Forschungszentrum Karlsruhe

in der Helmholtz-Gemeinschaft

Wissenschaftliche Berichte

FZKA 6872

Dust Explosion Experiments

Measurements of Explosion Indices of Graphite Dusts

Report on EFDA Subtask TW2-TSS-SEA5.2

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Forschungszentrum Karlsruhe GmbH, Karlsruhe
2003

Impressum der Print-Ausgabe:

Als Manuskript gedruckt

Forschungszentrum Karlsruhe GmbH
Postfach 3640, 76021 Karlsruhe

Mitglied der Hermann von Helmholtz-Gemeinschaft
Deutscher Forschungszentren (HGF)

ISSN 0947-8620

Abstract

An experimental program was initiated at FZK to evaluate the explosion hazard of ITER-relevant dusts. A standard method of 20-l-sphere was chosen to rank the dusts as explosive amongst other industrial dusts. The facility was built up. As a first step, graphite dusts were tested. Their explosion indices – maximum overpressure and pressure rise rate – were measured as a function of characteristic dust particle size, the latter being tested were 4 µm, 25-32 µm, and 40-45 µm. In addition, lower explosion concentrations and explosibility of these dusts were evaluated. It was shown that graphite dusts with particles sizes from 4 to 40 µm were able to explode in a wide range of concentrations. The dusts tested were ranked as St1 class (the mildest class). Dust particle size was shown to be very important for explosion properties. The finest dust appeared to have lowest minimum explosion concentration (70 g/m^3) and the lowest minimum ignition energy (1 kJ).

STAUBEXPLOSIONSEXPERIMENTE

Messungen von der Explosionskenngrößen von Graphitstaub

Zusammenfassung

Am FZK wurde ein Programm von Experimenten begonnen, mit dem das Explosionsrisiko von ITER-relevanten Stäuben abgeschätzt werden soll. Die Standardmethode einer 20 Liter Kugel wurde gewählt, um die Stäube hinsichtlich ihrer Explosivität unter andere Industriestäube einzuordnen. Die Versuchsanlage wurde aufgebaut. In einem ersten Schritt wurden Graphitstäube getestet. Ihre Explosionskenngrößen, d.h. maximaler Überdruck und maximale Druckanstiegsgeschwindigkeit, wurden als Funktion der charakteristischen Staubpartikelgrösse gemessen, wobei letztere Werte von 4 µm, 25-32 µm und 40-45 µm hatte. Zusätzlich wurden die untere Explosionsgrenze sowie die Explosivität dieser Stäube evaluiert. Es zeigte sich, dass Graphitstäube mit Partikelgrössen von 4 bis 40 µm in einem weiten Konzentrationsbereich explodieren können. Die untersuchten Stäube wurden in Klasse St1 eingeordnet (die am wenigsten gefährliche Klasse). Es konnte gezeigt werden, dass die Grösse der Staubpartikel sehr wichtig für die Explosionseigenschaften ist. Der feinste Staub scheint die geringste minimale Explosionskonzentration (70 g/m^3) und ebenso die geringste minimale Zündungsenergie (1 kJ) zu haben.

Contents

Introduction

One of the ITER safety problems is the explosion hazard of the dusts produced and accumulated during ITER operation inside its vacuum vessel; the dusts under concern are carbon, tungsten, and beryllium dusts [1]. Currently there are no reliable data on the ITER-relevant dust properties characterizing their explosion severity. An important issue is the content of hydrogen in the dusts that could not be evaluated from the data of the existing Tokamaks. So no reliable extrapolation using other dust data are possible at the moment. To address the explosion hazard in ITER, an experimental program was initiated at FZK to study the explosion properties of ITER-relevant dusts.

The objective of the first series of tests was to study the standard explosive characteristics of pure graphite dusts in air. The standard method of 20-l-sphere [2] was chosen for these tests. The main advantage of this method is that the explosion parameters of the ITER-relevant dusts can be compared with those of other industrial dusts and classified as explosives. In addition, important dust explosion parameters, such as minimum explosion concentration, maximum explosion pressure and minimum ignition energy can be evaluated with this method. Effect of the dust particle size on the explosion parameters can be also investigated.

Method description

The standard test method for classification of dust explosibility [2] is to generate a dust atmosphere in spherical bomb of 20 l volume by rapid injection of a dust sample inside the sphere from a dust storage container with a portion of compressed air via a dust outlet valve (see Fig. 1). Then the dust/air cloud, which should be at 1 bar pressure, is ignited at the sphere center. The pressure evolution in the course of the explosion is recorded as a function of time. Maximum explosion overpressure $\mathbf{P_m}$ and rate of pressure rise $\mathbf{(dP/dt)_m}$ are derived from the pressure-time curve at a nominal dust concentration. The dust concentration is varied in a wide range, where the dust can explode, to determine the maximum explosion overpressure $\mathbf{P_{max}}$ and rate of pressure rise $\mathbf{(dP/dt)_{max}}$ - maximum of $\mathbf{P_m}$ and $\mathbf{(dP/dt)_m}$, respecitively. The latter is volume-dependent; by applying the "cubic law" it is converted to $\mathbf{K_{st}}$ value:

$$K_{st}=(dP/dt)_{max}\bullet V^{1/3}$$

which is assumed to be independent on the test vessel volume and is dust-specific. Together with Lower Explosion Limit (LEL) – the minimum concentration at which the dust can explode – $\mathbf{P_{max}}$ and $\mathbf{K_{st}}$ are the explosion indices to be measured.

The $\mathbf{K_{st}}$ value is used to classify the explosibility of a dust:

Dust explosion class	$\mathbf{K_{max}}$ **(m•bar/s)**
St 1	**> 0 - 200**
St 2	**201 - 300**
St 3	**> 300**

A typical pressure time curve obtained using this method is presented in Fig. 2 [3]. Here $\mathbf{P_{ex}}$ is the maximum overpressure measured at nominal dust concentration. As additional amount of air comes into the sphere with dust, and the pressure at the ignition moment must be normal, the sphere must be pre-evacuated. The value of $\mathbf{P_d}$ is this expansion pressure. Standard volume of the dust storage container is 0.6 l; it is pressurized to 21 bar abs. So to provide 1 bar pressure inside the sphere after the dust injection, $\mathbf{P_d}$ must be -0.6 bar (0.4 bar abs). The timing of the process has a critical importance. After the dust injection into the sphere volume the atmosphere inside is turbulized; the turbulence level varies with time. As the effectiveness of the dust explosion depends strongly on the turbulence level, the latter is standardized via prescribing a specific delay (60 ms for the 20-l-sphere) between the start of pressure rise and the moment of ignition – $\mathbf{t_v}$ in the figure. Another important but not standardized parameter is the time delay between the moment of the dust outlet valve opening and the start of the pressure rise in the sphere – $\mathbf{t_d}$ in the picture. It indicates the clearness of the dust outlet valve and dispersion system (rebound nozzle in our case) and their proper operation.

Due to heat losses of the dust-air mixture during combustion, the measured maximum pressure value must be corrected. Another effect, which must be taken into account, is the burst of chemical igniters. As a standard, to measure $\mathbf{P_{max}}$ and $\mathbf{(dP/dt)_{max}}$ two pyrotechnical igniters of 10 kJ total release energy are used. In cases of slow combustion or low overpressures they influence the measured values of $\mathbf{P_m}$ and $\mathbf{(dP/dt)_m}$ which values must be corrected.

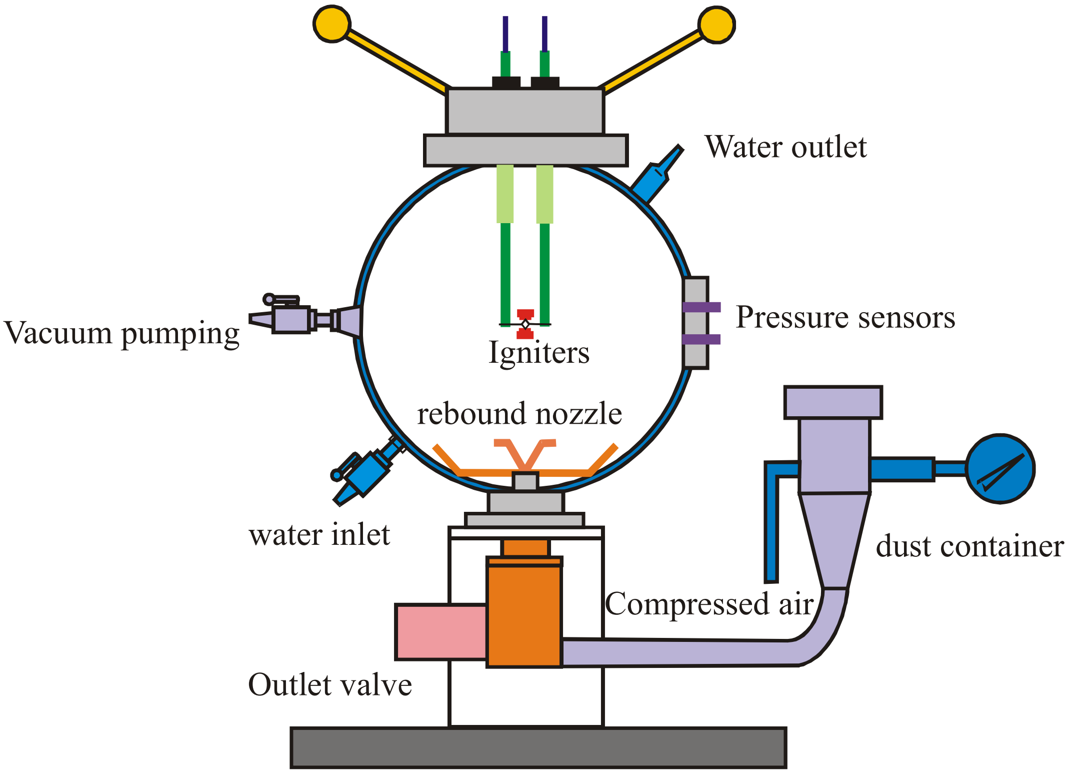

Figure 1. Scheme of 20-l-sphere.

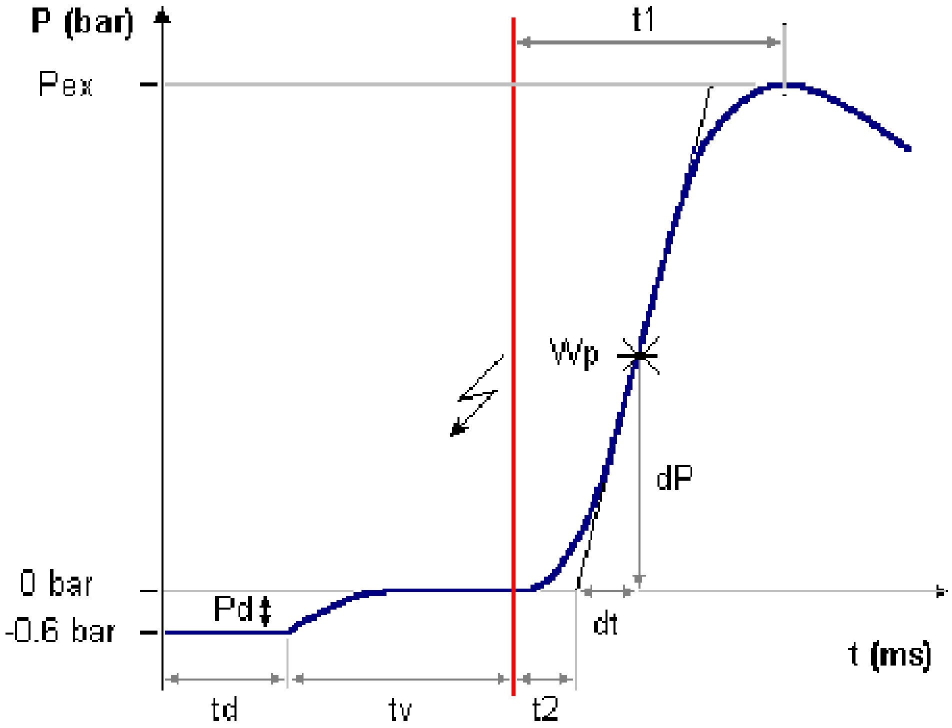

Figure 2. Typical pressure-time curve in 20-l-sphere method.

Experimental details

Facility description. The facility (see photo) consists of a combustion sphere of 20 l volume, a dust storage container connected with the sphere vie a dust outlet valve, a measurement system, a control system, a cooling system, vacuum system, and a compressed air system.

The facility scheme is shown in Fig. 3. The sphere unit (see Fig. 1) includes a flange on its top with bayonet-ring clamp to provide fast opening the sphere for cleaning, two flanges to port measurement gauges, and a vacuum outlet. Design pressure of sphere is 30 bar. There is a water-cooling jacket outside the bomb. To keep thermostatic conditions inside the sphere from test to test, cooling water is supplied into the jacket from a heat-insulated tank (closed-loop system). A refrigerator is used to keep the cooling water temperature below 25 ° C. The sphere is placed under a ventilated hood.

Tested dust cloud is formed inside the sphere by dispersion of a tested dust sample, which is first placed into the dust container and then injected into the sphere with a portion of compressed air via dust outlet valve. There is a rebound nozzle at the bottom of the sphere to facilitate dispersion. The dust outlet valve is activated electrically and driven pneumatically. The driving agent injecting dust sample into the bomb is also compressed air: before the test start, the dust container is pressurised with normal compressed air. To provide that, compressed air bottles are used (P>40 bar). They are connected with the dust outlet valve and the storage container via reductors and valves.

The initial pressure of the compressed air in the dust storage container before the injection is 21 bar abs., its volume is 0.6 l. So to provide a normal pressure (1 bar) inside the sphere after the dust injection before the ignition, the sphere is pre-evacuated to 0.4 bar abs by a vacuum pump with dust filter. The vacuum pressure is measured with 0.1 % accuracy; the pressure in the container – with 0.5 % accuracy.

The dust/air cloud is ignited at the sphere centre by two pyrotechnical igniters. The igniters are placed at the end of two electrode rods mounted at the top flange; the igniters are activated electrically (24 V, 4 A).

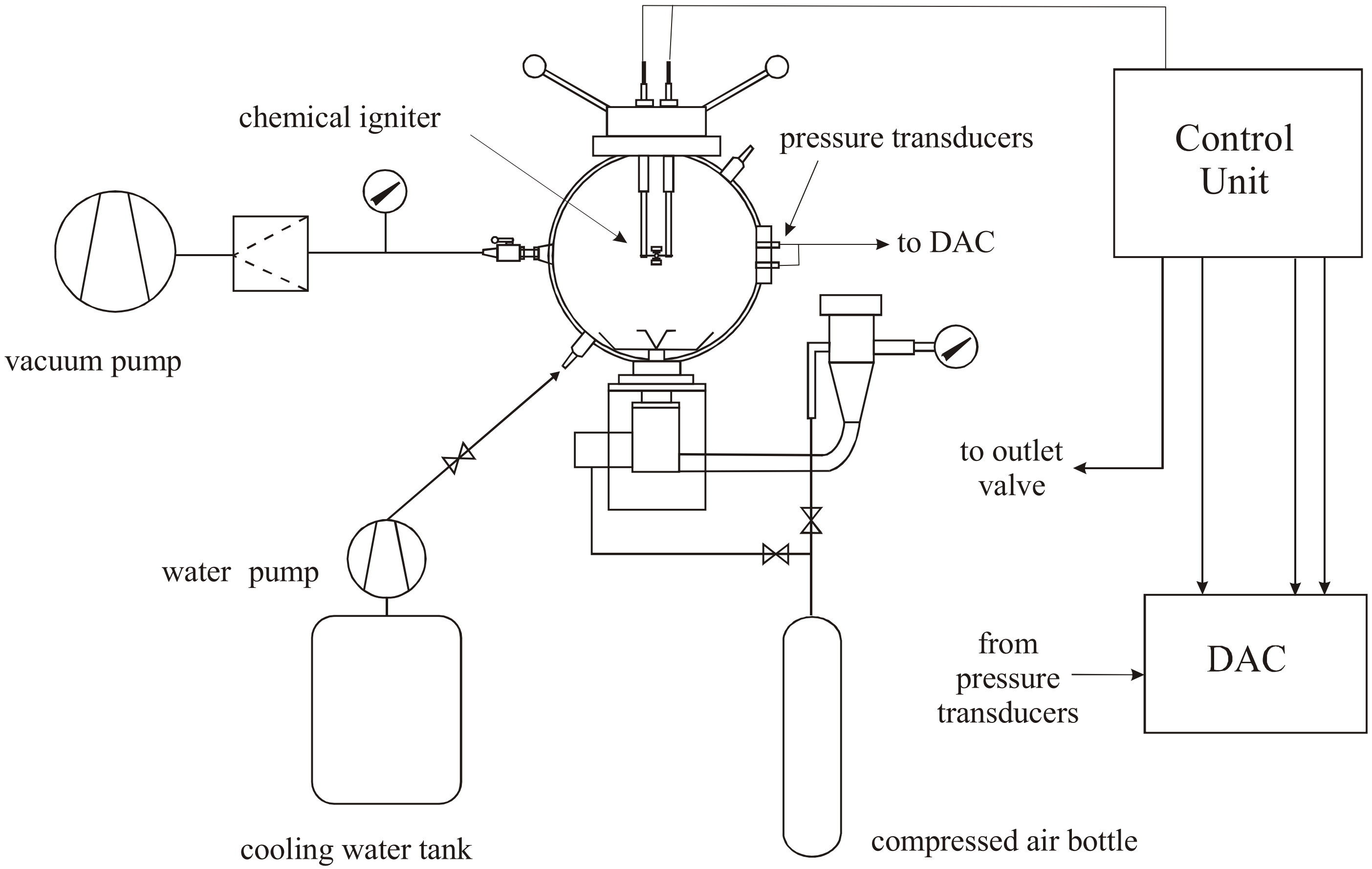

Figure 3. Scheme of the DUSTEX facility

The pressure-time dependence during dust explosion is measured with two pressure transducers: piezoelectric PCB113Aand piezoresistive Kulite KE-375 (500 and 160 kHz natural frequency, 1 and 1.5 % uncertainty, 0.368 and 0.112 V/bar sensitivity, respectively) with their faces flush with the sphere wall. The piezoelectric pressure transducer is heat-sensitive, so its face is protected by silicon lubricate. The transducers are mounted onto the sphere via teflon gaskets to avoid electric contact with the sphere and reduce the sonic noise. The piezoresistive transducer was calibrated at constant pressures up to 15 bar with a precise manometer. Data acquisition system is based on a 12-bit ADC controller installed in IBM PCs (Keithley KPCI-3103 type, up to 400 kHz sampling rate). The pressure transducer outputs are digitized at 5 kHz sampling rate, 2,000 to 10,000 measurements per channel/shot. Simultaneously, the timings of the dust outlet valve operation and of the ignition activation current are recorded. The controller is operated under "TestPoint" software package.

The standardized parts of the facility – spherical bomb of 20 l volume, dust storage container, and the outlet valve – and the pyrotechnical igniters were purchased, the other parts were manufactured at FZK.

Test procedure. The tests are executed as follows. Before the test a dust sample is charged to the storage container; a pair of igniters is connected to the rods and the rods are connected to the ignition lines; the sphere is pre-evacuated to 400 mbar; the dust container with dust sample inside is filled up with compressed air to 21 bar abs. At the test start the control system (1) t=0 ms: triggers the date acquisition board and activates the dust outlet valve to open, (2) t=90 ms: closes the dust valve, (3) t=100 ms: activates the igniters, and (4) t=120 ms: switches off the ignition activation supply. In addition, the control system prevents ignition if the bayonet flange and vacuum valve are not closed. After the test the sphere is evacuated, rinsed with compressed air, and cleaned, together with dust container, with vacuum cleaner. Before a test series with new type of dust the sphere, dust container, and dust outlet valve are cleaned deeply with full disassembling.

A test is considered as successful if:

1. the pressure inside the sphere (measured by the transducers) starts to rise from 30 to 50 ms since the dust outlet valve opening (in this case the dust valve and dispersion system are considered as clean and operating properly)
2. the time delay between the start of pressure rise and moment of ignition is 60±5 ms
3. the expansion pressure (difference between the initial pressure and the pressure at ignition) measured by pressure transducers is in the range of 0.55 to 0.7 bar
4. the difference between the maximum recorded pressure values of the two transducers does not exceed 0.3 bar
5. the difference of the rate of pressure rise of the two transducers differs less than 10% of the mean value.

Dust sample preparation. Two types of pure graphite dust (99.8 %) were used in the tests. The first one has a 4 μm characteristic particle size, the other one is characterized as > 32 μm. The samples were tested on hydrogen content. Hydrogen in the first sample was below the detection limit of 0.005%; in the second sample 0.265% hydrogen were found.

The samples before testing were dried at 190 °C during 15 min in SARTORIUS moisture analyzer MA 45; the moisture content was less than 1%. The finest dust was tested with no further treatment. Figure 4 presents a TEM photo of this dust. Using the photo, the particle size distribution was measured; the distribution is shown in Fig. 5. The coarser dust was sieved in RETSCH S100 sieving machine with the following sieve set: 56, 53, 50, 45, 40, 36, 32, 25, and 20 μm. Two fractions from this set were tested: 25 to 32 and 40 to 45 μm. A dust sample to test was weighed with SARTORIUS CP2202S balances (0.01 g accuracy). Tested concentrations ranged from 60 to 750 g/m^3, which corresponds to 1 to 15 g of dust sample weight. After a test only small amount of dust resides in the dust container.

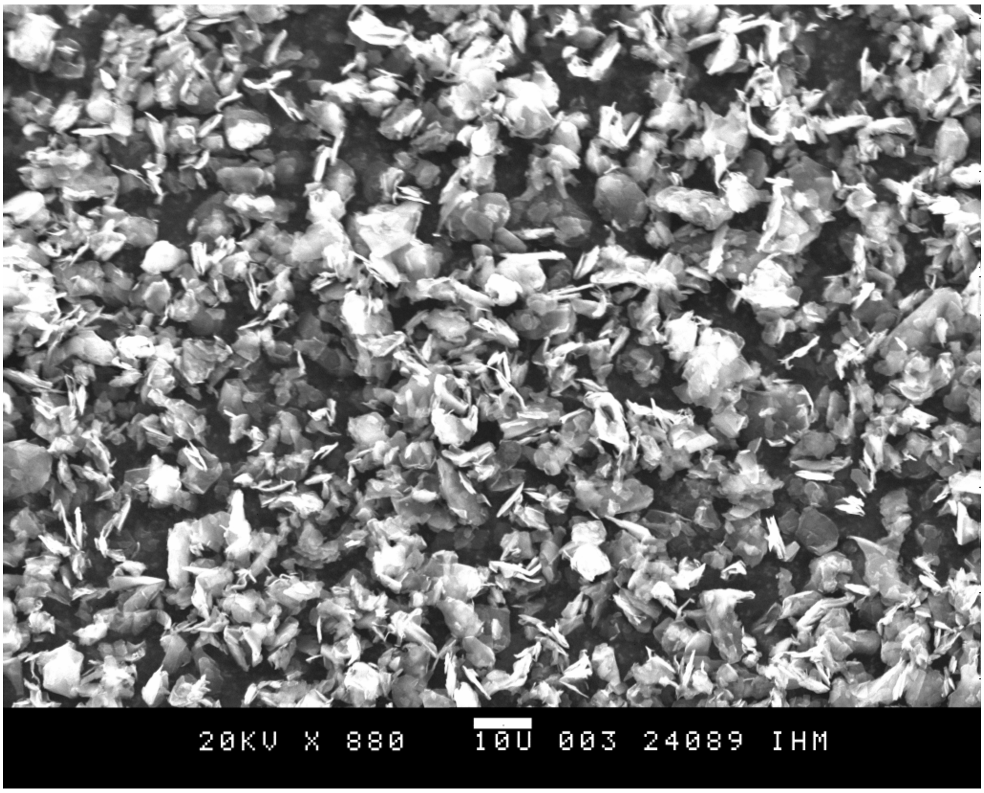

Figure 4. TEM photo of 4 µm graphite dust.

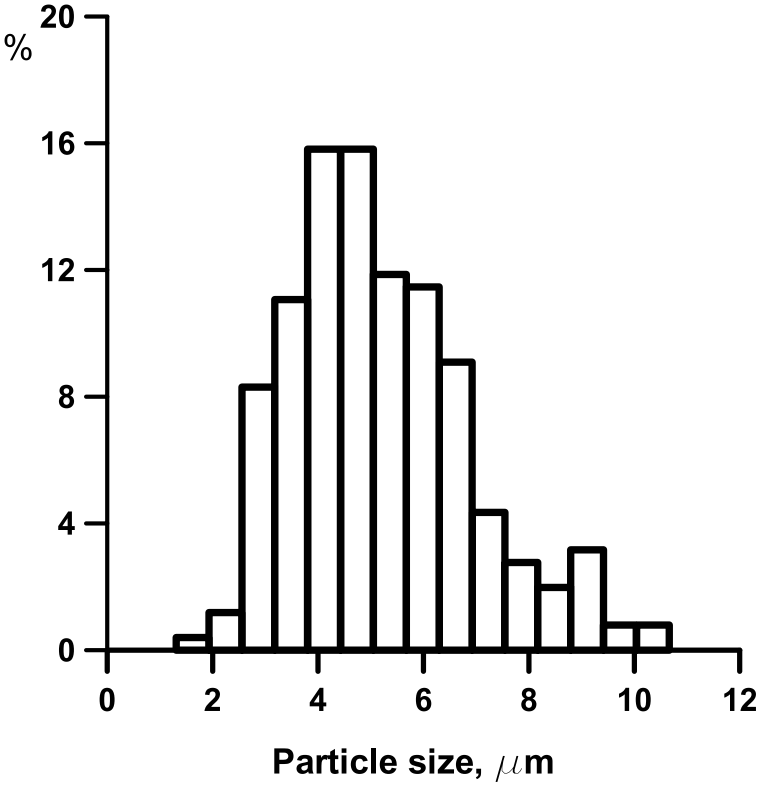

Figure 5. Particle size distribution of 4 µm graphite dust.

Pressure measurements processing. Two effects influence pressure values measured in the 20-l-apparatus. First one is cooling effect. A correction is made according to the formula [3]:

$$\mathbf{P_m = 0.775 \bullet P_{ex}^{1.15}}$$

where P_{ex} is maximum pressure recorded during the test.

The second one is the additional pressure generated by the chemical igniters. A blind test with 10 kJ energy igniters only gives maximum overpressure of 1.6 bar. The correction of this effect is made according to the equation [3]:

$$\mathbf{P_m = 5.5 \bullet (P_{ex} - P_{ci}) / (5.5 - P_{ci})\ bar}$$

where P_{ci} is pressure due to chemical igniters, P_{ci}=1.6bar•IE/10,000;
IE is igniters release energy in joules.

Another effect of chemical igniters is important in case of “slow” dusts characterized by less then 150 bar/s pressure rise. As 10 kJ igniters produce approximately 100 bar/s, their effect must be taken into account. As blind tests show, this effect is terminated after 50 ms. In such cases the value of $(dP/dt)_m$ was derived at least 50 ms after ignition.

Measurement results

The test results are presented in Table 1. Here C_{dust} is dust concentration determined as mass of dust sample divided by 20 l; P_m is a corrected maximum overpressure recorded in the test, $(dP/dt)_m$ is maximum value of pressure rise. Three graphite dust samples were tested differing in their characteristic particle size: 4 μm, 25-32 μm, and 40-45 μm. The effect of dust concentration and the lower concentration limits were measured for these dusts; in these tests the igniters' release energy was 10 kJ. In addition, the minimum ignition energy was evaluated in explosibility measurements. In the latter tests 10, 2, and 1 kJ energy igniters were used.

Effect of dust concentration. Results of measurements of maximum overpressures and pressure rise rates versus dust concentration are presented in Figs. 6-11 for graphite dusts with different characteristic particle sizes. Typical pressure-time curves are shown in Figs. 12 (4 μm dust), 13 (25-32 μm dust), and 14 (40-45 μm dust). Note that the finest dust burns much faster than coarse dusts. In case of 25-32 μm and 40-45 μm dusts the above mentioned effect of chemical igniters is clearly seen (Figs. 13 and 14). In these cases the value of $(dP/dt)_m$ was derived at least 50 ms after the ignition.

For all the tested dusts their maximum explosion overpressures rise with dust concentration from lower explosion limits on, reach maximum at 250 g/cm^3 (which is about two times higher the stoichiometric concentration for the reaction $C+O_2=CO_2$), and then decreases only slightly. The pressure rise rates also increase first with dust concentration and reach the maximum at about 250 /cm^3. Further behavior differs for the finest and coarser dusts. After reaching its maximum, $(dP/dt)_m$ noticeably decreases with concentration in case of 4 μm dust, while for coarser dusts it remains almost the same. Summary plots of $P_m(C_{dust})$ and $(dP/dt)_m(C_{dust})$ are presented in Figs. 15 and 16.

Table 1. Measurement results of graphite dust explosion characteristics.

Test name	**Dust size**	**C_{dust}**	**Ignition energy**	**P_m**	**dP/dt_m**
	µm	**g/m^3**	**kJ**	**bar**	**bar/s**
gd001, 20	**4**	**60**	**10**	**0.4**	**0**
gd21, 24,26, 28	**4**	**70**	**10**	**0.4**	**0**
gd22	**4**	**80**	**10**	**1.5**	**25**
gd002, 01, 02	**4**	**125**	**10**	**6.2**	**80**
gd05, 06	**4**	**200**	**10**	**6.5**	**180**
gd003, 03, 04	**4**	**250**	**10**	**6.6**	**250**
gd07, 08	**4**	**400**	**10**	**6.0**	**195**
gd004, 09,10	**4**	**500**	**10**	**5.3**	**135**
fd52, 53	**4**	**750**	**10**	**4.6**	**45**
gd11	**4**	**125**	**2**	**0.4**	**0**
gd12, 18, 19	**4**	**250**	**2**	**5.1**	**40**
gd13	**4**	**500**	**2**	**4.6**	**30**
gd14	**4**	**150**	**2**	**0.4**	**0**
gd15	**4**	**200**	**2**	**0.4**	**0**
gd16	**4**	**250**	**1**	**0.1**	**0**
gd17	**4**	**500**	**1**	**0.1**	**0**
gd29	**25-32**	**60**	**10**	**0.0**	**0**
gd31	**25-32**	**70**	**10**	**0.0**	**0**
gd33	**25-32**	**80**	**10**	**0.0**	**0**
gd34	**25-32**	**100**	**10**	**0.1**	**0**
gd35	**25-32**	**120**	**10**	**1.1**	**<10**
gd47	**25-32**	**150**	**10**	**4.4**	**35**
gd48	**25-32**	**200**	**10**	**5.9**	**50**
gd39	**25-32**	**250**	**10**	**6.0**	**90**
gd54	**25-32**	**300**	**10**	**5.8**	**80**
gd49	**25-32**	**500**	**10**	**5.9**	**55**
gd50	**25-32**	**750**	**10**	**5.3**	**75**
gd40	**25-32**	**250**	**2**	**0.0**	**0**
gd36	**40-45**	**120**	**10**	**0.2**	**0**
gd37	**40-45**	**140**	**10**	**4.6**	**40**
gd43	**40-45**	**200**	**10**	**5.3**	**60**
gd38	**40-45**	**250**	**10**	**6.1**	**60**
gd44	**40-45**	**500**	**10**	**5.1**	**70**
gd45	**40-45**	**750**	**10**	**5.2**	**60**
gd41	**40-45**	**250**	**2**	**0.0**	**0**

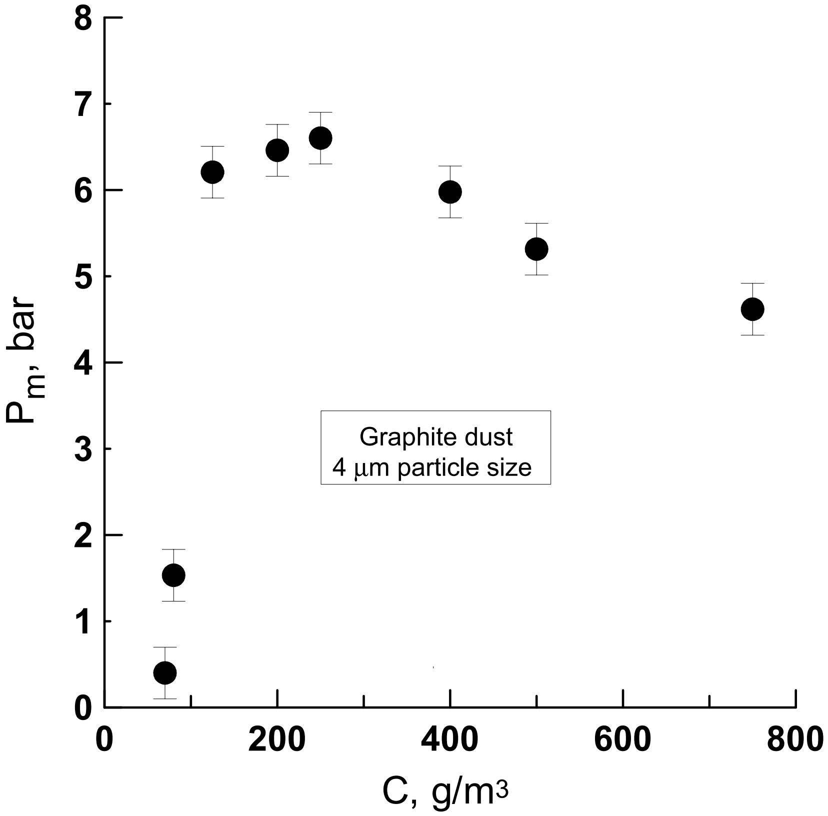

Figure 6. Maximum overpressure versus concentration for 4 µm graphite dust.

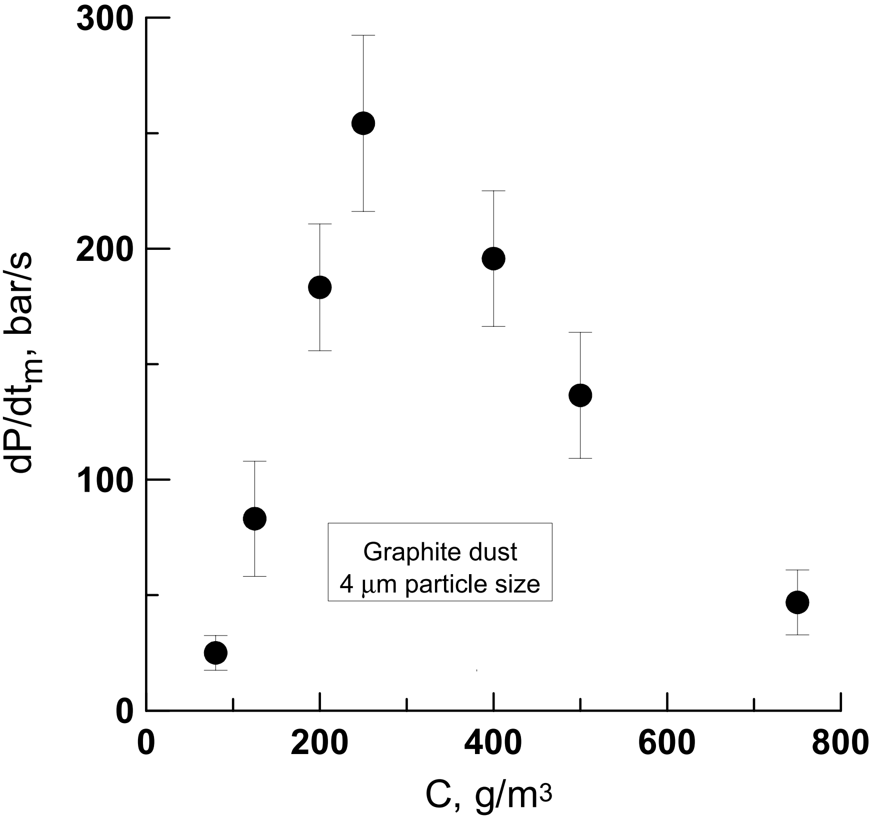

Figure 7. Maximum pressure rise rate versus concentration for 4 µm graphite dust.

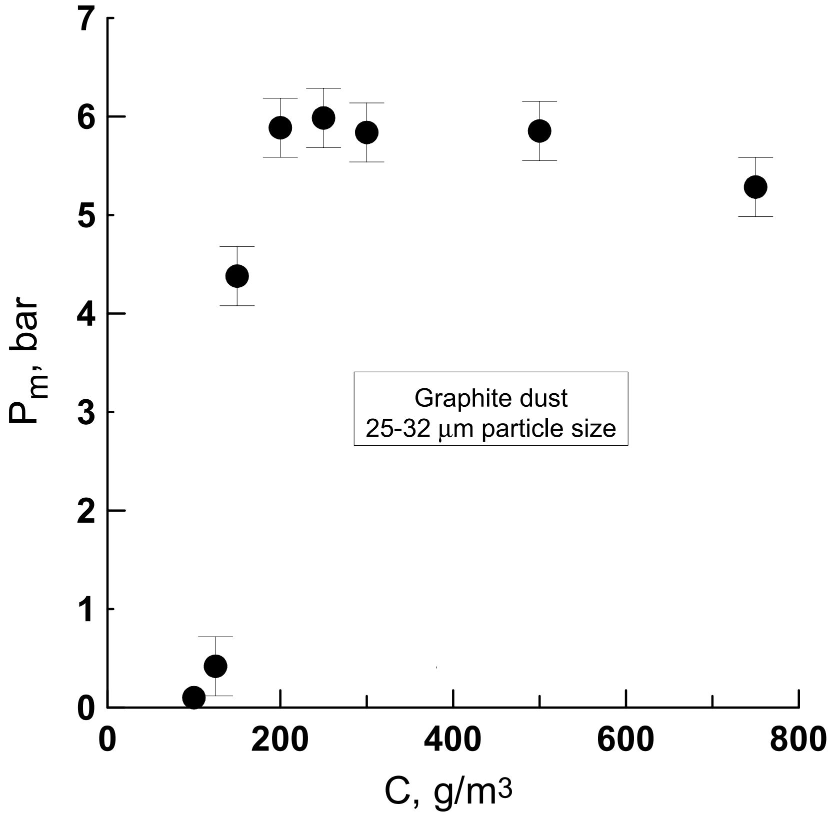

Figure 8. Maximum overpressure versus concentration for 25-32 µm graphite dust.

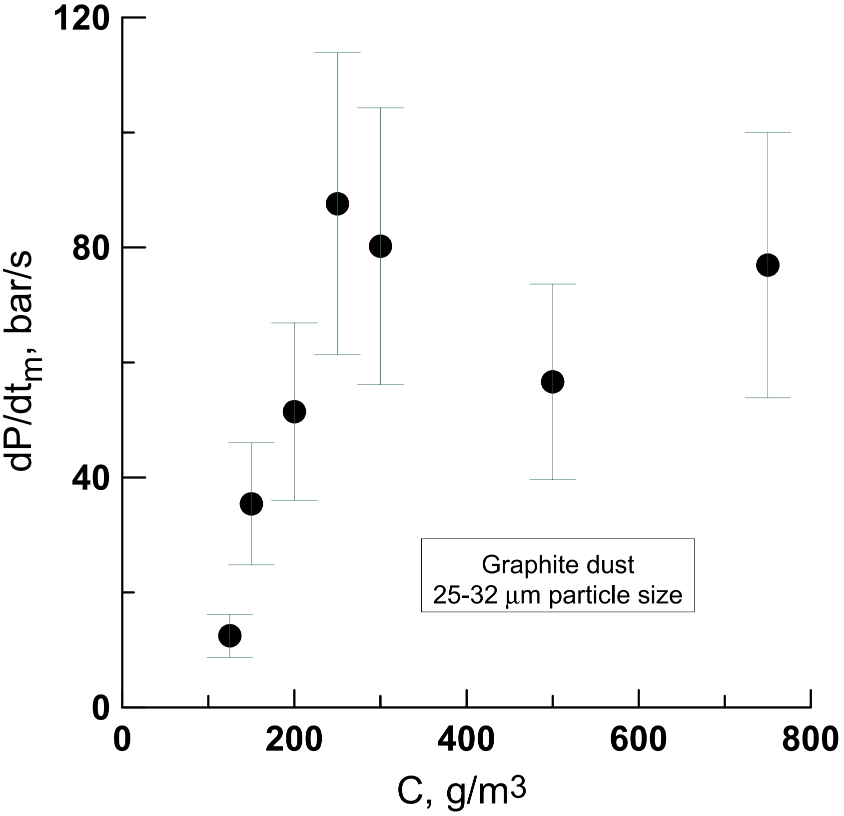

Figure 9. Maximum pressure rise rate versus concentration for 25-32 µm graphite dust.

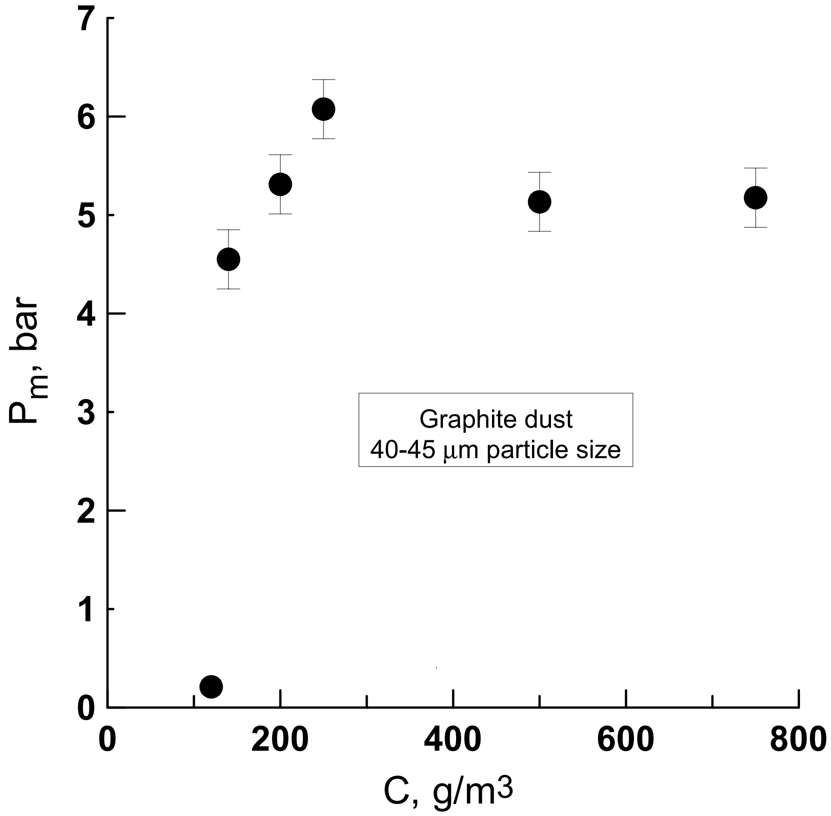

Figure 10. Maximum overpressure versus concentration for 40-45 μm graphite dust.

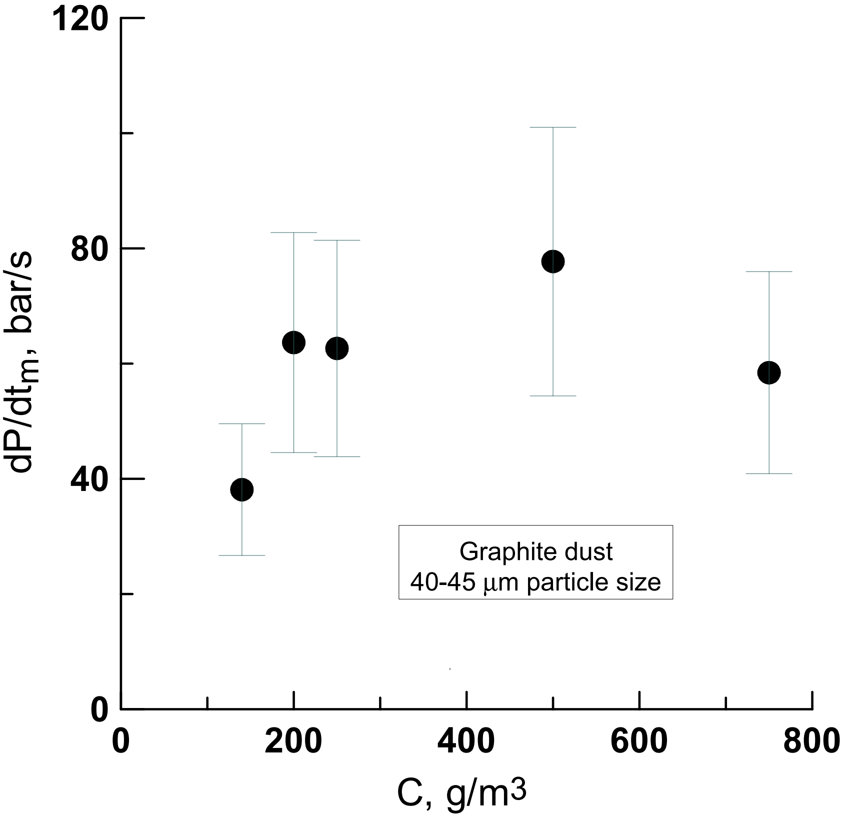

Figure 11. Maximum pressure rise rate versus concentration for 40-45 μm graphite dust.

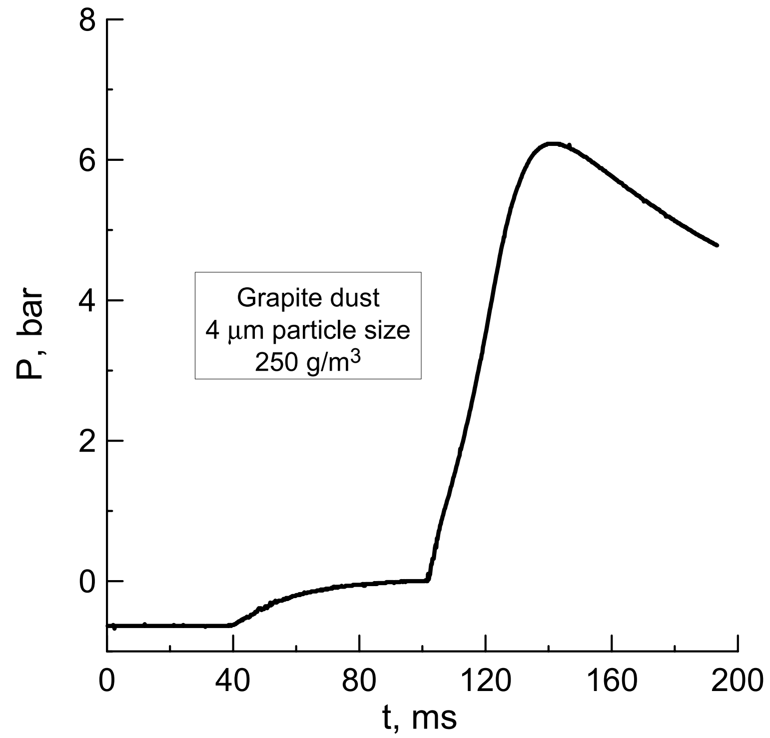

Figure 12. Pressure-time dependence in graphite/air dust mixture explosion. 4 µm particle size, 250 g/m^3.

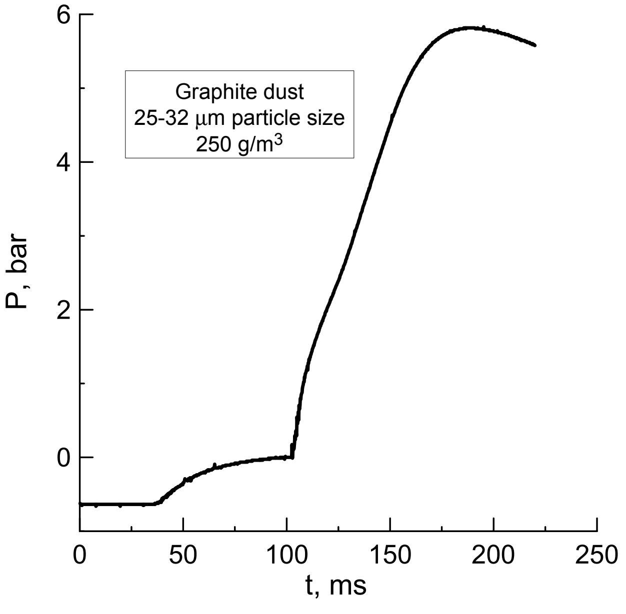

Figure 13. Pressure-time dependence in graphite/air dust mixture explosion. 25-32 µm particle size, 250 g/m^3.

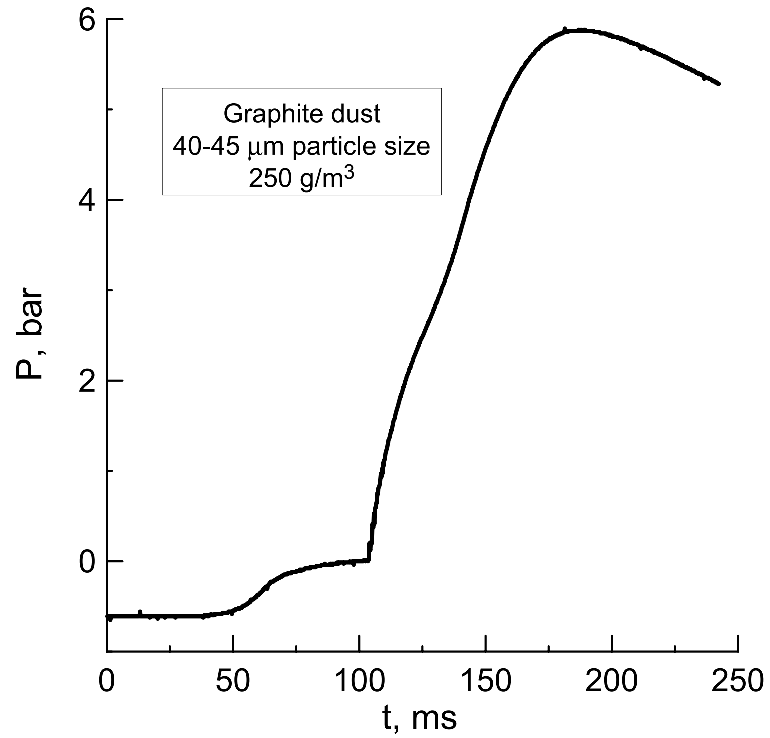

Figure 14. Pressure-time dependence in graphite/air dust mixture explosion. 40-45 µm particle size, 250 g/m^3.

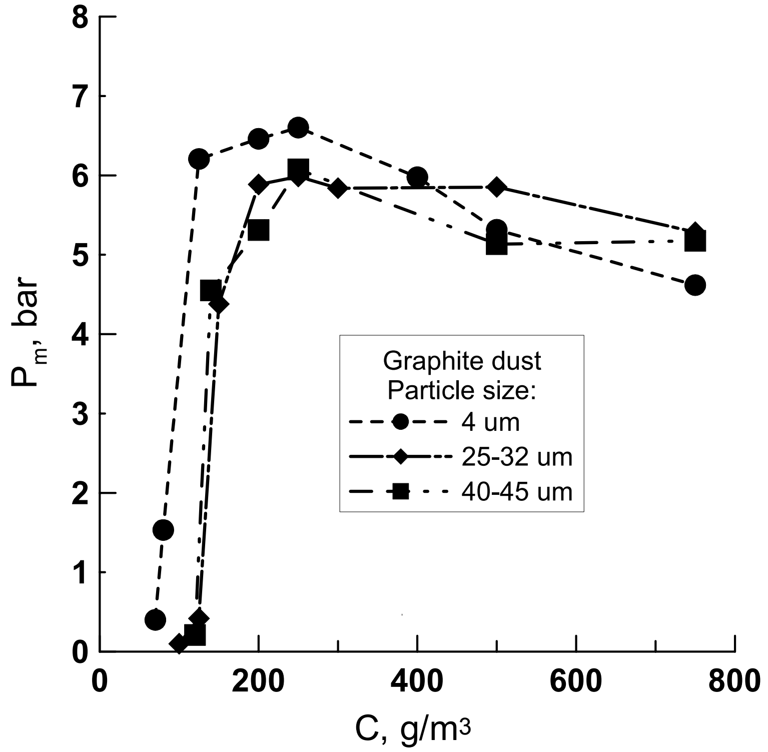

Figure 15. Dependence of maximum overpressure on dust concentration for 4, 25-32, and 40-45 μm graphite dusts.

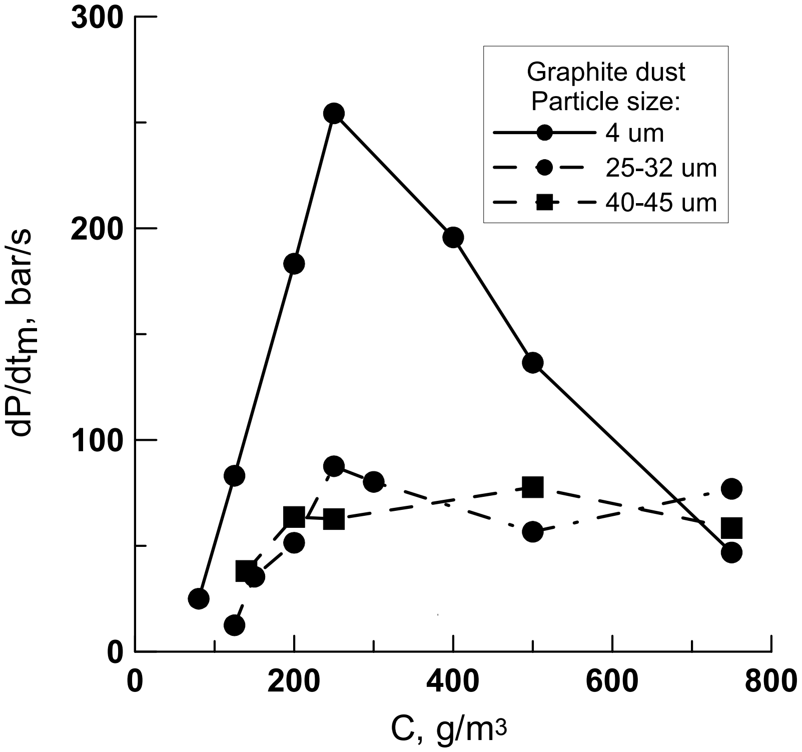

Figure 16. Dependence of maximum pressure rise rates on dust concentration for 4, 25-32, and 40-45 μm graphite dusts.

Effect of dust particle size. The measured results of explosion indices versus dust particle size are summarized in Fig. 17. One can see that P_{max} – maximum overpressure over the whole C_{dust} range – does almost not depend on particle size. The highest P_{max} value – 6.6 bar – was observed for 4 µm dust. It decreases with dust particle size to 6 bar at 40-45 µm, which is almost in the range of measurement accuracy. However, the other explosion index, maximum pressure rise rate, strongly depends on the dust particle size. Maximum value of $(dP/dt)_{max}$ – 250 bar/s – features the finest dust; this one is the most dangerous dust. With particle size it rapidly decrease down to 80 bar/s at 40-45 µm. As for K_{st} value, it ranges from 70 bar•m/s for 4-µm dust to 20 bar•m/s for the coarsest dust. It means that graphite dusts even for the finest tested dust belong to class ST1, the mildest one.

Lower explosion limits. The lower explosive concentrations were measured as follows [3]. The sphere, dust outlet valve, and dust container were thoroughly cleaned before each test. A test series was initiated with 50 g/m^3 and then continued with a systematic step-wise increase of 10 g/m^3 dust concentration until ignition of the dust\air mixture was observed. A test was reckoned as explosive if the maximum overpressure was higher than 0.4 bar, the igniters input having been taken into account. Then the test with 10 g/m^3 concentration lower was repeated, and the reduction was continued in further tests until no ignition was observed in three repetitive tests separated by a blank test (no dust, igniters only). The latter concentration is reported as the lower explosion limit.

The results of LEL measurements for graphite dusts are presented in Fig. 18, where the dependence of LEL on dust particle size is plotted. The lowest limit features the finest dust – 70 g/m^3. As it can be seen from the results, the lower explosion limit strongly depends on the dust size in the tested range. It is almost two times higher for 40-45 µm dust.

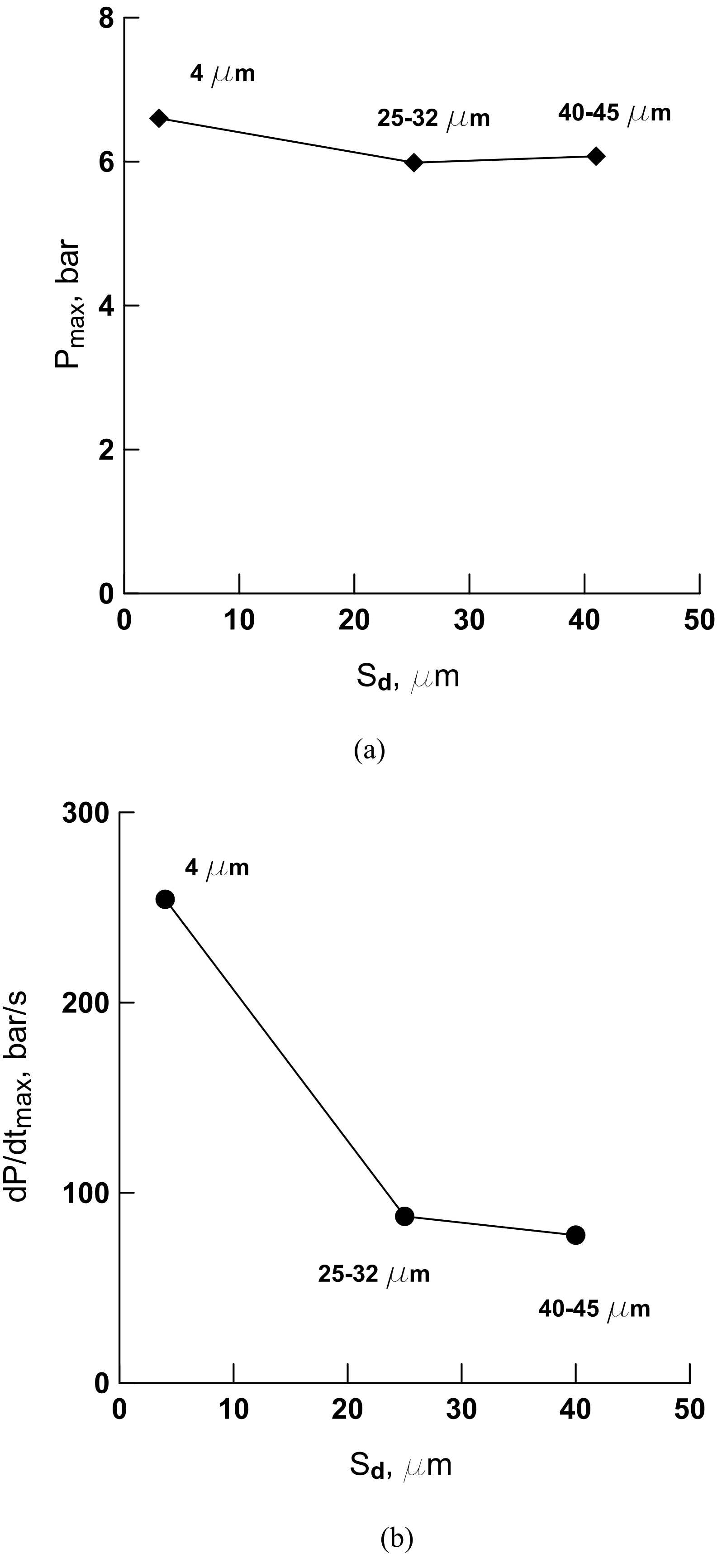

Figure 17. Explosion indices of graphite dust versus dust particle size.

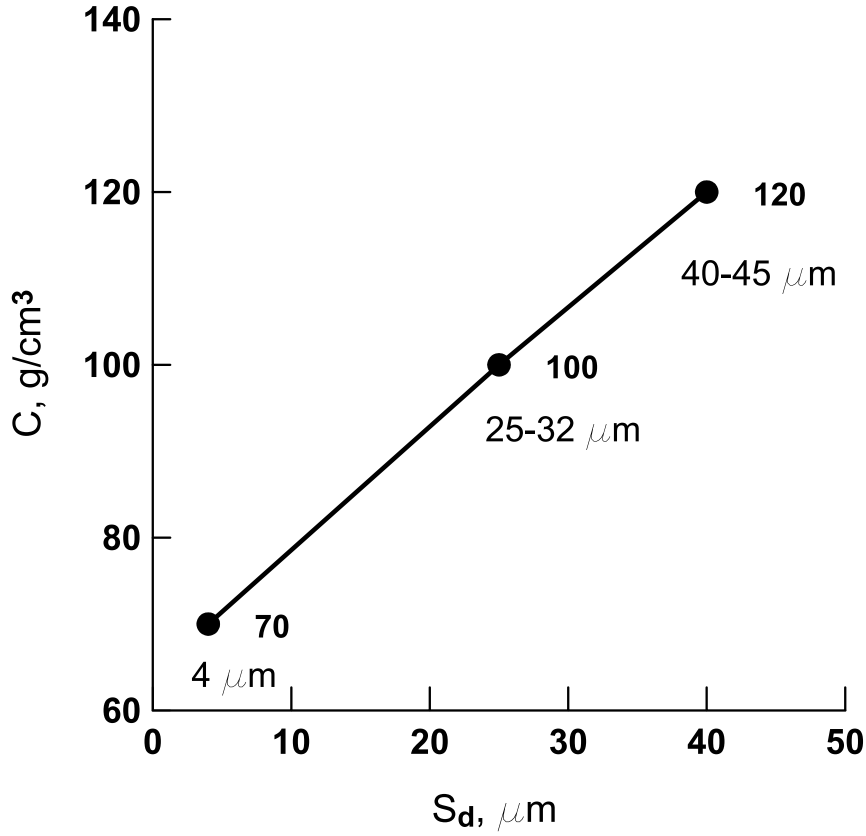

Figure 18. Lower explosive concentration of graphite dusts versus dust particle size.

Explosibility measurements. Explosibility of the tested graphite dusts and their minimum ignition energy were estimated using the method of 20-l-sphere. The possibility for a dust-air mixture to explode depends strongly on the ignition energy. Thus the ignition energy is an important parameter classifying a dust as explosible or not. It is suggested [3] that a dust is not explosible if it cannot be induced to explode in a wide range of concentrations at ignition energy equal 2 kJ. We should note that if a dust is classified as non-explosible according to this criterion, it is still possible to expect dust explosion with ignition energy exceeding 2 kJ.

The criterion for a test to be explosive is: maximum overpressure should exceed 0.2 bar (P_{ex}>0.5 bar). The test results are shown in Fig. 19. The finest dust was tested in a wide range of concentrations, from 125 to 500 g/m^3, with two chemical igniters of 1 kJ release energy each. The dust appeared to explode in this range. Then it was tested at 1 kJ ignition energy at two concentrations, at which the maximum values of explosion indices were measured at 2 kJ ignition energy – 250 and 500 g/m^3. In these tests the dust did not explode.

The coarser dusts were not tested in wide concentration range due to limited amount of available dust. Instead “the most dangerous concentration” of 250 g/m^3 was tested at 2 kJ ignition energy. No ignition was observed in these tests.

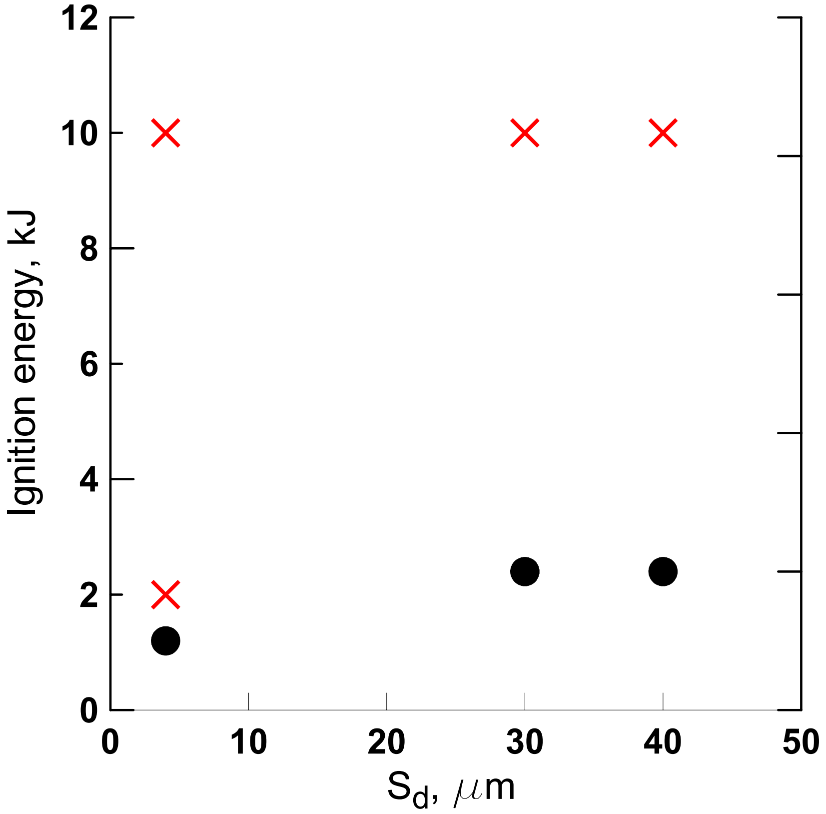

Figure 19. Explosibility of graphite dust versus particle size. Crosses – tests with explosions, circles – tests without explosion.

Summary and conclusions

- Standard methodology (20-l-sphere) was chosen as a first step to evaluate explosion properties of ITER relevant dusts
- Experimental facility was designed and constructed at FZK to study explosion properties of dusts
- Series of tests were carried out with graphite dusts
- Graphite dusts with particles sizes from 4 to 40 µm were able to explode in a wide range of concentrations
- Dusts studied were ranked as St1 class (the mildest class)
- Dust particle size was shown to be very important for explosion properties
- The finest dust appeared to have lowest minimum explosion concentration (70 g/m^3) and the lowest minimum ignition energy (1 kJ)

References

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