Crackling noise during failure of alumina under compression: the effect of porosity

Pedro O Castillo-Villa¹, Jordi Baró¹, Antoni Planes¹, Ekhard K H Salje², Pathikumar Sellappan³, Waltraud M Kriven³ and Eduard Vives¹

¹ Departament d’Estructura i Constituents de la Matèria, Facultat de Física, Universitat de Barcelona, Martí i Franquès 1, E-08028 Barcelona, Catalonia, Spain
² Department of Earth Sciences, University of Cambridge, Downing Street, Cambridge CB2 3EQ, UK
³ Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign, USA

Received 14 May 2013, in final form 13 June 2013
Published 2 July 2013
Online at stacks.iop.org/JPhysCM/25/292202

Abstract
We study acoustic emission avalanches during the process of failure of porous alumina samples (Al₂O₃) under compression. Specimens with different porosities ranging from 30% to 59% have been synthesized from a mixture of fine-grained alumina and graphite. The compressive strength as well as the characteristics of the acoustic activity have been determined. The statistical analysis of the recorded acoustic emission pulses reveals, for all porosities, a broad distribution of energies with a fat tail, compatible with the existence of an underlying critical point. In the region of 35%–55% porosity, the energy distributions of the acoustic emission signals are compatible with a power-law behaviour over two decades in energy with an exponent $\epsilon = 1.8 \pm 0.1$.

(Some figures may appear in colour only in the online journal)

1. Introduction
Understanding the failure of materials under compression has important implications for the prediction of collapses in many natural and artificial structures: mines, buildings, bones, etc. At very large scales, even earthquakes can be regarded as produced by failures of the Earth’s crust under compressive stresses. In these examples, the compressed materials exhibit a high degree of porosity which, in many cases, consists of voids with a complex geometry with sizes that range from mm to nm [1]. The problem has, therefore, a multiscale nature which makes it very difficult to formulate quantitative models [2].

Macroscopic observations show that the compression of porous materials leads to sudden partial collapses rather than a smooth elastic or plastic deformation. The partial collapses 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 [3]. Jerks are also seen not only under compression, but also under shear deformations where the microstructures of the sample change in sudden movements rather than continuously [4–6].

One of the experimental techniques that has been successful in yielding an understanding of the statistical properties of jerks is the detection of the acoustic emission (AE) associated with the microcracks occurring in the samples. This experimental technique already revealed strong similarities between the compression of natural rocks with low porosities and earthquakes, more than five years ago [7]. Very recent measurements [8, 9] on the compression of synthetic porous SiO₂ (Vycor) which has a 40% porosity have shown that the event energies and the times of occurrence show strong similarities with earthquake statistics, both concerning temporal correlations (aftershocks) and energy distributions. In particular, the distribution of event energies shows a Gutenberg–Richter behaviour, i.e. a lack of characteristic scales: the probability of a jerk with energy $E$ follows $P(E) \sim E^{-\epsilon}$ with $\epsilon = 1.40 \pm 0.05$. The energy interval for the power-law behaviour in this experiment [8, 10] spans over more than four decades.

Similar measurements on not so well defined materials like the mineral goethite have also been performed [11]. They
lead to slightly higher exponents and a more limited energy interval in which the power-law decay can be measured. Goethite data show an increase of the energy exponent from $\epsilon = 1.60 \pm 0.05$ to $\epsilon = 2.0 \pm 0.1$ with increasing porosity in the range between 55% and 89% [11].

Therefore, at present there is no clear understanding about what factors may alter the energy distribution of AE events. Interesting questions are: Is the degree of porosity a relevant parameter determining the critical properties? Is there a unique porosity value for which the distribution of energies is really critical? What is the influence of the material properties?

The two materials studied so far (Vycor and natural goethite samples) are relatively soft. This raises the question of whether much harder materials follow the same trend. This question is important also for the potential application of the results for the analysis of earthquakes and also impact damage. Faulting during earthquakes evolves from the sliding of minerals against each other. These movements involve the breaking of chemical bonds. These bonds are, in the Earth's crust, in their majority related to Si–O, Al–O and Fe–O, so the sliding mechanism becomes akin to the collapse of porous minerals where similar phenomena determine locally the collapse mechanism. We have investigated the Si–O bond breaking in the synthetic SiO$_2$ material, ‘Vycor’ and the Fe–O bond breaking in the natural mineral goethite. In this paper we will show that the Al–O bond breaking in synthetic alumina ceramics leads to jerk distributions very similar to those in the other samples. We focus in this study on alumina, Al$_2$O$_3$. One of its crystalline polymorphic phases, $\alpha$-Al$_2$O$_3$, is corundum and the only thermally stable, equilibrium phase. Diamond does not form significant porosity, so corundum becomes a material of choice for this study.

Comparing the three materials with vastly different bond strengths we will show that there is no systematic dependence on the bond strength. There is some indication that the degree of porosity does play a minor role in determining the effective energy exponent, however. We will argue that this dependence relates to the criticality of the collapse process and the effect of the cut-off of a limited range of power-law dependences rather than being an intrinsic feature of the topology of the pore distribution of the porous materials.

2. Experimental details

2.1. Synthesis and characterization of the samples

Porous and phase-pure alumina samples were produced using commercially available alumina (Almatis, Leetsdale, PA, USA) and graphite powders (Aldrich Chemical Company, St. Louis, MO, USA) as precursor materials. The fine alumina powders were better than 99.8% chemically pure with an average particle size of $(D_{50})$ 0.45 $\mu$m and a surface area of 8.5 $m^2$ g$^{-1}$, and had a density of 3.98$\pm$0.01 g cm$^{-3}$. Graphite particles were used as pore formers and had 1.9 g cm$^{-3}$ density and 1–2 $\mu$m particle size. Appropriate amounts of graphite particles, namely 20, 30, 40, 50, 60, and 70 vol%, were introduced into the alumina powder matrix to prepare alumina–graphite mixtures by milling in a ball mill (at 100 rpm for 24 h). 1 wt% of polyethylene glycol (PEG, Mn = 200, Sigma Aldrich, St. Louis, MO, USA) was also added to the mixture as a binder and ethanol (99.5%, Acros Organics, Geel, Belgium) was used as a solvent with yttria stabilized zirconia cylinders as milling media. The ball milled slurry was then dried using a hot plate, while continuously stirring to remove the ethanol, and then dried at 100$^\circ$C for 24 h and stored. The dried and crushed powders were initially compacted using a 19 mm cylindrical hardened steel die, uniaxially pressed under less than 5 MPa and cold isostatically pressed (CIP) under $\sim$344 MPa for 10 min.

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 and cracks, which would result in inhomogeneity in the bulk samples during and after sintering. CIPed samples were then heated slowly to 900 $^\circ$C at a heating rate of 1 $^\circ$C min$^{-1}$ to remove the pore formers (graphite particles) and binders and then heated to 1450$^\circ$C at a heating rate of 5 $^\circ$C min$^{-1}$ and held for 3 h. Powder x-ray diffractometry (XRD) with Cu Kα radiation (Siemens–Bruker D5000, Germany) was used to analyse the phases present in the materials.

The average bulk densities and apparent porosity values were measured by the Archimedes method using boiling water (ASTM C373). Table 1 lists the density and apparent porosity of the samples studied.

2.2. Phase and microstructural analysis

In order to analyse the phases present in the processed porous alumina pellets, XRD patterns of the 20 vol% (lowest pore former values) and 70 vol% (highest pore former values) cases were compared with the XRD patterns of the starting alumina and graphite particles. Results are shown in figure 1. Adding the graphite particles to alumina and the processing conditions did not result in any intermediate phase formation.
even though the graphite particle content increased from 20 to 70 vol%. XRD analysis confirmed that the pore former phase (graphite particles) was completely removed during the heat treatment.

Figure 1. XRD diffractometry patterns of porous alumina samples prepared using 20 and 70 vol% graphite particles dispersed in alumina particles, compared with the starting alumina and graphite particles.

Samples were sectioned using a low speed diamond tipped saw and cross-sectioned regions were polished down to 0.25 μm to reveal the microstructure. Figure 2(a) shows the microstructure of sintered alumina samples with uniformly distributed fine porosity (pore sizes were <5 μm). Very fine pores are a common feature in alumina microstructures when sintering takes place without high pressure or sintering aids [12]. In the present case, the fine particle size of the starting material, high surface area and high cold isostatic compaction resulted in ~97% dense material compared to the theoretical density of pure α-alumina. The microstructures from figures 2(b) to (e) show that the porosity level increased linearly as the graphite particle content (pore formers) increased from 0 to 50 vol% as compared to figure 2(a). The pores were very fine (less than or equal to 10 μm) and uniformly distributed on the alumina matrix. Figure 2(f) shows that for 60 vol% graphite content, the pore distribution is less uniform compared with those for less porous samples. This trend was more severe when the graphite particle content increased to nominally 70 vol% as shown in figures 3(a) and (b).

2.3. Compression and acoustic emission set-ups

The experimental arrangement for the uniaxial compressional test has been described elsewhere [8]. It consists of two

Figure 2. SEM micrographs of polished alumina and porous alumina samples sintered at 1450 °C for 3 h. (a) Alumina without any pore formers, (b) 20 vol% graphite particle content, (c) 30 vol% graphite particle content, (d) 40 vol% graphite particle content, (e) 50 vol% graphite particle content, and (f) 60 vol% graphite particle content. All images were observed in the normal SEM image mode under the same conditions at 500× magnification.
parallel circular aluminum plates perpendicular to the vertical direction. The bottom plate, hanging from the load cell at the top of the arrangement, is static. The upper plate is pulled downwards by means of three guides sliding through convenient holes drilled in the bottom plate. The pulling device consists of a water container acting as a dead load. Small pump rates for the inflowing water allowed us to impose a slowly increasing load. Acoustic emission sensors are embedded into the compression plates, centred at a distance of 4 mm from the sample surface. The sensors are acoustically coupled to the aluminum plates with Vaseline. A small amount of Vaseline is also placed between the samples and the aluminum plates. The signal from the sensors is preamplified to 60 dB and input in a PCI-2 system (Europhysical Acoustics, Mistras group, France) operating at 10 MHz.

A laser extensometer (Fiedler Optoelektronik, Germany) measures the vertical separation between the plates with a resolution of 100 nm. The load cell (1 kN range) signal is read with a lock-in amplifier and has been calibrated with standard weights.

Special care has been taken in order to avoid noise from friction of the guides. By performing blank measurements, we have identified the properties of the noise and have designed software filters in order to suppress such signals from the statistical analysis that will be presented.

In order to perform the compression experiments, the original cylindrical samples have been cut using a stainless-steel blade into suitable parallelepipedic specimens. For high porosities, the specimens tend to be more irregular and acceptable sample shapes are only obtained after some trial and error. Details of the specimens studied are presented in table 2. The height of the specimens (z) has been chosen in the range 3–6 mm, which is suitable for the laser extensometer range. Transverse sections (A) have been chosen in order to have failure strengths below the maximum load that can be applied in our set-up (900 N). In order to do systematic comparisons, we have chosen the transverse sections of a series of specimens (2A30, 2A40, ..., 2A70) with different porosities to have similar nominal compression rates in the range 22–47 kPa s\(^{-1}\). Moreover, for the samples with porosities 36.8% and 52.2%, specimens with different transversal sections (F401-F407, F601-F606) have been chosen in order to perform a study as a function of the compression rate in the range 5–80 kPa s\(^{-1}\).

3. Analysis of the acoustic activity

The analysis of acoustic emission reveals that the failure process under compression is rather complex, exhibiting different regimes that can be distinguished by the qualitative measurement of the activity rate (number of signals detected per unit time), the behaviour of the sample height as a function of time \(z(t)\) and the square of the velocity \((dz/dt)^2\). Some illustrative examples are shown in figure 4, corresponding to different porosities and similar nominal compressions in the range 22–47 kPa s\(^{-1}\). Note the logarithmic scale for the AE activity and the square velocity. One can identify the following regimes:

(i) **Adaptation**: After the sample is placed between the compression plates, we have waited \(\sim\)200 s without increasing the load in order to stabilize oscillations of the compressing set-up. This first regime already shows some acoustic activity since the compression plates (and the empty container) already represent a certain load on the sample. It was not further considered for the analysis because it was too difficult to separate the instrumental noise from the signals related to the collapse of the sample.

(ii) **Loading**: The second regime starts with the onset of a constant compression rate and ends after a big failure event or crash. These big failure events reduce the sample height by an amount \(\Delta z\), representing more than a 50% relative variation (>3 mm). The loading regime is not fully stationary: there are sudden steps in \(z\) and/or short transient periods with peaks of acoustic activity (see figure 5). The statistical variability of the AE signal is very large in this regime. In some cases we observe a first run with stationary low activity while the repetition of the experiment with a different specimen differed greatly. The time evolutions of the AE signals were poorly reproducible, which resembles observations of seismicity during earthquakes. The value of the applied force at the start of the major failure event \((F_c)\) defines the compressive strength \(P_c = F_c/A\) where \(A\) is the sample transverse section measured at the beginning of the experiment. The excellent time resolution of our experiment allowed us to measure the duration \(\Delta t\) of

Figure 3. SEM micrographs of a polished alumina with 70 vol% graphite particle content sample sintered at 1450 °C for 3 h.
Figure 4. Examples of compression experiments at 32 ± 10 kPa s\(^{-1}\). Different columns correspond to different specimens. The first row shows the behaviour of the sample height, the second row the behaviour of the square velocity and the third row the AE activity.

Table 2. Summary of the dimensions, compression conditions and measured compression strengths for all specimens studied.

| Specimen | App. porosity (%) | Height (mm) | Section (mm\(^2\)) | Mass (mg) | Stress rate (kPa s\(^{-1}\)) | Comp. strength (MPa) |
|----------|-------------------|-------------|---------------------|-----------|-----------------------------|---------------------|
| A30      | 30.3 ± 0.04       | 3.75        | 5.04                | 48        | 27.40                       | 188.07              |
| 2A30     | 30.3 ± 0.04       | 4.05        | 3.90                | 41        | 34.80                       | 221.39              |
| A40      | 36.8 ± 0.06       | 4.35        | 7.45                | 72        | 18.80                       | 73.84               |
| 2A40     | 36.8 ± 0.06       | 4.50        | 6.00                | 60        | 22.00                       | 92.70               |
| F401     | 36.8 ± 0.06       | 4.35        | 3.96                | 35        | 11.90                       | 91.20               |
| F402     | 36.8 ± 0.06       | 4.10        | 4.00                | 38        | 25.72                       | 93.60               |
| F403     | 36.8 ± 0.06       | 4.20        | 4.09                | 35        | 33.75                       | 87.20               |
| F404     | 36.8 ± 0.06       | 4.05        | 4.00                | 35        | 45.72                       | 80.50               |
| F405     | 36.8 ± 0.06       | 3.90        | 3.71                | 29        | 63.93                       | 94.89               |
| F407     | 36.8 ± 0.06       | 3.75        | 4.20                | 31        | 80.43                       | 86.70               |
| A50      | 45.8 ± 0.04       | 4.65        | 15.44               | 142       | 28.60                       | 51.55               |
| A50DF    | 45.8 ± 0.04       | 4.75        | 15.41               | 143       | 8.00                        | 51.11               |
| 2A50     | 45.8 ± 0.04       | 5.25        | 5.75                | 62        | 25.10                       | 75.41               |
| 2A50R    | 45.8 ± 0.04       | 4.80        | 5.40                | 51        | 41.10                       | 97.98               |
| A60      | 52.2 ± 0.05       | 5.45        | 12.48               | 119       | 12.20                       | 54.74               |
| 2A60     | 52.2 ± 0.05       | 4.85        | 5.06                | 41        | 29.20                       | 49.29               |
| F601     | 52.2 ± 0.05       | 4.00        | 5.06                | 35        | 5.63                        | 63.96               |
| F602     | 52.2 ± 0.05       | 4.40        | 5.18                | 42        | 17.05                       | 56.85               |
| F603     | 52.2 ± 0.05       | 4.50        | 5.18                | 42        | 24.57                       | 64.02               |
| F604     | 52.2 ± 0.05       | 3.90        | 5.18                | 37        | 34.44                       | 67.48               |
| F604R    | 52.2 ± 0.05       | 3.20        | 5.00                | 25        | 37.03                       | 83.14               |
| F605     | 52.2 ± 0.05       | 4.65        | 4.73                | 36        | 47.96                       | 70.58               |
| F606     | 52.2 ± 0.05       | 4.90        | 4.73                | 40        | 67.10                       | 70.05               |
| A70      | 58.8 ± 0.01       | 5.45        | 13.32               | 97        | 12.20                       | 20.49               |
| 2A70     | 58.8 ± 0.01       | 4.95        | 6.37                | 47        | 22.20                       | 29.92               |

the big failure. During this crash we find a multitude of individual AE events, so the major event is not a snapping of the sample, but rather a jerky avalanche event in its own right. The statistical properties of the signals during the crash are not very different from those found in the whole loading regime. In particular, the signals during the crash are not necessarily more energetic than those previous to the crash. An illustrative example is shown in figure 5 that corresponds to an enlarged section of the experiment 2A60 shown in figure 4. The dots indicate the detection of AE events and the colour the energy of each event. These figures show that large precursor events
occur well before the major failure event. After the crash, the sample height reaches again a very stable value that can be identified with a sharp change of the slope in the $z(t)$ plot (see figure 4). We can define an average failure speed $v = \Delta z / \Delta t$. The duration $\Delta t$ of the big failure correlates with porosity: highly porous samples show a shorter crash duration.

(iii) *Pause*: The major failure event is followed by a long period with no AE signals (activity below 1 count s$^{-1}$ in our experiment). This regime lasts longer for higher porosities. We will argue that the duration of the pause $\Delta t_{\text{pause}}$ relates to the momentum absorbed by the sample at the end of the big failure event.

(iv) *Fragmentation*: When the compression load increases further, after the pause, we observe a fourth regime with rather stationary activity. We associate this regime with the consecutive fragmentation of the pieces of the material that are left after the major failure. Within the durations of the experiments presented here (several hours) and at the compression rates studied, we have not found evidence of how this regime ends.

Figure 6 shows the behaviour of the compressive strength ($P_c$) as a function of the porosity ($\Phi$). The observed behaviour is fully compatible with the measurements of Magdeski [13]. The line shows the fit of the behaviour $P_c \propto [1 - \Phi/100]^m$ with $m = 3.8$. This value is in agreement with experimental findings [14] but much larger than the predictions based on isotropic reticular models [16].

We measured, in more detail, the collapse of samples with 52.2% and 36.8% porosity as a function of the compression rate in the range from 5 to 70 kPa s$^{-1}$. As illustrated in figure 7, no significant dependence of the compressive strength $P_c$ on the rate was found.

The study of the failure details allows us to extract some conclusions about the origin of the pause regime. Figure 8 shows the duration of the pause, expressed as a force interval ($\Delta F$) as a function of the impulse ($\Delta p$) transmitted to the sample when the big crash is arrested. This impulse can be estimated by multiplying the falling mass at the failure point ($F_c/g$) by the average speed ($\Delta z / \Delta t$) during the crash. The figure reveals a clear correlation between the two quantities. We argue that the pause in the AE activity is, therefore, due to an effective overshoot associated with this momentum transfer ($\Delta p$) that deactivates all the avalanches that would have occurred within $F_c$ and $F_c + \Delta F$.

4. Energy distributions

In this section, we present the study of the statistical properties of the energies of individual acoustic signals. Figure 9 shows an example of the distribution of energies $p(E)$ corresponding to 45.8% porosity. The data correspond to AE events recorded during the loading regime. The distributions follow approximate power laws over some six decades with extended tails (see figure 9). In order to examine in more detail
Figure 8. Duration of the pause regime (expressed as a force increment) as a function of the impulse delivered to the sample by the falling weight during the big failure event. Labels indicate the specimen from table 2.

Figure 9. Energy distribution of the AE events for a case with 45.8% porosity (2A50R). The histogram corresponds to the signals registered in the loading regime.

Whether or not the distribution tails decay like a power law, we apply the method presented in [15, 10]. The technique consists of studying the behaviour of the power-law exponent ($\epsilon$) fitted with the maximum likelihood method as a function of a lower and a higher cut-off of the data. A true power-law behaviour can then be identified as a plateau (see figures 10 and 11) if $\epsilon$ is stable within error bars for several decades.

Figure 10 shows an example of the behaviour of the exponent $\epsilon$ as a function of the lower cut-off for a higher cut-off of $10^3$ aJ for a $\Phi = 45.8\%$ specimen. The different curves correspond to the analysis of the data in the loading and fragmentation regimes. We also show the behaviour of the data in two subsets of the loading regime: loading-1 corresponds to the loading previous to the big crash and loading-2 to the data acquired during the big crash. As can be seen, a flat plateau can be observed during the full loading regime, and also for the two subsets studied, 1 and 2. Contrarily, the fragmentation regime does not show a clear plateau.

Figure 11 shows a similar analysis of the fitted exponent (in the loading regime) as a function of the porosity. The plateau can be clearly identified for $\Phi = 45.8\%$ porosity. For the other cases, although the plateau is not as extended, by averaging the curves between 0.5 and 100 aJ, we obtain an effective exponent ($\epsilon_{\text{eff}}$) which shows a tendency to decrease with increasing porosity.

Figure 12 presents the energy exponents for $\Phi = 52.2\%$ and $\Phi = 38.8\%$ as functions of the compression rate. No systematic dependence can be found within our experimental resolution.

Figure 13 shows a compilation of the fitted effective exponents in the loading regimes as a function of porosity. Previous data from goethite and Vycor are also shown for comparison. We can observe that the behaviour of the alumina data shows a clear tendency for the effective exponent to increase with porosity, in agreement with previous results in
goethite [11]. Nevertheless, the overall picture suggests that data concentrate around a value of the exponent \( \varepsilon = 1.8 \) for both goethite and alumina.

The quality of the power-law fit (range of the measured plateau in figure 11) for intermediate values of the porosity (\( \Phi = 45.8\% \)) is much better than for much higher or lower porosities. This is an indication that we could encounter a tuned criticality scenario in porous Al\(_2\)O\(_3\): for low porosities \( \Phi < \Phi_c \) the behaviour is supercritical (yielding a small \( \varepsilon_{\text{eff}} \)) while for high porosities \( \Phi > \Phi_c \) the behaviour is subcritical with an exponential cut-off (yielding a higher effective exponent). This behaviour will be compatible with what has been observed in natural goethite. The two materials will share the same value of the critical exponent \( \varepsilon = 1.8 \) and have different values of \( \Phi_c \). On the other hand, the existing data for SiO\(_2\) would correspond to a different universality class, with a value of the exponent which is clearly lower. Further studies are needed in order to identify the reasons behind these differences. Moreover, since avalanche criticality is known to depend on the driving mechanism [17], a comparison of the behaviour obtained under stress-driven and strain-driven conditions is planned for the near future.

5. Summary and conclusions

We have performed systematic studies of the AE during the uniaxial compression on alumina samples with different porosities. The samples have been characterized by using XRD diffraction and SEM. Samples in the form of parallelepipeds were prepared with different heights and compressed under rates between 5 and 80 kPa s\(^{-1}\). The compression process is dominated by a major failure event with a change in height of more than 50%. The signals recorded during the loading regime (including the big crash) exhibit similar stationary statistical properties. The energy distributions of the AE signals were studied by using maximum likelihood techniques. The results show essentially a power-law distribution, at least for porosities in the range \( \Phi = 40\%–50\% \). The energy range of the power-law distribution depends on the porosity and we can speculate that AE during compression of porous Al\(_2\)O\(_3\) exhibits criticality in the loading regime. Criticality would be observed only for a certain value of the porosity (\( \Phi_c \)). For lower porosities \( \Phi < \Phi_c \) the behaviour is supercritical (yielding a small effective exponent), with fewer small acoustic events and big events associated with the failure. For higher porosities \( \Phi > \Phi_c \) the behaviour is subcritical with an exponential cut-off (yielding a higher effective exponent). This behaviour will be compatible with what has been observed in natural goethite. The two materials will share the same value of the critical exponent \( \varepsilon = 1.8 \) and have different values of \( \Phi_c \).

Acknowledgments

We acknowledge financial support from the Spanish Ministry of Science (MAT2010-15114). EKHS thanks the Leverhulme Foundation (RG66640) and EPSRC (RG66344) for financial support. PC-V acknowledges support from CONACyT (Mexico) under scholarship No. 186474. PS and WMK acknowledge a United States Army Research Office MURI grant (W911NF-09-1-0436), through Dr David Stepp. The scanning electron microscopy (SEM) work was carried out in the Frederick Seitz Materials Research Laboratory at the University of Illinois at Urbana-Champaign.

References

[1] Dunlop J W C and Fratzl P 2013 Scr. Mater. 68 8
[2] Girard L, Weiss J and Amitrano D 2012 Phys. Rev. Lett. 108 225502
[3] Sethna J P, Dahmen K A and Myers C R 2001 Nature 418 242–50
[4] Romero F J et al 2011 Appl. Phys. Lett. 99 011906
[5] Harrison R J and Salje E K H 2010 Appl. Phys. Lett. 97 021907
[6] Salje E K H et al 2009 Appl. Phys. Lett. 95 231908
[7] Davidsen J, Stanchits S and Dresen G 2007 Phys. Rev. Lett. 98 125502
[8] Salje E K H et al 2011 Phil. Mag. Lett. 91 554–60
[9] Baró J, Corral A, Illa X, Planes A, Salje E K H, Schranz W, Soto-Parra D E and Vives E 2013 Phys. Rev. Lett. 110 088702

[10] Baró J and Vives E 2012 Phys. Rev. E 85 066121

[11] Salje E K H, Lampronti G I, Soto-Parra D E, Baró J, Planes A and Vives E 2013 Am. Mineral. 98 609

[12] Rahaman M N 2003 Ceramic Processing and Sintering 2nd edn (New York: Dekker)

[13] Magdeski J S 2010 J. Chem. Technol. Metall. 45 143

[14] Salje E K H, Koppensteiner J, Schranz W and Fritsch E 2010 Mineral. Mag. 74 341

[15] Clauset A, Shalizi C R and Newman M E J 2009 Power-law distributions in empirical data SIAM Rev. 51 661

[16] Liu P S 2010 Phil. Mag. Lett. 90 861

[17] Pérez-Reche F J, Truskinovsky L and Zanzotto G 2008 Phys. Rev. Lett. 101 230601