

Size distribution of sapphire fragments in shock fragmentation

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The sapphire fragments size distribution in one-shock fragmentation is obtained in a mass range from 0.002 to 100 mg. Two methods of fragment size determination are used — the method of weighing and the method of photographing with subsequent measurement of the image area. The comparative analysis of methods is carried out, and a good agreement of the results is shown. The form of large-scale asymptotic of size distribution of fragments is determined. It has power distribution form with exponent −2.5. The crack-resistance coefficient is calculated on the basis of obtained results.

Экспериментально получено распределение по размерам осколков сапфира в одноударной фрагментации в диапазоне масс осколков от 0,002 до 100 мг. Использованы два метода определения размеров осколков — метод взвешивания и метод фотографирования с последующим измерением площади изображения. Проведен сравнительный анализ методов, показано хорошее согласие результатов. Определён вид крупномасштабной асимптотики распределения размеров фрагментов. Она имеет вид степенного распределения с показателем степени −2,5. На основании полученных результатов вычислен коэффициент трещиностойкости.

1. Introduction

Shock fragmentation, or, as it is called in [1], "weak fragmentation", represents the destroying of a material sample with one impact with given energy. The shock fragmentation is the central stage of many technological processes and testing methods. With its results we can determine the material strength characteristics — related both to shock destroying as such and "intrinsic" material characteristics, such as effective binding energy.

The shock fragmentation can be used for quick obtaining a lot of material samples with a specified size distribution, in particular — of micro-fine powders. In contrast to multi-shock fragmentation or prolonged fragmentation under pressure, the properties of fragment size distribution in case of one-shock fragmentation can be well controlled by parameters of the fragmented sample (for example, choice of its size) and the destroying device (in particular — the energy injected in the impact). For this reason, the study of fragments distribution properties in shock fragmentation seems to be very important.

Sapphire is a transparent crystal, it is very rigid. It is possible to grow such crystals of comparatively large size. Nowadays some producers offer windows 500×2500 mm with thickness 30 mm. The specific features of this material determine the sapphire application in transparent high-speed shock protection constructions.

There are some special methods for evaluation of strength characteristics in high-speed shock [2, 3]. However, they are oriented on studies of separate characteristics of amorphous materials and are distinguished by stresses created in the sample in destroying. Due to anisotropy of its crystal structure, the sapphire has different properties in different crystallographic directions. Also strength characteristics essentially vary depending on the crystal surface state (grinding, polishing, chemical or thermal etching) [4].

The crystal strength characteristics can be determined from the fragments size distribution that is formed in the process of destroying it by impact. Our work is dedicated to obtaining this distribution, its analysis and also crystal strength characteristics calculation on the base of it. In the capacity of the "fragment size" value we can use its mass, volume or specific geometric size — the cube root of volume. In most of our work we used the fragment mass in this capacity.

We have focused on obtaining the fragment size distribution, because knowledge of this distribution both has an independent significance and, simultaneously, allows one to calculate other physical characteristics, such as crack-resistance coefficient, average fragment size and fragment size dispersion (which is important for obtaining fine-dispersed powders). In addition, knowledge of dispersion on a sufficiently wide scale range of fragment mass allows extrapolating this distribution on scales inaccessible in frames of other measuring methods. This allows making predictions about fragment sizes, direct measuring of which is technically difficult.

Finally, it is useful to compare distributions obtained as a result of our experiments with distributions found in other works dedicated to shock fragmentation. It will allow making reasonable assumptions about the relative effects on the distribution of specific parameters of the experiment (material, size and shape of sample, and also the method of impact bringing and injected energy quantity), on the one hand, and general universal shock fragmentation properties on the other hand.

We have set the experiment on shock fragmentation of sapphire samples. Fragment masses formed in this experiment were measured, and with these data fragment distributions histograms over masses for different measuring methods and on different scales were constructed. On the base of constructed histograms we determined the form of the large-scale distribution asymptotic. The obtained asymptotic was theoretically grounded in work [1]. The comparison of it with the form of asymptotics discovered in other works ([5–8]) allows making an assumption about the form universality of the fragments distribution on large scales. Also, we calculated the crack-resistance coefficient.

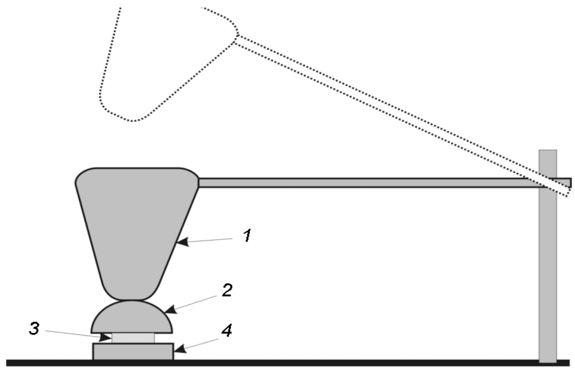

Fig. 1. The scheme of sample breaking device. 1 — projectile, 2 — upper layer of hardened steel, 3 — crystal, 4 — bottom layer of hardened steel.

2. Experiment set-up

Sapphire crystals in the form of disks with diameter 4.4 mm and width 1.2 mm were used for the experiments. The basis plane (0001) was parallel to the disk plane. The crystals were grown by the Stepanov's method and annealed in vacuum. The samples had identical quantity and quality of defects, because they have been cut from one crystal.

For sample destroying we used the impact device (Fig. 1). Load mass — 3.3 kg, falling height was changed within the limits of 12 cm to 35 cm.

We selected the projectile falling height equal to 18 cm, because with this load the fragments of every crystal had sizes in a wide mass range. With smaller load (projectile falling height 12 cm) crystals were destroyed into 2–5 fragments, and with larger load (projectile falling height 30 cm) the crystals were destroyed into a large quantity of small fragments which could not be weighted using an analytical weighing machine and separated on an object-plate for photographing.

Among all size characteristics, the fragment mass is determined with the least ambiguity. For example, the "line size" of a fragment can be defined both as its average diameter and as the cube root of its volume and, in some case, as square root of the area of its projection. This choice is, in fact, arbitrary.

Therefore, we accept the fragment mass as the size characteristic. We determine it by direct weighing in the mass range in which it is possible and calculate from the other measurements outside of this range as described below. We also apply additional measurement methods to those fragments which we can weigh. It is necessary, first of all, for the estimation of applicability of alternative measurement methods, and, secondly, for the determination of constants required for computation of fragment mass on the basis of the results obtained with these methods.

3. Data processing methods. Comparison of the results

Fragment masses were determined using two methods:

1. In first method, fragments was scattered on glass and photographed. Then the area of their images on the photo was determined and fragment masses were calculated on the basis of this area data.
2. The second method was weighing of fragments.

Weighing was carried out with the help of an electronic weighing machine, with precision up to 0.5 mg. From fragments of 40 experiments, all fragments with masses from 3 mg were weighed. There were 255 fragments.

Method with photographing. The image areas (in pixels) were calculated using an appropriate software, but before this the photo needed manual correction — deleting of traces of dust and artifacts, as well as covering over the inner regions of large fragments. The resolution of the photos was determined by marks plotted on the glass: 48 pixels to millimeter. Thus, the areas of fragment projections on glass in mm^2 were obtained. The fragment masses were calculated on the basis of these values using the following formula

$$m = \alpha\rho S^{3/2}, \qquad (1)$$

where ρ is the sapphire density (4 g/cm^3) and α is a "form-factor", i.e., a coefficient related to the fragment shape.

The form factor of fragments obtained in the experiment is determined on the basis of agreement between the data obtained by both methods. Of course, such agreement is realized in the region accessible for both methods i.e., the large-scale region. It is important, because the number of fragments measured using the photographing method is essentially larger then of the weighed fragments. This is due to the fact that the method with photographing allows recording of fragments of much lower mass as compared with the method using a weighing machine. Let us estimate the lower mass limit that can be measured in the photographing method. On a photo, one can record fragments with area of several pixels. We take, for definiteness, 10 pixels. Such an area on a photo corresponds to the area of fragment projection on glass close to $4.36\cdot10^{-3}$ mm^2 and, supposing that the fragment shape is close to cube, we obtain that its volume is about $2.875\cdot10^{-4}$ mm^3 and mass $-1.15\cdot10^{-3}$ mg. This mass is by three orders of magnitude smaller than the mass accessible for reliable weighing with acceptable precision using a weighing machine.

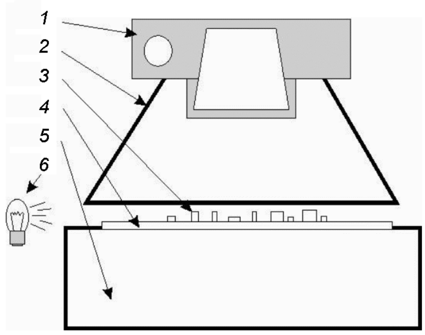

Fig. 2. The scheme of sample fragments photographing. 1 — camera, 2 — screen protecting camera objective from exposure, 3 — sample fragments after breaking, 4 — quartz object-plate, 5 — black box guaranteeing dark background for sample fragments, 6 — highlight lamp for sample fragments.

Let us describe the procedure of the form factor determination. The form factor value α is somewhat different on different scales. We suppose, however, that with the exception of several largest fragments (that represent almost unbroken initial samples), we can take an identical value for all scales. The criterion for determination of α, thus, must be insensitive to size deviations in a small quantity of the largest fragments. We take the equality of average fragment mass measured by the both methods as this criterion.

The average mass value that was obtained in weighing is determined in the usual way. For obtaining a similar value in the method with photographing, we need, first of all, to determine the area range of projections corresponding to the weighed fragments. It can be seen from (1) that it is a set of areas not less than $S_0 = m_0/\alpha\rho^{3/2}$, where m_0 is the smallest of masses obtained during weighing. Further, we find the aver-

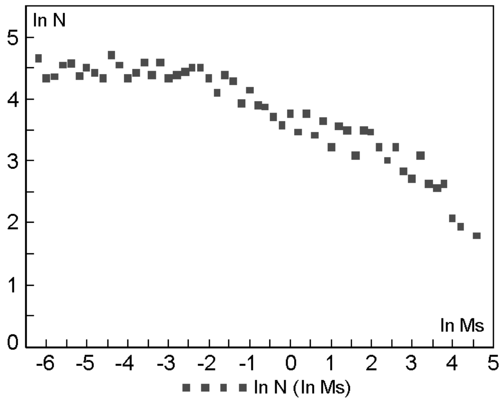

Fig. 3. The histogram of mass distribution of fragments obtained by the method with photographing.

age by calculating fragment masses using the above formula as

$$\langle m \rangle = \alpha\rho\langle S^{3/2} \rangle,$$

where the averaging is over the mentioned areas range.

As it can be seen, α enter both in the definition of range and in the averaging formula. We shall use the following procedure for determination of α. We shall choose as S_0 all the areas sequentially in the decreasing order. Further, we shall calculate, in this supposition, α ($\alpha = m_0/\rho(S_0)^{3/2}$) and $\langle m \rangle$. We shall compare this average with the average result of weighing. And we shall take as correct that value of α which ensures the equality of the average values. The form factor value calculated in this way was $\alpha \approx 0.63$.

Let us determine, for fragment shapes close to simple geometric figures (parallelepiped, ellipsoid), the values of parameters. We consider some examples to show how the form factor is related to parameters of geometric figures.

For a plate-like parallelepiped ($a \times a \times b$, $b < a$) the volume is equal to $V = a^2b$, and the projection area is $S = a^2$, so the form factor is equal to $\alpha = \frac{a^2b}{(a^2)^{3/2}} = \frac{b}{a}$.

For a rod-like parallelepiped ($b \times b \times a$, $b < a$), $V = ab^2$, $S = ab$ and $\alpha = \frac{ab^2}{(ab)^{3/2}} = \sqrt{\frac{b}{a}}$.

For a plate-like ellipsoid (semiaxes ($a \times a \times b$, $b < a$) $V = 4\pi a^2b/3$, $S = \pi a^2$), i.e.

$$\alpha = \frac{\frac{4}{3}\pi a^2 b}{(\pi a^2)^{3/2}} = \frac{4}{3\sqrt{\pi}}\frac{b}{a}.$$

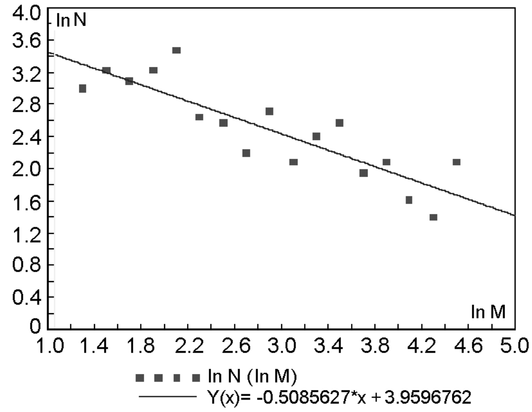

Fig. 4. Mass distribution of fragments in the large-scale region. Weighing method, double logarithm scale.

Finally, for a rod-like ellipsoid (semiaxes $b \times b \times a$, $b < a$) $V = 4\pi ab^2/3$, $S = \pi ab$, from here $\alpha = \frac{\frac{4}{3}\pi ab^2}{(\pi ab)^{3/2}} = \frac{4}{3\sqrt{\pi}}\sqrt{\frac{b}{a}}$.

Thus, α close to 0.63 can correspond to, for example, plate-like parallelepiped with its width about 0.63 of the other two dimensions. For plate-like ellipsoids such form factor value gives the axis length ratio of 0.84, i.e., it is but slightly different from sphere.

The histogram of fragments quantity as function of their "masses" obtained by recalculation of the image areas on the photos in the described way is given in Fig. 3.

4. Large-scale asymptotics of fragment distribution

We can obtain the mass distribution of fragments in the large-scale range from the data of direct weighing without additional recalculation.

Exponential and power large-scale asymptotic are mentioned most frequently in the works on fragmentation. The presence of exponential asymptotic in the distribution density $n(m)$ can be easily determined from the histogram of the fragment number logarithm $\ln N$ vs. mass — the points in the range of large masses are well approximated by a straight line. Similarly, we can easily see the power asymptotic on the histogram $\ln N$ vs. $\ln m$.

The histogram $\ln N(\ln m)$ is presented in Fig. 4. The arrangement of histogram points close to linearity can visually be seen, and this is confirmed by the determination coefficient (≈ 0.75) close to unity.

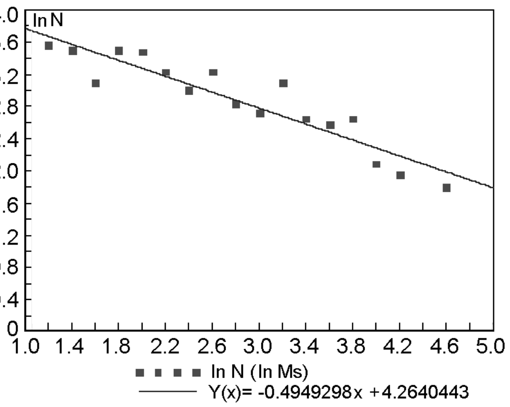

Fig. 5. Mass distribution of fragmentss in the large-scale region. Method with photographing, double logarithm scale.

The angle coefficient (slope) of the approximating line is close to −0.5. So, according to

$$n_m(m)dm = n_{\ln m}(lnm)d\ln m,$$

we obtain for the mass distribution density

$$n_m \propto m^{-1.5}.$$

The fragment distribution histogram obtained with the help of the method with photographing in a similar mass range is presented in Fig. 5. As it can be seen, the histogram is also well approximated by a straight line, practically with the same angle coefficient.

We obtain for the size distribution of fragments $r = V^{1/3}$, so,

$$n_r(r) \propto r^{-2.5}.$$

The histogram $N(r)$ in the large-scale range is presented in Fig. 6 with the approximating curve. Similar exponents of large-scale power asymptotic are mentioned in experimental works on fragmentation [5–8].

Thus, the comparison of large-scale distribution asymptotic obtained with the two methods involved shows good consistency of these methods. It confirms the high applicability of the method with photographing, which is very important because the method with photographing allows measurements of much smaller fragments.

We compared our asymptotics with those described in other works. It turned out that power asymptotic with exponent −2.5 for distribution $N(r)$ obtained in our study can also be found in other experiments on shock fragmentation, with other materials and load methods. Moreover, the observed exponents were close to those predicted theoretically [1]. It allows us to suppose the presence of universal behavior of fragments size distribution in the large-scale range for shock fragmentation.

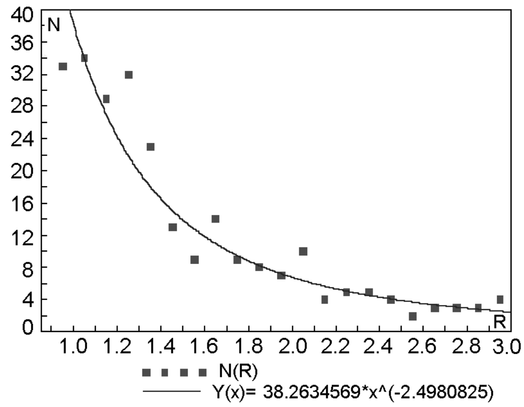

Fig. 6. Size distribution of fragments. Weighing method, ordinary scale.

5. Crack-resistance coefficient

Crack-resistance coefficient (also known as "frailness coefficient") is determined in the following way:

$$C = \frac{F - A}{A},$$

where A is the area of initial sample (samples) surface, F — total area of the fragments surface. As it can be seen, the impact energy is not included in the definition, i.e. crack-resistance coefficient is determined in given experiment conditions. The experiment conditions in our case were described above.

Let us calculate the area of initial sample surface. The sample is cylindrical. The base area is equal to 31678 px or 13.75 mm^2. Height is 83 px (1.73 mm), that means the side surface area is equal to 22.73 mm^2 and the total surface area is equal to 50.23 mm^2.

The fragment surface area S can be calculated from fragment projection on glass area S_0 as

$$S = \beta S_0.$$

Coefficient β is determined by the fragment shape. Visually, the fragments are close to plate-like parallelepipeds. For such plates $\beta = \frac{2a^2 + 4ab}{a^2} = 2 + 4\frac{b}{a}$. In our case, the form factor $\alpha \approx 0.6$, so $b/a = 0.6$ and

$\beta = 4.4$. We shall calculate the total fragments area considering only fragments with $S_0 \geq 20$ px, because the quantity of only such fragments can be determined accurately enough. The total area of projections of such fragments is 1519.284 mm^2. So $F = 4.4 \cdot 1519.284 = 6684.85$ mm^2. And the crack-resistance coefficient is equal to $C \approx 133$.

We have mentioned above that this coefficient is determined within a particular experiment, i.e. it depends upon the mass and the form of the sample, the lead method and the injected energy value. The sense of calculation of this coefficient in conditions of our experiment is the following: firstly, we can estimate the values of strength characteristics of an investigated crystal when we know both the value of this coefficient and parameters of the sample and impact device. Secondly, investigating further, in the same conditions, similar (with the same form and mass) samples of sapphire, annealed in special atmosphere, we shall obtain the spectra of crack-resistance coefficient values for different materials in the same conditions. That allows us to watch the changing of material crack-resistance under different kinds of processing.

6. Conclusions

1. Comparison of results obtained by photographing and direct weighing methods showed high applicability of the photographing method. Much smaller scales of fragment masses are accessible for measuring using this method.

2. The distribution of sapphire fragments in shock fragmentation was obtained in the mass range 0.002–100 mg.

3. The form of large-scale distribution asymptotic was established. It is the power distribution with the exponent about −1.5 (for mass distribution) or −2.5 (for size distribution).

4. The crack-resistance coefficient was calculated in conditions of the present experiment.

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Розподіл по розмірах уламків сапфіра при ударній фрагментації

Р.Є. Бродський, П. В. Коневський, Р.І.Софронов

Експериментально отриманий розподіл по розмірах уламків сапфіра в одноударній фрагментації в діапазоні мас уламків від 0,002 до 100 мг. Використано два методи визначення розмірів уламків - метод зважування і метод фотографування з наступним виміром площі зображення. Проведено порівняльний аналіз методів, показана добра згода результатів. Визначено вид крупномасштабної асимтотики розподілу по розмірах фрагментів. Вона має вигляд степеневого розподілу з показником степеня -2,5. На підставі отриманих результатів обчислений коефіцієнт тріщиностійкості.