an earthquake from pre-shocks, where few experimental observations exist in the literature. First observations of foreshock sequences go back to 1988 were a full sequence was observed at the Chalfat earthquake by Smith and Priestly [21] and large sequences of Californian earthquakes by Dodge et al [22]. In each case the statistical evidence was rather limited and related to technical issues of seismological observations. Many large data sets can be obtained from laboratory experiments, such as from observations in porous goethite, FeO(OH), where two scenarios have been identified. Samples with porosity < 60% showed no evidence for any precursor effects and no ‘early warning’ signal could be extracted from the compression noise. Samples with porosities > 60%, on the other hand, did show some precursor noise and opened the possibility to use pico-seismic observations to predict the collapse of a mine [3]. This first study could not identify the physical mechanism which changed the collapsing behavior in weak and strong porous materials, mainly because very few well-characterized natural goethite samples were available to explore the noise statistics in detail. We overcame this problem by using a synthetic porous AlPO₄, berlinite, which could be produced in large quantities, allowing us to explore the collapse mechanism in much more detail than previously possible.

It is the purpose of this analysis to show that the first observations in goethite were indeed correct and that the key to the change of collapse mechanism is related to an approach to critical behavior of the noise pattern near the failure stress. Similar expectations were derived previously from computer modeling by Girard et al [23] on the compressive failure of heterogeneous materials using a continuous progressive-damage model. However, we found increased activities only in samples with high porosity, where the typical power law statistics of the avalanches is much less well realized, than in denser materials where no such increased activities occur. Girard et al [23] argue that the size distribution of damaged clusters leads to a critical interpretation of fracture, with highly increased activity near the failure point. Friedman et al [24] studied the compression of metallic nano-pillars and concluded that the plastic regime followed the behavior of tuned criticality. These two examples show that the observation and understanding of precursor effects near failure points is experimentally extremely demanding and theoretically controversial. It is the purpose of this article to present experimental evidence to show that precursor effects exist in porous materials but only for high porosities.

Our experimental technique for this study is the detection of acoustic emissions (AEs) associated with structural collapse. This experimental technique already has strong statistical similarities with the compression of natural rocks with low porosities and earthquakes over a huge interval of energies from the emitted jerks (see [10] and [25] and references there in). In porous Vycor, the distribution of event energies was shown to follow a Gutenberg–Richter behavior, with no characteristic length- and time-scale. The probability of a jerk with energy E follows a power law \( P(E) \propto E^{-\varepsilon} \) with \( \varepsilon = 1.40 \pm 0.05 \). The energy interval for the power-law behavior in this experiment [10, 13] spans over more than eight decades. Similar experiments in highly porous goethite showed an increase in the energy exponent from \( \varepsilon = 1.68 \) to \( \varepsilon = 2.0 \pm 0.1 \) with increasing porosity. Collapse in the hardest material analyzed so far, alumina (Al₂O₃), equally showed, to a good approximation, a power law distributions for the AE and the measured shape changes [9] with \( \varepsilon = 1.8 \).

2. Sample preparation

2.1. Powder synthesis

AlPO₄ powder used in this study was synthesized through a simple, solution-polymerization route, as reported previously [26–29]. Aluminum nitrate nonahydrate, \( \text{Al(NO}_3\text{)}_3 \cdot 9\text{H}_2\text{O} \) (Alfa Aesar, Ward Hill, MA, USA) and ammonium phosphate dibasic, \( \text{(NH}_4\text{)}_2\text{HPO}_4 \) (Fisher Scientific, Hampton, NH, USA) were the cation sources for AlPO₄. Stoichiometric amounts of precursors were dissolved in deionized water and stirred for 1 h before the addition of the polymeric solution. A 5 wt% solution was made by dissolving 80% hydrolyzed PVA (Sigma Aldrich, St. Louis, MO, USA) in deionized water by stirring for 24 h at room temperature. The ratio of PVA to cation salts in the solution was adjusted in such a way that there were four times more positively charged valences from the cations than negatively charged functional end groups of the organics (in the case of PVA, OH groups). There were more cations in solution than the hydroxyl functional groups of the polymer with which they could chemically bond. In AlPO₄ the total positively charged valences numbered eight. Since each PVA monomer has one (OH) functional group, two PVA monomers were used per each mole of AlPO₄ resulting in a cation valence to anion functional group of 4:1. Gelation did not occur during the reaction, due to the acidic nature of the solution, resulting from the large amounts of nitrates, and also the addition of a few drops of nitric acid (HNO₃), which were added to ensure complete dissolution of the salts.
The precursor solutions were then heated on a hot plate with continuous stirring until the water of the solution evaporated, and a crisp, light-brown aerated gel formed. The temperature of the solution on the hot plate varied during the drying process; however, it was generally < 300 °C. The dried gel was then ground using an alumina mortar and pestle, after which it was calcined in air at 800 °C for 1h. For the first case, calcined powders were then attrition-milled for 1h using yttria-stabilized zirconia beads with propanol as the milling medium, in order to reduce the particle sizes and to increase the specific surface area. They were then dried using a hot plate, with continuous stirring to remove the ethanol, at ~100 °C for 24h and finally stored. For the second case, dried gels were heat treated at 1250 °C for 2h and the particle size was reduced as explained for the calcined powders.

2.2. Bulk sample preparation

In order to achieve differences in the volume of the porosity, four different mixtures were attempted: (i) Bulk samples made with as-calcined and milled powders; (ii) bulk samples made with as-crystallized and milled powders; (iii) bulk samples made by adding 30; and (iv) 50 vol% graphite particles (Aldrich Chemical Company) to as-calcined powders so as to alter the porosity contents. Graphite particles which were used as pore formers in the latter case had a 1.9 g/cc density and a 1–2 μm particle size. In all these cases, 2 wt% of polyethylene glycol (PEG, Mₚ = 200, Sigma Aldrich, St. Louis, MO, USA) was added to the powders and ball-milled with ethanol and yttria-stabilized zirconia as the milling medium (at 100rpm for 24h). The dried, crushed and classified powders were initially compacted using a 19 mm cylindrical hardened steel die, uniaxially pressed under < 5 MPa and cold isostatically pressed (CIP) under 50,000 psi for 10 min (~ 344 MPa). A cold isostatic press (CIP, Model CP 360, American Isostatic press, Columbus, OH, USA) was employed to consolidate the powder particles homogeneously and the high pressure involved also allowed removal of intergranular pores, which would result in inhomogeneity in the bulk samples during sintering. CIPed samples were then heated slowly to 900 °C at a heating rate of 2.5 °C/min to remove the pore formers (graphite particles) and binders and then heated to 1600 °C at a heating rate of 5 °C/min before being held at final temperature for 5h in air.

2.3. Sample characterization

The phase formation and precursor-to-ceramic powder conversion were studied via differential scanning calorimetry and thermogravimetric analyses (DSC/TGA). Powder X-ray diffractometry (XRD) with Cu Kα radiation (Siemens-Bruker D5000) was used to analyze the phases present in the materials. The density of the sintered specimens was measured by the Archimedes method in distilled water at controlled temperature (ASTM C373). Samples were sectioned with a low speed, diamond-tipped saw and cross-sectioned regions were polished by a ‘Buehler Ecomet III’ polishing apparatus, using diamond polishing discs and polishing pads (Buehler) down to 0.25 μm. Scanning electron microscopy (SEM, JEOL 6060LV) was used to carry out the microstructural analysis on the polished surface of the specimens. A few nanometer-thick layer of gold-palladium was coated onto the sample surface, prior to observation, to ensure electronic conductivity. The sectioned surfaces from sintered samples were examined both for microstructural analysis as well as for XRD analysis.

DSC/TGA results showed that there was no weight loss observed above 800 °C in the as-prepared powders and the powders completely lost all of the organic materials well before 800 °C. Powder X-ray analysis (see figure 1) indicated that the AlPO₄ powders prepared by organic/inorganic steric entrapment (the PVA synthesis route) remained X-ray amorphous, even after calcination at 800 °C for 1h. The powders started to crystallize after 1100 °C, as shown in the XRD pattern of the as-prepared powder. Room temperature analyses showed that the powder samples heat-treated at 1200 °C for 1h exhibited a high-temperature cubic tridymite symmetry; whereas, the samples heat-treated at 1550 °C for 1h had a low-temperature tetragonal α-cristobalite structure. This is consistent with the operation of a critical particle size effecting which nucleation of the low temperature phase on cooling is not permitted until
a critical particle size has been exceeded [30]. SEM micrographs of sintered AlPO$_4$ made from as-calcined powders are seen in figure 2. In a microstructure of sintered samples with uniformly distributed fine porosity, pore sizes were < 10 μm. The microstructure clearly revealed that the sintered samples could not be densified under the experimental conditions that were followed, without the help of high pressure or the use of a sintering aid.

The magnified microstructure in figure 2 (right panel) reveals that the grains in the dense region of AlPO$_4$ had grown to more than 10 μm in size. Porous AlPO$_4$ samples resulting from the addition of graphite particles in the AlPO$_4$ matrix are shown in figure 3. The pore shapes and sizes were different to those in AlPO$_4$ sintered without any graphite particles. However, figure 2 indicates that the volume fraction of pores in AlPO$_4$, sintered without graphite particles, was less compared to the graphite-added sample (figure 3). When amorphous powders undergo sintering, in addition to the regular diffusion mechanism, the re-arrangement due to crystallization (volume change) may also induce microcracks, and hence porosity. Bulk density and apparent porosity values, measured by the Archimedes technique, are given in table 1. Preliminary density and porosity analyses show that the porosity values changed with a change in the starting AlPO$_4$ powders, and also with an increased volume of graphite particles in the AlPO$_4$–graphite mixture. Apparent porosity values were calculated using the measured bulk density values and the theoretical density value of AlPO$_4$ (2.566 g/cm$^3$).

### 3. Acoustic emission measurement

The experimental arrangement for the uniaxial compression setup has been described elsewhere [9, 10, 13]. It consists of two parallel circular aluminium plates, perpendicular to the vertical (see inset of figure 4). The bottom plate, hanging from the load cell at the top of the arrangement, was static. The upper plate was pulled downwards by means of three guides sliding through precision ball-bearing elements, mounted on convenient holes drilled in the bottom plate. The pulling
device consisted of a water container acting as a dead load. Small pump rates for the inflowing water enabled the imposition of a slowly increasing load. An acoustic emission sensor was embedded into the upper compression plate, as depicted in the inset of figure 4. The sensor used was a model micro–80 from Physical Acoustics Corporation and it was placed 4 mm away from the specimens. It was encapsulated in stainless steel in order to reduce electrical noise and it had a broadband frequency response extending from ~175 kHz to ~1 MHz (maximum sensitivity of 0.3 V/mbar). A thin vellum layer was used between the compression plate and the sensor and between the sample and the compression plate, in order to ensure a good ultrasonic and acoustic coupling. The signal from the sensor was pre-amplified to 60 dB and input in a PCI-2 system (Europhysical Acoustics, Mistras group, France) operating at 10 MHz and with a digital pass band filter 1 kHz–2 MHz. A laser extensometer (Fiedler Optoelektronik, Germany) measured the vertical separation z between the plates to a resolution of 100 nm. The load cell (1 kN range) signal was read with a lock-in amplifier and had been calibrated with standard weights. Possible noise arising from the friction of the guides with the bottom compression plate was first calibrated using blank experiments. A software filter was then employed to all measurements in order to identify and suppress signals originated from this source.

We performed an avalanche analysis from the acoustic emission signal. The beginning of an avalanche event (hit) was defined as the time \( t_1 \) at which the voltage from the transducer exceeded a predefined threshold (27 dB). The end of the event \( t_2 \) occurred when the voltage remained below the threshold for more than 100 \( \mu \)s. The energy \( E \) of every event was computed as the integral of the square voltage between \( t_1 \) and \( t_2 \), normalized by a reference resistance. The macroscopic compression process was monitored with two averaged quantities: (i) The acoustic activity, measured as the number of hits per time unit (measured during 10s) (ii) The energy emission, measured as the sum of energies of the individual hits recorded every 10 seconds.

4. Results

Results presented in this section were obtained from the specimens listed in table 1. We have checked that similar results could be obtained with samples of the same porosity and comparable geometrical characteristics cut from the same ingots. We have also checked that the results were little affected by small changes in the position and acoustic coupling of the transducer and the studied sample in the compression device. Hence, the location of the transducers and samples was kept identical for all the experiments performed in this study.

The failure stresses of four samples and a theoretical interpolation based on a statistical model by Salje et al [6] are shown in figure 4. In this study, stresses were simply estimated as the ratio of the applied force and the section of the studied specimen given in table 1. In a previous study of porous alumina under compression [9], compressive strengths between 25 and 250 MPa were fitted with the behavior \( P_c = 70 (1–\Phi/100)^m \) with \( m = 3.8 \). In the present case, the same relation seems to apply for smaller compressive strengths \( (P_c < 25) \) but with a higher exponent \( m = 6.5 \). Examples of the AE recordings for four samples are shown in figure 5. The failure of each sample is clearly seen in the upper panel, showing the height \( z \) versus time. The collapse of the sample occurs at the smallest stress for the highest porosity (50%) and increases with decreasing porosity to the samples with 30% porosity (AlPO4–cry). The rapid sample contraction is measured by \( J(t) = (dz/dt)^2 \) where \( z \) is the height of the sample and \( t \) is the time elapsed under constant stress (and is hence proportional to the applied stress). The jerk function (second panel from top) \( J(t) \) represents a first indication of the jerk distribution and is compared with the acoustic activity (third panel) and the energy emission (bottom panel). Several conclusions can be reached simply from visual inspection of figure 5. Along with the compression process, both the acoustic activity and the emission energy exhibit a rather variable behavior, characterized by several peaks that precede the main failure. Even after the failure event some further activity occurs in the debris. For the high density samples (denoted as calc and crys) many large events took place before the failure so that a detailed ‘early warning’ system cannot be envisaged. The number of false alarms was high and randomly distributed. On the contrary, for the most porous samples, the first clear increase in the energy emission and AE activity could be clearly correlated with the main failure.

4.1. Statistics of AE events

The distribution of individual AE jerks over the full compression process was considered for all the studied specimens.
Figure 6 shows the probability function to find a jerk event \( P(E) \) with energy in the interval between \( E \) and \( E + dE \). The curves followed the same power-law behavior, \( P(E) \sim E^{-\varepsilon} \), over five decades with few subtle differences. The power-law exponent, indicated by the straight line, corresponds to the value \( \varepsilon = 1.8 \), in agreement with the value reported for goethite [3] and alumina [9].

The power-law exponents could be obtained from the data using the maximum likelihood method (see 31). The exponent was fitted by considering a higher cut-off equal to the maximum energy measured, and a lower cut-off varying within several orders of magnitude. It is expected that it should exhibit a plateau when the fitted exponent is represented as a function of the lower cut-off.

While the overall trend follows a power law with an exponent 1.8 we found a weak decay of \( P(E) \) for large energies for the most porous minerals. The activity of the acoustic emission was also statistically invariant for all the time intervals and all the samples. This leads to a first conclusion that the emission activity and the overall energy exponents are within experimental uncertainties the same for all the samples and hence for all the porosities. This result agrees with the analysis of micro and pico-seismicity in boreholes by Davidsen and Kwiatek [32]. For further comparison we have equally analyzed waiting time distributions \( D_{E_{\text{min}}} (\delta) \), where \( \delta = t_j - t_{j-1} \) s the waiting time between two consecutive events with energy larger than a given threshold energy \( E_{\text{min}} \) (which takes values from \( 10^{-1} \text{aJ} \) to \( 10^2 \text{aJ} \)). In figure 7, these distributions have been plotted in a scaled representation for all the studied samples and different values of \( E_{\text{min}} \) (indicated in the figure). In order to compare the shape of the distributions we have rescaled the axes as \( (\langle rE_{\text{min}} \rangle)^{-1} D_{E_{\text{min}}} (\delta) \) and \( (\langle rE_{\text{min}} \rangle)^{\delta} \) respectively, where \( \langle rE_{\text{min}} \rangle \) is an average activity given by the mean number of events per unit time with energy \( E > E_{\text{min}} \). For every value of \( E_{\text{min}} \), the number of analyzed hits is indicated between parentheses in the legend. The two black lines show the equivalent waiting times in Vycor with two power law distributions. The short waiting times have an exponent of \(-0.93\), the longer waiting times correspond to a higher exponent \(-2.45\).
4.2. Small time intervals near the big failure for high porosity samples

In figures 8–10 we show the histogram P(E) for the three samples, AlPO$_4$–30, AlPO$_4$–calc and AlPO$_4$–cry, analyzed for limited time intervals close to the big failure. As observed in figure 5, while the observed precursor signals were similar in these three samples, we found that the largest plateau in the exponent versus $E_{\text{min}}$ plots corresponded to the sample with 32% porosity (figure 8). This sample reveals a ‘classic’
power law behavior with a well-defined exponent of 1.4 near the main failure event, while the power law statistics were less defined for times outside this interval (purple curve in figure 8). The overall exponents were larger and fit the average exponents of all curves (1.8). The similar sample with 30% porosity (figure 9) did not follow the same trend, although the effective exponents tended to become smaller near the main failure event for all samples. A weak plateau at slightly higher exponents (1.55) occurred for a sample with 39% porosity in figure 9.

5. Discussion

We have found experimental evidence for precursor effects before a major collapse event in porous berlinite (AlPO₄). The precursor events were found only in highly porous material but not in relatively denser samples. This observation confirms a similar tendency in a typical mining material, namely goethite [3]. Both materials exist in the geological context as porous minerals so that our observations are directly relevant to the observation of warning signs in collapsing mines. The approximate power law dependence of the jerks’ signals encourages us to think that the scale invariant of the observation is, at least approximately, valid, so that small and large geological events can be described by the same power laws. Surprisingly, the collapse of SiO₂-based materials (Vycor) was shown previously to scale with even larger events, such as earthquakes in California [10].

The distinguishing feature of collapses in various porous materials is the time scale over which the collapse occurs.

The strain rate in our experiments was ~ 3×10⁻⁶ m/sec for the most porous berlinite, which is close to man-made stress rates in a mining scenario (typical advance of a mineshaft in coal mining). This strain rate is slower than that of breaking bones in accidents and much slower than shock absorption in porous materials under ballistic impact, say in flack-jackets. It is faster by some three orders of magnitude than tectonic movements, which lead to earthquakes. We are undertaking computer modeling to explore the rate dependence of acoustic emission, which is unknown so far.

The second open question is related to the universality class of the porous collapse. Porous collapse under stress is not necessarily the same universal class as depinning transitions of Barkhausen noise of strongly disordered magnetic materials and plastic charge density wave depinning [33], which is yet more different to the universal class of soft magnets and crystal plasticity. One possible explanation of our experimental finding is that the collapse behavior of porous Vycor (as a prominent member of this class) itself constitutes a universal class, with power law statistics and an energy exponent near 1.4. Such a class would then be observable in berlinite, close to the major failure collapse in materials with porosities near 32%. All other regimes seem to display effective exponents near 1.8 with no significant plateau in the exponent versus $E_{\text{min}}$ curves. One might be tempted to allocate a different universal class to these materials. Significant precursor activity is only observed in this group of materials. An alternative consideration lies in the differences in microstructures leading to the observed differences in mechanical behavior: specifically, grain sizes and porosity, as seen in figures 2 and 3.

The answer to this question has significant implications: it remains unclear whether earthquakes in a given geological region fall into the same universal class. If they do, then we can understand why laboratory experiments reproduce so extraordinarily well the earthquake dynamics. This would mean that we could extrapolate our findings of precursor shocks to some earthquakes (or their absence to others).

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