Impact and indenting damage of CVD-produced ZnS and ZnSe ceramics

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Abstract

Time series of acoustic emission pulses were excited in ductile ZnS and ZnSe ceramics either by a falling weight or by indenting the Vickers pyramid. Energy distributions in emitted acoustical emission pulses were found to be random (Poisson-like) in the events of short (0.3–0.5 ms) impact forcing. In the case of the gradual (~1 s) indenting, the energy distributions followed a power law typical for the self-similar structures appearing through long-range interactions between nucleating microcracks (scaling). In ductile materials, the rate of straining governs the dislocation motion. Provided enough loading time, such as in indenting experiments, the sliding dislocations form bunches, which serve as weak points for the crack nucleation. Given a tend to self-organizing in the ensemble of dislocations, the energy release in impeded cracking process (i.e. indenting) exhibits statistically ordered behavior.

1. Introduction

Semiconductor materials ZnS and ZnSe are used for production of focusing elements and windows in optics as well as pressure sensing elements in various mechanical transducers. Owing to their transparency in the atmospheric window (8–14 µm), the ZnS and ZnSe ceramics are designed to protect both IR sensors established on mobile carriers [1, 2] and solar cells made of A₂B₆ [3–7], which are exposed to knocks of dust particles and atmospheric precipitation [8, 9]. Because of their well-pronounced fractoluminescent properties, these compounds have application in impact [10] and pressure [11] sensors.

In this work, the damage initiation in ZnS and ZnSe ceramics under either pointed impact loading [12] or indenting a Vickers pyramid [13, 14] has been investigated with the acoustic emission (AE) method. The microcracking process in A₂B₆ semiconductors is accompanied with the light emission (fractoluminescence) caused by the strain-stimulated motion of charged dislocations [15, 16]. When reaching the ultimate strain, a fault with growing cracks emerges, which manifest themselves by sound generation [17]. The penetration speed of an indenter into ceramics was about three order of magnitude lower than that of a striker used in our impact experiments.

2. Experimental

ZnS and ZnSe samples were prepared using the CVD (chemical vapor deposition) technology that is widely used to produce high-quality optical ceramics. The studied polycrystalline ceramics differed substantially in their microhardness—1800 MPa and 1350 MPa for ZnS and ZnSe, respectively. Samples were shaped to discs of 20–30 mm in diameter and 1–2 mm in thickness. The surface damage was produced either by a weight falling onto a pointed harden striker established on a sample or through indentation of the Vickers pyramid. The pyramid was indented with a significant load (100 to 200 N) to excite sufficiently strong AE signals required for reliable detection. Full time of the indentation procedure was 1 s.
The AE signals from growing cracks were detected by a broadband piezotransducer. As far as both ZnSe and ZnS ceramics exhibit intrinsic piezoelectric effect, the piezotransducer made of highly sensitive Pb(ZrxTi1-x)O3 (PZT) ceramics was applied in place of commonly used quartz crystals. The piezoelectric constant of the PZT is by two orders of magnitude higher than that of studied ceramics (320 pC/N against 3.2 pC/N and 1.1 pC/N in ZnSe and ZnS, respectively) So, the intrinsic piezoeffect in samples could not affect noticeably the intensity of measured AE signals.

The digital low frequency sound filtration was applied at the level of 100 kHz to cut off parasitic oscillations of the sample and setup. The voltage-to-digit converted AE signals were stored in a PC; the dynamic range of the converter was 2 mV to 10 V (70 dB).

3. Results

3.1. Impact

Figure 1 shows the time series of AE signals excited in both ceramics subjected to the impact forcing. The period of the shock-induced AE activity was of 0.3–0.5 ms. The amplitude squared ($A^2$) of each AE pulse was proportional to the energy, $E$, released in a pulse and, vice versa, $E \propto A^2$.

Figure 2 shows the distributions of the AE pulses over their energy constructed in the form of the formula $N(E > \varepsilon)$ versus $\varepsilon$ dependence, where $N$ is the number of pulses that the energy $E$ was higher than a varying ‘threshold’ $\varepsilon$. In other words, the parameter $\varepsilon$ goes through all energy release values in pulses detected in the interval of 0 to 0.5 ms (horizontal coordinate), and the number of pulses, the energy of which $E$ exceeded a current $\varepsilon$ value, is shown on the vertical axis.

The same data were plotted in two coordinates. The linear coordinates were utilized in a left panel (figure 2(a)), while a right panel (figure 2(b)) represents the same data in semi-logarithmic coordinates (with a linear scale along the horizontal axis). One can see that the distributions plotted in linear coordinates represent smooth descending curves. In semi-logarithmic coordinates, the data points fall upon straight lines with the different slopes $a$: 
The relation (1a) is equivalent to the exponential law of the Poissonian type:

\[ N(E > \varepsilon) \propto e^{-a\varepsilon}, \]  

which is indicative of random events occurring independently from each other.

### 3.2. Indention

The time series of AE signals excited by indenting the pyramid are depicted in figure 3. The period of the high emission activity (4–5 ms) followed by a very weak sporadic pulses during 60–80 ms after applying an indenter.

Just as in the preceding section 3.1, the distributions were plotted in coordinates of two different kinds. However, this time, the semi-logarithmic coordinates (figures 4(a), (b)) were supplemented with double-log ones (figures 4(c), (d)). One can see that the \(N(E > \varepsilon)\) versus \(\varepsilon\) distributions do not represent straight portions in semi-log coordinates (left panels in figure 4). Consequently, the process of damage accumulation through cracking did not follow a random (Poissonian) law. However, the same distributions plotted in double-log coordinates exhibited the log-linear dependences:

\[ \log_{10} N(E > \varepsilon) \propto -b \log_{10}(\varepsilon), \]

where \(b\) is the slope of straight portions. This parameter characterizes a relative contribution of varying energy release in detected cracks. The lower \(b\)-value, the higher contribution of ‘larger’ pulses into the whole \(N(E > \varepsilon)\)
versus $\varepsilon$ distribution. Compared together figures 4(c) and (d), the $b$-values in ZnS-distributions at both loads are notably lower than that in ZnSe-distributions. This means that the energy release in harder ZnS ceramics proceeds mostly in larger cracks, while the cracking in the loaded ZnSe samples occurred predominantly through smaller damages. Moreover, in both materials, the $b$-value was higher in smaller loaded samples than in their lesser loaded counterparts (200 N against 100 N) that is the slope of log-linear dependences (2) was sensitive to the loading conditions.

Portions in semi-log coordinates (left panels in figure 4). Consequently, the process of damage accumulation through cracking did not follow a random (Poissonian) law. However, the same distributions plotted in double-log coordinates exhibited the log-linear dependences:

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In a nonlogarithmic form, the $N(E > \varepsilon)$ versus $\varepsilon$ distribution transforms into the power law:

$$N(E > \varepsilon) \propto \varepsilon^{-b}.$$  

Contrary to the quick-decaying exponential function, the power law provides long-range interactions between elements/events. Consequently, the external energy income dissipates in the dynamically interconnected system where the random damage accumulation is impossible.

4. Discussion

In loading experiments on the ZnS and ZnSe ductile ceramics, two quite different energy patterns of the cracking process were observed. The energy release in sequences of AE pulses excited by crack nucleation under falling weight was random that is followed a Poisson-type energy distribution (equation (1b)) unlike the crashing of hard materials such as granite [18], silicon carbide [19], silicon dioxide [20], just to name a few, which exhibit pronounced features of the cooperative damage process due to long-range elastic interactions. The observed
behavior of the tested ductile solids means that the primary straining caused by a short-time shock decays exponentially. The lack of effective interactions between overstressed hypocenters results in a random damage accumulation.

In the indenting experiments, the time series of AE activity extended up to 1 s (against ~0.2 ms activity after the impact loading). The energy distribution in detected pulses followed a power law in both ceramics thus exhibiting the scale-invariant damage accumulation. A feature of the scale-invariance is a manifestation of the lack of any characteristic energy release in cracks because the function \( N(E) \) in equation 2a is the single solution of a scaling equation:

\[
N(\lambda E) = \lambda^{-\delta} N(E)
\]

where \( \lambda \) is the scaling index. In the case of cracking, the number of AE pulses of various energy changes accordingly the scale of detection \( \lambda E \). In words, the scaling means that any (statistically significant) fragment of the time series contains approximately the same proportion of ‘small’ and ‘large’ events.

A power law ascertains that any mechanical effect of the microcrack nucleation manifests itself at distances, which exceed significantly the size of a newly formed defect. In hard materials, the long-range interactions of this kind realize through elastic waves propagating from newly formed defects.

The damage accumulation in ductile crystalline solids proceeds mainly through the dislocation motion, which depends significantly on the time parameters of loading.

The short-time impact does not stimulate the virtual interactions between nucleated cracks through sliding of isolated dislocation. However, under condition of the sustained external forcing (indenting), the cooperative motion of dislocations results in their self-organizing in clusters [21]. The dislocation clusters and pileups serve as ‘weak points’ for crack nucleation. Long-range interactions in the dislocation ensemble govern the mode of cracking process in the ductile solids.

5. Conclusion

Mechanical response of the CVD-produced ZnS and ZnSe ceramics on micro-damaging performed either by impact or by impeded forcing (through a Vickers pyramid) was studied by the acoustic emission method. The shock-induced fracturing of tested ductile ceramics exhibited the random energy distributions in AE pulses due to the slack in time for establishing the efficient elastic interactions between stressed hypocenters. The energy distribution in the AE time series of gradually indented ZnS and ZnSe ceramics followed a power law specific for cooperative damage formation. The straining of these ductile crystals occurs through the dislocation motion with forming the dislocation clusters whose layout represents a self-similar pattern of ‘weak points’, which stimulate the scale-invariant nucleating of microcracks.

So, the temporal characteristics of the applied loading are crucial for the prevailing—random or self-organized—mode of damage accumulation in ductile ceramics.

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