

# Light Metals 2014

**ALUMINUM ALLOYS:  
DEVELOPMENT,  
CHARACTERIZATION  
AND APPLICATIONS**

## **Emerging Technologies**

*SESSION CHAIR*

**Yansheng Liu**

Kaiser Aluminum

Spokane Valley, WA, USA

## DEFORMATION OF OPEN-CELL MICROCELLULAR PURE ALUMINUM INVESTIGATED BY THE ACOUSTIC EMISSION TECHNIQUE

<sup>1</sup>Michal Knapek, <sup>1</sup>Patrik Dobroň, <sup>1</sup>František Chmelík, <sup>1</sup>Mariia Zimina, <sup>1</sup>Josef Pešička, <sup>2</sup>Etienne Combaz, <sup>3</sup>Andreas Mortensen

<sup>1</sup>Department of Physics of Materials, Faculty of Mathematics and Physics, Charles University, Prague 121 16, Czech Republic

<sup>2</sup>Novelis Switzerland SA, Rte des laminoirs 15, Sierre 3960, Switzerland

<sup>3</sup>Ecole Polytechnique Fédérale de Lausanne, Laboratory for Mechanical Metallurgy, Lausanne, CH-1015, Switzerland

Keywords: aluminum foam, compression, acoustic emission, power-law distribution.

### Abstract

Acoustic Emission (AE) is used to characterize the plastic deformation of open-cell pure aluminum foams produced by salt replication. Measurements were performed on samples with cells 25, 75 or 400  $\mu\text{m}$  in average diameter, all with a relative density near 24%. AE signals were measured during compression tests conducted with a constant cross-head speed. Deformation is uniform along the porous metal samples. Recorded AE accompanying plastic deformation of the cellular structure exhibits intermittent behaviour, with the probabilities of AE event energies distributed according to a power-law similar to those previously found in many different materials. The present study thus confirms the universal scale-free character of global plastic deformation dynamics and extends the observation to pure aluminum.

### Introduction

Various cellular materials are used in light-weight structures, in transport industry or in packaging [1]. Metal foams are a relatively novel class of cellular materials with outstanding mechanical, thermal, acoustic and electrical properties due to their combination of attractive features of metals and porous materials. Metal foams are light and stiff, they are good flame arresters and energy absorbers and therefore they possess potential for applications as lightweight structural components in load-bearing systems, in transport and aerospace industries [2,3,4].

Many different manufacturing techniques have been developed for the production of open-cell and closed-cell microcellular metal [2,3,4]. Open-cell foams made by replication have special interest since these have reproducible macroscopically uniform structures, and can be produced with systematic variations several parameters, e.g., cell shape, cell size or relative density [5].

There are still numerous questions concerning the deformation mechanisms in these materials, particularly when their pores are small [2,3,4]. In this work, the compressive behaviour of salt replicated foams of different cell sizes was studied by the acoustic emission (AE) technique. AE stems from transient elastic waves generated within the material due to sudden localized structural changes, e.g. collective dislocation motion and damage processes, it thus yields interesting *in-situ* information on the dynamic processes involved in deformation and failure of the material. In particular, it is now well established that processes that underlie plastic deformation are far from being smooth and steady; rather, an intermittent and avalanche-like signature of dislocations moving through the material is observed [6]. Experiments on creeping ice and metallic single crystals [6,7] furthermore indicate that, during plastic deformation, the dislocations self-organize into a scale-free pattern that generates dislocation avalanches, characterized by power law distribution of

avalanche amplitudes  $A_0$ , i.e.  $P(A_0) \sim A_0^{-\tau}$ , independently of material and testing mode [6], characteristic of self-organized criticality (SOC). The validity of the SOC concept for dislocation dynamics in aluminum foams is the object of this paper.

### Experimental procedure

Compression test samples of three different open-cell microcellular aluminum materials were produced at EPFL using NaCl-based replication. In brief, the process comprises: (i) producing a preform of cold-pressed uniformly sized (400, 75 or 25  $\mu\text{m}$  diameter) NaCl particles, (ii) infiltrating this preform with pressurized 99.99% pure aluminum, (iii) solidifying the metal within the preform, (iv) machining the resulting preform/metal composite to produce test samples and (v) leaching the preform in water, using a corrosion inhibitor; more details can be found in Refs. [5, 8, 9]. The relative densities of samples were in each case approximately 25%. One 25  $\mu\text{m}$  sample (diameter 10 mm, length 20 mm), two 400  $\mu\text{m}$  and two 75  $\mu\text{m}$  samples (diameter 10 mm, length 15 mm) were prepared in this way for testing. Compression test data included stress-time curves and AE responses, recorded versus time during the deformation.

Compression tests were performed using a universal testing machine (Instron 5882) at room temperature with a cross-head speed of  $1 \times 10^{-3} \text{ s}^{-1}$ . The AE response was monitored with a computer controlled DAKEL-XEDO-3 AE system on the basis of two-threshold-level detection (recommended by ASTM standard E1067-85 [10]). A miniaturized MST8S piezoelectric transducer (diameter 3 mm, flat response in the frequency band from 100 to 600 kHz), was attached to the specimen surface using silicon grease and a spring. The total gain was 88 dB. The AE signal sampling rate was 4 MHz, and the threshold voltages for the total AE count and for the burst AE count were 824 and 1450 mV, respectively. The full scale of the A/D converter was  $\pm 2.4 \text{ V}$ . A comprehensive set of AE parameters involving count rate  $N_{C1}$  (count per second) at the lower threshold level (giving total AE count) were evaluated. Scanning electron microscopy (SEM) micrographs were obtained using a Tesla BS343 microscope (secondary electron imaging) operating at 15 kV.

### Results and discussion

Relative densities of Al foam samples with 25, 75 and 400  $\mu\text{m}$  cell size were 24 %, 24 % and 23.5 % respectively: these values are sufficiently close not to affect the compression behavior significantly. Thus, the major effect on the deformation curves can be ascribed to different cell sizes of the specimens, or in other words to the plasticity size effect, as documented elsewhere [4,11]. The respective stress-strain curves are shown in Fig. 1. Due to the presence of noisy signals, AE data for the tests of Samples 25 and 75a were aborted.

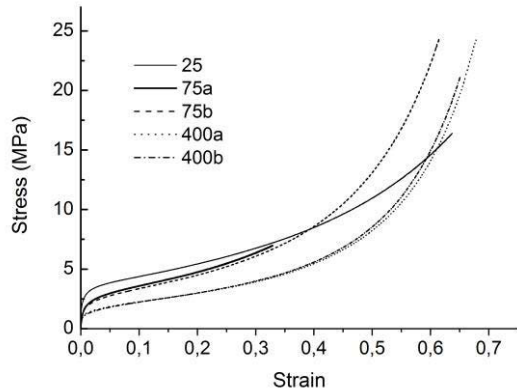


Fig. 1. Compressive stress-time curves for Al foam samples. Almost identical deformation behaviour of samples with the same pore size (75a and 75b; 400a and 400b) is observed

Curves in Fig. 1 are smooth, as is typical of replicated microcellular aluminum compression, and exhibit three different stages of strain hardening typical of microcellular metals in general, in which cell struts deform, buckle and eventually are pressed together (densification), causing the stress to increase rapidly [1-4,11-13]. Stress-strain curves of each pair of Samples 75a and 75b, 400a and 400b almost superimpose, showing a good reproducibility of the results. Further, finer pore samples have a higher flow stress, as seen by comparing the deformation curves from 400 to 75 and to 25  $\mu\text{m}$  pore sizes, Fig. 1, confirming the plasticity size effect previously reported for this material [4,5, 11,14].

Owing to the fabrication process, the grain size of the metal in the samples is very large (millimeters). Nearly all struts, for this reason, are single crystals of pure aluminum.

Since the foams are comprised of many struts connected by nodes, the deformation of a foam material under compression is driven by the collective deformation of single struts, which primarily deform by bending and also by some degree of torsion, tension and compression. The stress-strain dependence and the corresponding AE response recorded in the compression tests are, therefore, the collective signals coming from many variously stressed small single crystals of irregular shape.

As these crystals deform, they deform independently, interacting one with another, from strut to strut, via the nodes. The struts diameters are approximately of the order of one-tenth of the pore diameter. Thus, at these sizes, each strut is likely to deform plastically in intermittent mode [15]. This is suggested in a scanning electron microscopy micrograph (Fig. 2), where discrete slip steps along the surface of individual deformed struts are observed. The slip steps are not of a negligible magnitude, hence the struts are likely to “feel” the strain bursts. Therefore, plastic deformation is likely to be intermittent for each strut, while the recorded stress-strain curves are a collection of strain bursts from individual struts.

The AE response of Samples 25, 75b and 400a recorded during the tests simultaneously with the respective stress-time curves are presented in Fig. 3-5. It is observed that the AE activity varies with the cell size of the sample. The count rate, which is the rate of counts registered by the measuring instrument per second, reflects the number and activity of individual AE sources within the tested material. Thus, in our case, the samples with a higher

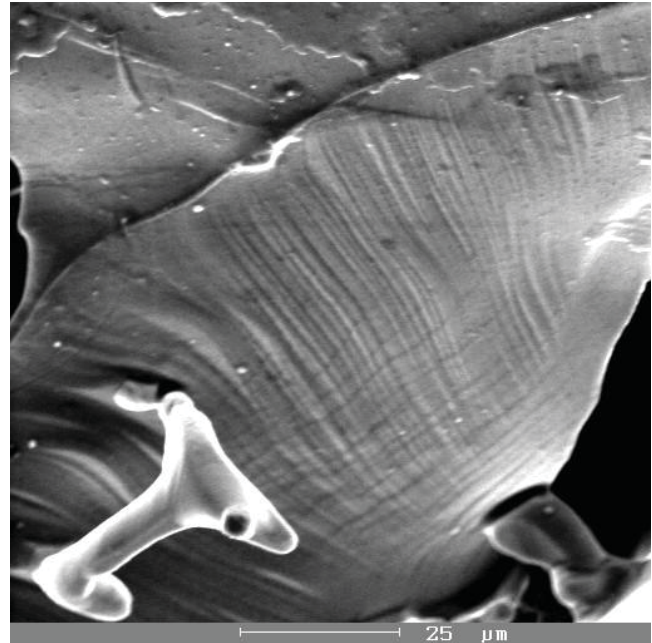


Fig. 2. Surface of the struts in the microcellular aluminum after deformation (sample 75b). The discrete slip steps along the surface of deformed struts are observed.

density of struts (or smaller pores) should render a higher number of AE signals than those with a lower strut density (larger pores). On the other hand, in large pore samples, the struts are larger and one may therefore expect the individual AE sources to be more intense. A non-monotonous dependence of the AE count rate on pore size can therefore be anticipated. This is confirmed, as clearly seen from Figs. 3-5, showing that the largest AE count rate is observed in the sample with the 75  $\mu\text{m}$  pore size (Fig. 4).

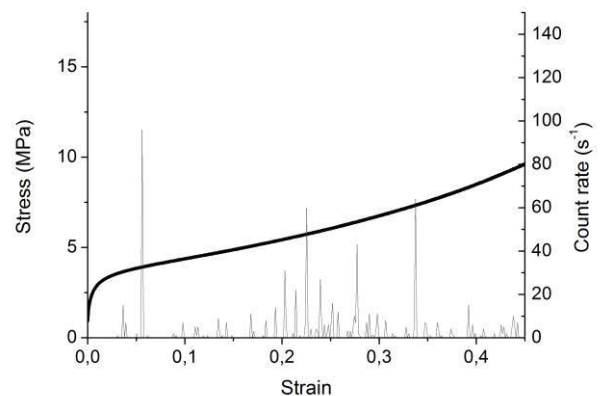


Fig. 3. The stress-time curve and the respective AE count rate observed during the compression test of the sample 25.

In order to evaluate the statistical properties of the recorded AE signal, the probability density distributions of the squared AE event amplitudes were examined (the squared AE amplitudes are proportional to the AE event energy). Corresponding log-log plots for the Samples 25, 75b and 400a are shown in Fig. 6-8, respectively. Clear signatures of universal scaling are present for the 75b and 400a samples, showing power-

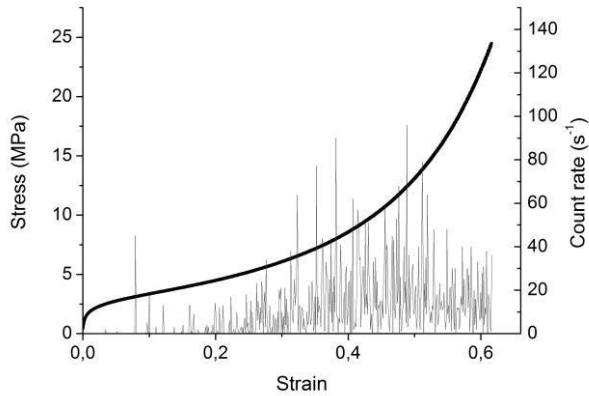


Fig. 4. The stress-time curve and the respective AE count rate observed during the compression test of the sample 75b. The largest AE count rate is observed in this sample.

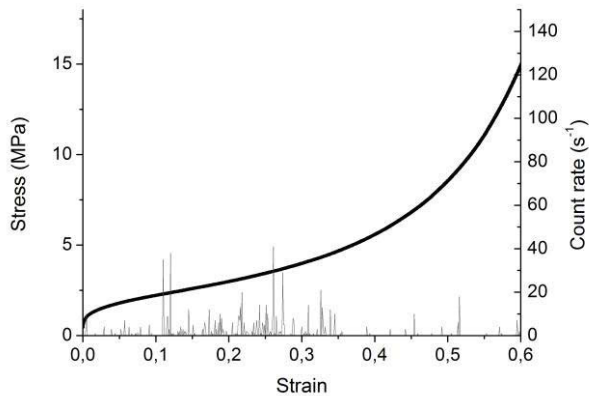


Fig. 5. The stress-time curve and the respective AE count rate observed during the compression test of Sample 400a.

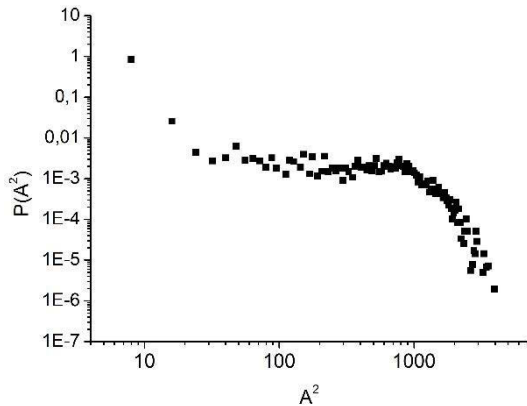


Fig. 6. Power-law distribution of the squared AE amplitude probability density for the sample 25. The power-law distribution was not observed.

law distributions with critical exponents  $\tau \sim 2.1$  and  $\tau \sim 1.5$ , respectively. These results are consistent with the earlier works [6,7,16-18]. Yet, the energy probability curve of the sample 25 apparently does not exhibit a power-law distribution. Therefore, the universal exponent  $\tau$  is found only for Al foams with pore size of 75 and 400  $\mu\text{m}$ .

This inconsistency found with Sample 25, as analyzed above, may indicate e.g. a change in deformation mechanism or a second deformation mechanism being active in small sized pore Al foams. Thus, a further research, primarily on the AE dependence on the cell-size is necessary.

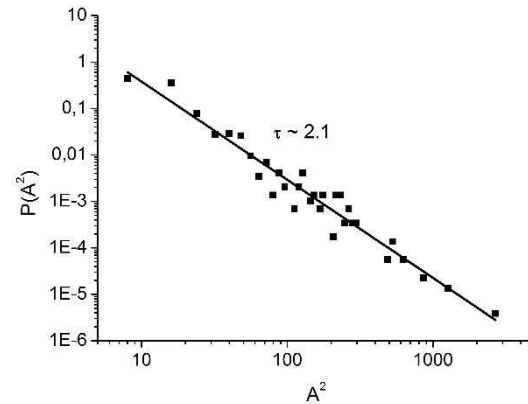


Fig. 7. Power-law distribution of the squared AE amplitude probability density for Sample 75b. Critical exponent  $\tau \sim 2.1$  is found.

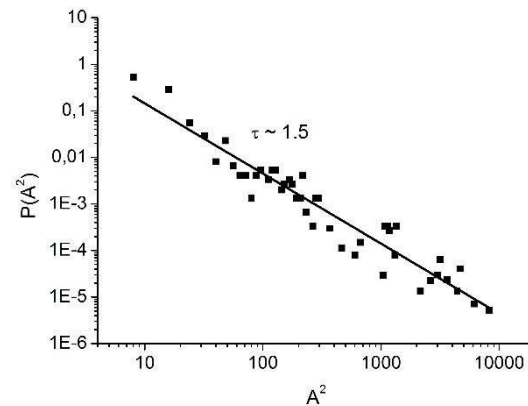


Fig. 8. Power-law distribution of the squared AE amplitude probability density for Sample 400a. Critical exponent  $\tau \sim 1.5$  is found.

## Conclusions

Compression tests were performed on 25, 75 and 400  $\mu\text{m}$  salt replicated microcellular aluminum. The smooth deformation curves typical for microcellular metals were found. Further, it was shown that the stress-strain curves separate according to sample pore size (finer pore samples have higher flow stress). The acoustic emission activity is also dependent on the cell size. The largest acoustic emission count rate was recorded during the compression of the finer pore sample 75b

(with a larger number of struts), by comparison to the sample 400a, while the behavior of the sample 25 was unexpected. Statistical properties of the acoustic emission data were also evaluated. Samples 75b and 400a exhibit power-law distributions of the acoustic emission energies with the exponent similar to those previously found in several works. It suggests the universal scaling character of plastic deformation (micro)processes in the materials. However, the inconsistencies found by the small pore-sized sample 25, which might be a result of a change in the deformation mechanism or the second deformation mechanism being active, require further study.

### Acknowledgements

This work received support from the Grant Agency of Charles University (Grant No. 946213). M.K., P.D., F.C. are grateful to the Czech Science Foundation for financial support under research grant No. P108-11/1267. M.K. and M.Z. are grateful for financial support from the grant SVV-2013- 267303. The EPFL contribution to this study was funded by the Swiss National Science Foundation, Project No. 200020-1182134.

### References

- [1] L.J. Gibson, M.F. Ashby, "Cellular Solids—Structure and Properties", second ed., Cambridge University Press, Cambridge, 1997.
- [2] M.F. Ashby, A. Evans, N.A. Fleck, L.J. Gibson, J.W. Hutchinson, H.N.G. Wadley, "Metal Foams: A Design Guide", first ed., Butterworth-Heinemann, Boston, 2000.
- [3] J. Banhart, Manufacture, characterisation and application of cellular metals and metal foams, *Progress in Materials Science*, 2001, 559-632.
- [4] R. Goodall, A. Mortensen (2013), Chapter 3.05 "Porous Metals", in Hono K, Laughlin D (eds), *Physical Metallurgy: 5th Edition*, Elsevier, Amsterdam, NL (in print).
- [5] R. Goodall, J.-F. Despois, A. Marmottant, L. Salvo, A. Mortensen, "The effect of preform processing on replicated aluminium foam structure and mechanical properties", *Scripta Mater.* 54, 2006, 2069-2073.
- [6] T. Richeton, P. Dobron, F. Chmelik, J. Weiss, F. Louchet, "On the critical character of plasticity in metallic single crystals", *Mater. Sci. Eng: A* 424 1–2, 2006, 190-195.
- [7] T. Richeton, J. Weiss, F. Louchet, "Dislocation avalanches: Role of temperature, grain size and strain hardening", *Acta Materialia*, Vol. 53, 2005, 4463-447.
- [8] C. San Marchi and A. Mortensen, "Deformation of Open-cell Aluminum Foam", *Acta Materialia*, 49 (19), 2001, 3959-3969.
- [9] Y. Conde, J.-F. Despois, R. Goodall, A. Marmottant, L. Salvo, C.S. Marchi, A. Mortensen, "Replication (a.k.a. Space-holder) Processing of Highly Porous Materials", *Advanced Engineering Materials*, vol. 8(9), 2006, pp. 795-803
- [10] Standard Practice for Acoustic Emission Examination of Fiberglass Reinforced Plastic Resin, ASTM E 1067-85, Tank/Vessels, 1985.
- [11] R. Goodall, J.F. Despois, and A. Mortensen, "The Plasticity Size Effect in Replicated Microcellular Aluminium", *Scripta Materialia*, 69 (6), 2013, pp. 469-472;
- [12] C. Kádár, F. Chmelík, M. Cieslar, J. Lendvai, "Acoustic emission of salt-replicated foams during compression", *Scripta Materialia* 59, Issue 9, 2008, 987-990.
- [13] E. Koza, M. Leonowicz, S. Wojciechowski, F. Simancik, "Compressive strength of aluminium foams", *Materials Letters* 58, Issues 1–2, 2004, 132-135.
- [14] F. Diologent, R. Goodall, A. Mortensen, "Surface oxide in replicated microcellular aluminium and its influence on the plasticity size effect", *Acta Materialia*, 57(1), 286-294.
- [15] D. M. Dimiduk, C. Woodward, R. LeSar, M. D. Uchic, "Scale-Free Intermittent Flow in Crystal Plasticity". *Science* 312, 2006, 1188-1190.
- [16] J. Weiss, T. Richeton, F. Louchet, F. Chmelík, P. Dobroň, D. Entemeyer, M. Lebyodkin, T. Lebedkina, C. Fressengeas, R.J. McDonald, "Evidence for universal intermittent crystal plasticity from acoustic emission and high-resolution extensometry experiments", *Physical Review B*, 76, 2007.
- [17] T. Richeton, J. Weiss, F. Louchet, "Breakdown of avalanche critical behaviour in polycrystalline plasticity", *Nat. Mater.* 4, 2005, 465-469.
- [18] J. Weiss, J.R. Grasso, M.C. Miguel, A. Vespignani, S. Zapperi, "Complexity in dislocation dynamics: experiments", *Materials Science and Engineering: A*, 309-310, 2001, 360-364.