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Mechanical quality control of β-alumina membranes using acoustic emission

Beta-alumina membranes are most usually used in the sodium–sulphur battery system as a solid electrolyte. The performance of this battery system strongly depends on the properties and quality of the solid electrolyte. Along with a high ionic conductivity, β-alumina membranes must have a high mechanical strength which is a prerequisite for a satisfactory cycle life.

Usually, the fracture strength of β-alumina membranes is determined from the mechanical load under which they break down [1–3]. The development of a non-destructive method of determination of the fracture strength would make it possible to perform screening tests prior to the incorporation of the solid electrolyte membrane in the cell.

The present paper presents a possible solution of the problem.

It is known [4–6] that the formation of microcracks and the widening of already existing ones leads to the release of energy as elastic waves. This process is known as acoustic emission (AE). The study of AE under mechanical loading of various materials may yield useful information on the existence of macroscopic defects in the structure and on the mechanical characteristics of the sample.

In our experiments we determined the fracture strength of β-alumina membranes with simultaneous recording of the AE.

The membranes were shaped like closed-end tubes with the following dimensions: outer diameter 9 mm, wall thickness 1.2 mm and length 70 mm. The membranes were prepared from the same starting materials under identical conditions. The density of the sintered samples was 3.20 g cm^{-3}. Membrane B was additionally treated at 1400° C for 10 h. This procedure is known as post-sintering, aimed at improving the mechanical and electrical characteristics of the membranes. The samples were loaded using a four-point bending method at a constant rate (0.5 mm min^{-1}).

A Brüel and Kjaer (B & K) resonance transducer 8314 with main resonant frequency about 750 kHz was attached to the open end of the membrane by means of adhesive tape and silicon grease as a coupling medium. The signal obtained from the transducer was amplified by a 2638 B & K wide-band conditioning amplifier and was recordered with a Y–t recorder. At the same time the cumulative sum of AE counts was recorded after processing of the amplified signal with a 4429 B & K AE pulse analyser. The gain was selected so that in the absence of mechanical loading no AE signal was detected.

Fig. 1a and b illustrates the AE results during four-point mechanical loading of membranes A and B. It can be seen that the post-sintered sample, B, which has a higher fracture strength (1708 kg cm^{-2}) displays no AE. This may be connected with the absence of macroscopic defects in the structure as a consequence of the post-sintering

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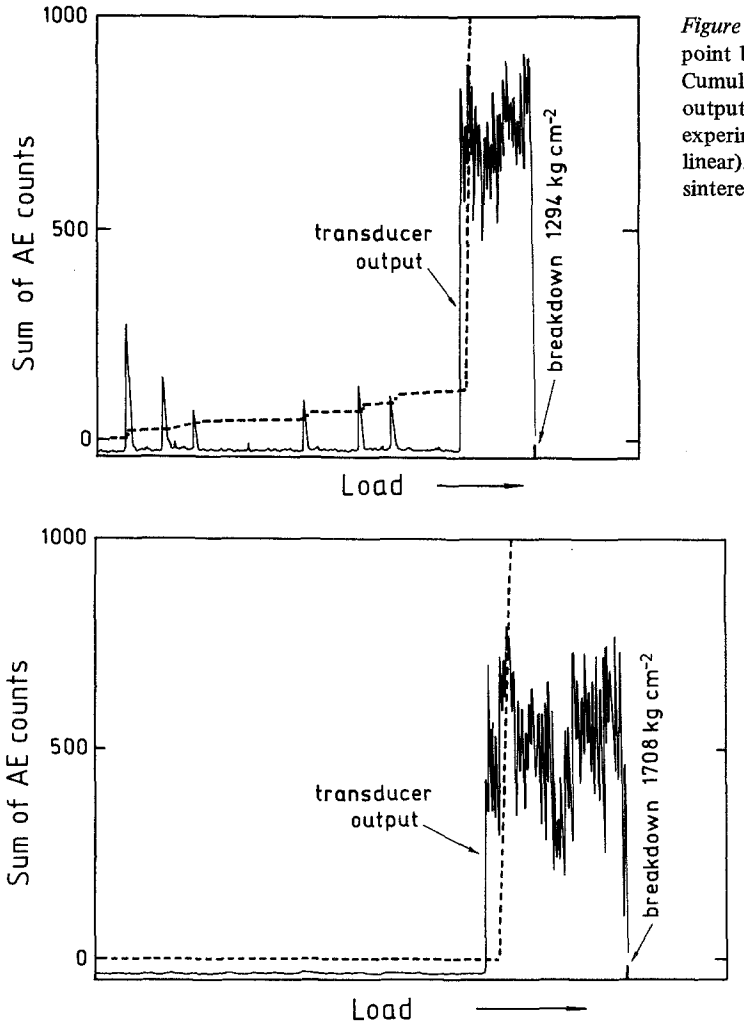

Figure 1 Acoustic emission during the four-point bending of β-alumina membrances. – – – Cumulative sum of AE counts, —— transducer output. Amplification, 76 dB (N.B. under the experimental conditions the load scale is not linear). (a) Sample A; and (b) Sample B post-sintered at 1400° C for 10 h.

procedure. Under the amplification conditions used detectable AE signals are obtained only at higher loads close to the break-down point. The latter can be connected with the appearance of microcracks which originate in the bulk of the membrane and propagate under the effect of the increasing load thus leading to the destruction of the membrane. The existence of AE at relatively low loadings (Fig. 1a) is obviously an indication of the existence of macroscopic defects leading to a decrease in the fracture strength of sample A (1294 kg cm^{-2}).

Fig. 1a and b shows that the cumulative sum of AE counts as a function of load yields equivalent information concerning the AE activity of the material under test. It has a step-like shape for samples with macroscopic defects.

These preliminary results lead us to believe that it is possible to develop a non-destructive method for the determination of the mechanical quality of β-alumina membranes by analysing their AE under mechanical loading.

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A simple measuring method for the characteristic curve of $S(\theta)\cos\theta/S(0)$

In the last two decades there has been a growing interest in the study of ion-etching of solid surfaces. Many experiments have been carried out to investigate the development of surface topographies [1–6]. In many cases, since we are interested in the thickness of the material removed by the ion-etching process and in particular when we want to predict the evolution of a surface contour by ion-erosion, it is worth plotting $S(\theta)\cos\theta/S(0)$. Barber *et al.* [7] have produced such a graph, taking data from Bach [8], who used the interference–microscopic method to determine the sputtering yield.

It appears that it is convenient to utilize the expression

$$d(\theta) = \frac{\Phi t}{n} S(\theta)\cos\theta \qquad (1)$$

which expresses the depth d sputtered from a plane surface [9, 10]. Φ is the number of ions per second striking the unit area of surface normal to their direction, t is the time of bombardment, n is the number of atoms per unit volume of target material, $S(\theta)$ is the sputtering yield (sputtering ratio) and θ is the angle between the incident beam and the surface normal. For given values of t and Φ, and n being constant we can write

$$d(\theta) \sim S(\theta)\cos\theta \qquad (2)$$

or

$$d(\theta)/d(0) \sim S(\theta)\cos\theta/S(0). \qquad (3)$$

Hence, if we want to know the plot $S(\theta)\cos\theta/S(0)$ it becomes necessary to define $d(\theta)/d(0)$. A simple glow discharge ion gun for etching [11, 12] and a glass surface as a target were used for this purpose.

This kind of ion source can be used with electrically insulating materials due to the presence of energetic neutrons and electrons in the ion beam. Glass was chosen as a subject for study since it is single-phase and non-crystalline, and because the assumption can be made that the amount of surface etched is entirely dependent on the inclination of the ion beam to the surface. After 1 h of ion-etching under the conditions described earlier [13] a funnel-shaped pit could be observed on the glass surface. A profile of the funnel was recorded on a profilograph. Etchings of identical duration were made at various angles of beam incidence, θ, and for each angle the maximum depth, d, of the funnel was measured. In this way we obtained [13] a plot for $d(\theta)$, which after normalization, $d(\theta)/d(0)$, is proportional to $S(\theta)\cos\theta/S(0)$ according to Equation 3. The method described above is only an approximation but is very simple and useful, especially in the case of insulating materials.

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