Dust Explosion Experiments

Measurements of Explosion Indices of Tungsten Dusts and Graphite-Tungsten Dust Mixtures

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Report on EFDA Subtask TW3-TSS-SEA5.2

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ABSTRACT

Explosion indices of tungsten dusts and graphite/tungsten dust mixtures are measured using a standard method of 20-l sphere. Three tungsten dusts differing in characteristic particle size, 1, 5, and 12 μm, are tested. Two coarser dusts do not appear to explode under the standard conditions; the finer dust is able to explode in a wide range of concentrations. The maximum overpressure and rate of pressure rise attain at least 4.7 bar and 260 bar/s, respectively. The explosion indices of graphite/tungsten dust mixtures composed of 1- μm tungsten and 4- μm graphite dusts are measured. The maximum overpressure measured for the mixtures decreases monotonically with graphite fraction, while maximum rate of pressure rise measured for the mixture composed of equal mole fractions of graphite and tungsten is 1.5 times higher than that measured for the pure tungsten or graphite dusts alone. The explosibility of this mixture is tested; the mixture can be induced to explode at 2 kJ ignition energy and does not explode at 1 kJ ignition energy. The lower explosion limits are measured for the tested graphite/tungsten dust mixtures.

STAUBEXPLOSIONSEXPERIMENTE
Messungen der Explosionskenngrößen von Wolframstaub und Graphit-Wolframstaub-Mischungen

ZUSAMMENFASSUNG

Explosionskenngrößen von Wolframstaub und Graphit-Wolfram-Staubmischungen wurden mit der Standard-20-Liter-Kugel-Methode gemessen. Zunächst wurden drei Wolframstäube mit Korngrößen von 1, 5 und 12 μm getestet. Es zeigte sich, dass die zwei gröberen Stäube unter Standardbedingungen anscheinend nicht explodieren, während der feine Staub in einem weiten Konzentrationsbereich explodieren kann. Die Maximalwerte des Überdrucks und der Druckanstiegsrate betragen dabei mindestens 4,7 bar bzw. 260 bar/sek. Des weiteren wurden die Explosionskenngrößen von Graphit-Wolfram-Staubmischungen gemessen, die aus Wolframstaub von 1 μm und Graphitstaub von 4 μm Korngröße bestehen. Die folgenden Messergebnisse wurden erhalten:

- Der maximale Überdruck nimmt monoton mit dem Graphit-Anteil ab.

- Die maximale Druckanstiegsrate für eine Mischung, die aus gleichen Molanteilen an Graphit- und Wolframstaub besteht, ist 1,5 mal höher als die Rate für reinen Wolfram- oder Graphitstaub.

Außerdem wurde die Explosionsfähigkeit dieser Mischung untersucht. Es ergab sich, dass diese Mischung mit 2 kJ Zündungsenergie zur Explosion gebracht werden kann, nicht aber mit 1 kJ.

Die unteren Explosionsgrenzen der untersuchten Graphit-Wolfram-Staubmischungen wurden ebenfalls gemessen.

EXECUTIVE SUMMARY

Following the graphite dust tests made in year 2002, tungsten dusts and tungsten/graphite dust mixtures are tested using the standard method of 20-l-sphere. Three tungsten dusts differing in characteristic particle size, 0.6-09 µm, 5 µm, and 12 µm, are tested. The tungsten/graphite mixtures are composed of 0.6-0.9 µm tungsten and 4 µm graphite dusts; the mixtures of the tungsten-to-graphite molar ratios W/C=1/30, 1/4, 1/1, and 3/1 are tested. The maximum overpressure, maximum rate of pressure rise, and lower explosion concentration limit are measured of the finer tungsten dust; the coarser dusts do not appear to explode under the standard method conditions in the concentration range of 250 to 6000 g/m^3 (12-µm dust) and 300 to 7000 g/m^3 (5- µm dust). The 1-µm tungsten dust is tested in the concentration range from 250 to 7500 g/m^3. The lower explosion limit of this dust is measured to be 450 g/m^3. Starting from this limit, the maximum overpressure increases first with concentration, reaches its maximum of at least 4.7 bar at 5000 g/m^3 and then slowly decreases. The pressure rise rate shows the same behavior, its maximum is reached at the same concentration to be at least 260 bar/s. These values are measured at initial pressure lower 1 bar due to rather high explosive concentrations; the correction to the 1 bar initial pressure gives the calculated values of 5.7 bar and 310 bar/s, respectively.

The explosion characteristics of graphite/tungsten dust mixture are studied. To study the conservative case, the mass amount of each mixture in the tests is chosen so that the mixture combustion consumes all the oxygen in the dust cloud atmosphere. The mass concentrations tested are 410 g/m^3 (W/C=1/30), 1100 g/m^3 (W/C=1/4), 2300 g/m^3 (W/C=1/1), and 3300 g/m^3 (W/C=3/1). The maximum overpressure measured for these mixtures decreases slightly with increasing tungsten content. The maximum rate of pressure rise has a pronounced peak of 360 bar/s at W/C=1/1, i.e. this mixture burns faster than both pure graphite and pure tungsten dusts (250 bar/s) alone.

The lower explosion concentration limits are measured of the tungsten/graphite mixtures as a function of mixture composition. The lowest explosive molar concentration decreases monotonically with increasing tungsten content from 5.8 mol/m^3 for pure graphite to 2.4 mol/m^3 for pure tungsten.

The explosibility of the "fastest" tungsten/graphite dust mixture (W/C=1/1) is tested. The mixture appears to be explosive at 2 kJ igniter energy and does not explode at 1 kJ.

CONTENTS

1. INTRODUCTION

During the operation of a tokamak, small-size particulates are produced and accumulated inside its vacuum vessel as a result of interaction of plasma with the tokamak plasma-faced components. The particulates can have a form of flakes, globules, chunks, and other debris. They are found in operating fusion machines as re-deposited films of eroded material, which are friable and weakly adhered to their underlying surfaces. They can be easily broken up to form fine dusts that are capable of being mobilized. There are extensive data available in literature on the properties of the dust collected in operating tokamaks DIII-D, TFTR, ALCATOR-Cmod, Tore Supra, ASDEXUpgrade, and T-10 [1-8]. The dusts collected have been analyzed on their chemical composition, characteristic particle size and specific surface area, and dust concentration. The measurement results have been reviewed comprehensively in [9, 10]. The dust chemical compositions matched mainly the material of the tokamak walls faced by the plasma (C in carbon machines, Be and Mo in metal tokamaks). An important feature of tokamak dust layers was found, i.e. the carbon films deposited onto cool surfaces, which were shaded from plasma, might contain appreciable amounts of co-deposited fuel hydrogen isotopes; the atomic ratio D/C could reach almost unity. Characteristic particle size of the collected dusts ranged from a 0.5 μm to tens of micron, though some data are reported on the dusts having tens-nanometer characteristic particle size [7]. Most of the collected dust samples featured log-normal size distribution. The dust particle shapes were characterized by low aspect ratios (maximum to minimum particle sizes) that was confirmed by the measurements of the dust specific surface areas. Mostly the measured values differed not so much from that of monodisperse spherical particles, though in some cases the difference was several tens times greater indicating significant agglomeration or porosity of the analyzed dust.

Though found in every fusion machine, the dusts have posed neither safety nor operational problems and been recognized as a safety issue in designing the next-step tokamak International Experimental Thermonuclear Reactor (ITER). With the increase in the tokamak duty cycle, the longer pulse duration, and the higher heat loads during normal operation, the dust generation will increase. Due to their origin, the tokamak dusts can be radioactive, chemically reactive and/or toxic. In case of air or steam ingress into the tokamak vacuum vessel the dusts can be oxidized either delivering hydrogen in reaction with steam or directly exploding.

How hazardous the dust may be is determined by their chemical composition, characteristic particle size, and accumulated mass. As the plasma-faced components, ITER design employs beryllium, carbon, and tungsten. The safety approach developed for ITER to confine the dust and limit its chemical reactions is based on assuming administrative guidelines for the maximum tolerable amount of dust mass at locations inside the vacuum vessel [11, 12]. Two inventory limits have been defined, the total amount of dust in the vacuum vessel available for accidental release is limited due to radiological and toxicological concerns, and the quantity of dust residing on hot surfaces of divertor is limited due to steam chemical reactivity concerns. Inventory guidelines are set to limit the mobile activation product inventory inside the VV, to ensure that chemical reactivity is adequately controlled, and to avoid the hazard of dust explosions. The carbon and beryllium dust limits stem primarily from chemical reactivity concerns. The tungsten dust limit is determined by both chemical reactivity and activation hazards. Total mobilisable dust limits are 100 kg

for beryllium, 200 kg for carbon, and 100 kg for tungsten (tungsten dust the Assessment Value is set to 350 kg to account for uncertainties). Dusts on the divertor surface are limited by 6 kg for Be, C, and W each. The baseline dust size specification is given as a log normal distribution with count median diameter of 0.5 µm and geometric standard deviation of 2 (mass median diameter of 2 µm) and specific surface area of 4 g/m^2 [10, 12].

In case of air or steam ingress into VV under vacuum conditions up to 100 % of the accumulated dust is assumed to be mobilisable [12, 13]. To evaluate the dust explosion hazard in ITER and clarify the criteria on maximum permissible amounts of dusts in its vacuum vessel, a program was started at FZK to study the explosion characteristics of ITER-relevant dust. As a first step in this direction, the explosion indices of graphite dusts have been measured using a standard method of 20 l sphere [14]. Maximum overpressures, rates of pressure rise, and lower explosive concentration limits have been measured of graphite dusts as dependent on the dust characteristic particle size (4 to 45 µm), and explosibility of the dust has been evaluated. The presented work continues the measurements of the explosion indices of ITER-relevant dusts, tungsten dusts and graphite/tungsten dust mixtures.

2. TEST METHOD

Characteristics of dust explosion severity are maximum overpressure P_m generated during the explosion and maximum rate of pressure rise $(dP/dt)_m$. The first one indicates the level of pressure loads that may be generated, and the other one – how fast such a load can develop. Measurements of maximum explosion overpressure and rate of pressure rise are usually performed in closed-volume chambers. While the maximum overpressures of a dust cloud explosion in a closed volume depends on dust chemical composition and particle properties, initial pressure and temperature, and turbulence level in the cloud, the maximum rates of pressure rise depend also on the chamber volume V. It is assumed that applying 'cube-root law'

$$(dP/dt)_m V^{1/3} = K_{st}$$

gives a volume-invariant explosion index K_{st}, which is used to classify the dust explosibility.

One of standard test methods to measure the dust explosion indices uses a 20-l spherical combustion chamber [15-17]. A facility scheme is shown in Fig. 1. A dust sample is put into the dust storage container, which is then pressurized with dry compressed air. At the test start a valve connecting the sphere and the container opens for a short time, and the dust sample is injected inside the sphere with a blast of the compressed air and dispersed inside the sphere forming a cloud. The dispersion is facilitated with a rebound nozzle (shown in Fig. 1) or another dispersing element, e.g. dispersion ring. The container volume is 0.6 l, the pressure inside is 21 bar abs., so the amount of air there is quite comparable with that in the sphere at normal conditions. To provide 1 bar pressure inside the sphere after the injection is completed, the sphere is pre-evacuated to 0.4 bar abs. At the moment when the pressure inside the sphere (and the container) equals 1 bar, the air-dust cloud is ignited at the sphere center. The pressure evolution during the explosion is measured by a pressure transducer installed at the sphere wall. The maximum overpressure and rate of pressure rise are derived from the recorded pressure-time curve.

Typical pressure-time curve measured in 20-l-sphere is shown in Fig. 2 [18]. The pressure trace starts at 0.4 bar abs at the moment of dust outlet valve opening. The pressure starts to rise with a delay time t_d,

when the air from the dust container enters the combustion chamber and, after the time t_v elapses, reaches 1 bar. At this moment the igniters are activated and ignite the dust-air cloud. The further evolution of the pressure is determined by the explosion process. During the explosion the pressure rises progressively until the rate of pressure rise reaches its maximum $(dP/dt)_m$, and then continues to rise, with decreasing pressure rise rate, up to the maximum overpressure P_{ex}, when the combustion process is completed. The further decrease in pressure is due to cooling effect of the combustion products.

During the dust injection the air blast generates the turbulence in the dust-air cloud which level then decreases. As the turbulence level is known to affect strongly the intensity of dust cloud combustion, it is kept the same from test to test by specifying the time delay t_v between the start of air blast and the moment of ignition. In this procedure this parameter is set to 60 ms (in case of 1 m3 test chamber it is ten times longer). Note that it is so called 'Siwek' 20-l chamber that is described above and used in our tests. Another type of 20-l chamber is designed at Pittsburgh Research Laboratory and used to test dusts at the US Bureau of Mines [19, 20]. The latter features different design and, more important, has different air injection parameters – 16 l reserve tank at 9 bar issues an air blast during 0.3 s, and the chamber is pre-evacuated to 0.14 bar abs. Then, the ignition delay time is also different and equals 0.1 s. This results in appreciably lower turbulence level of the dust-air cloud giving lower values of measured K_{st} as compared to those measured in 1 m^3 vessel. Generally, the explosion indices measured in 1 m^3 test facility are considered as reference ones; however, it requires much greater dust quantities and labor efforts. The explosion indices measured in 'Siwek' 20-l apparatus are assumed to be the same as in 1 m^3 chamber [15]. However, this statement is criticized concerning the K_{st} values in Ref [21] where the measurement results of the turbulence level in a model 20-l sphere are compared with that exists in 1 m^3 vessel and the conclusion is made that K_{st} values cannot be directly applied to evaluate the rates of pressure rise in larger vessels. Nevertheless, testing dust explosion properties in 20-l apparatus are of wide use, and extensive data on a wide variety of industrial dusts are available. In this view the measured explosion indices reported in this work can be used to rank the ITER-relevant dusts as explosives among other industrial dusts.
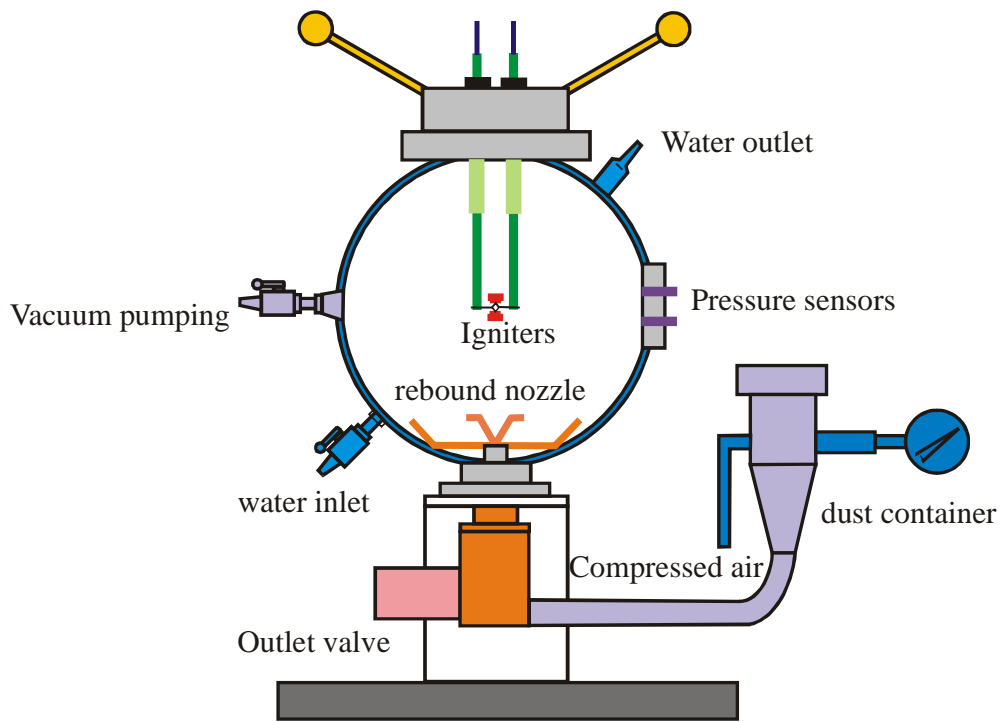
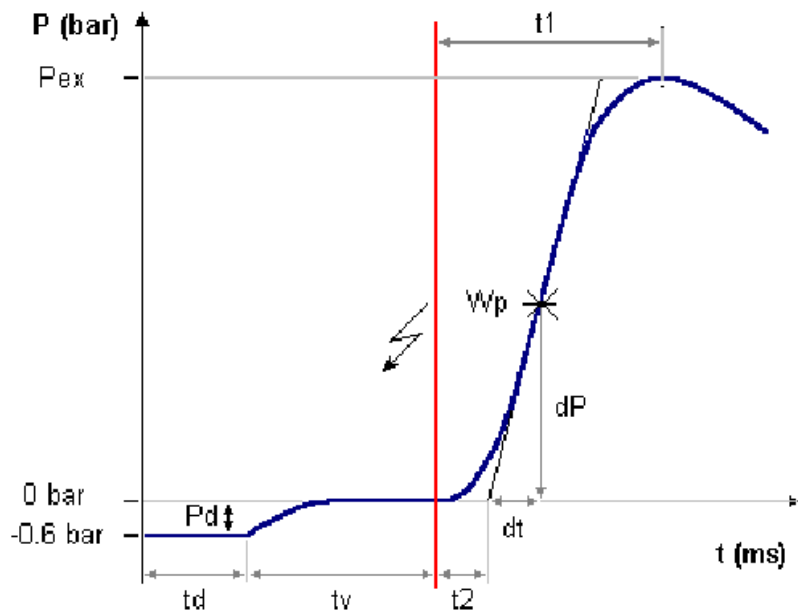Figure 1. Scheme of 20-l-sphere.

Figure 2. Typical pressure-time curve recorded during a dust cloud explosion in 20-l sphere.

3. TEST FACILITY

The test facility is shown schematically in Fig. 1. It consists of a spherical combustion chamber made of stainless steel, dust storage container connected with the chamber via a dust outlet valve, a pair of e-lectrodes holding chemical igniters at the sphere center, and two pressure transducers placed flush with the sphere wall. Design pressure of the combustion chamber is 30 bar. The chamber is surrounded by a jacket with flowing cooling water to keep the chamber wall temperature the same from test to test. The water cooling system is closed-loop; it consists of a heat-insulated water tank, a refrigerator cooling the water in the system to 20° C, and a water pump supplying the water from the tank to the chamber jacket at 1 l/min rate.

To pressurize the dust storage container, a dry compressed air from a compressed air bottle kept at 40-150 bar pressure is used. The same air is used to supply the dust outlet valve, which is operated pneu-matically and activated electrically. The pressure in the container (21 bar abs) is measured with a mano-meter installed at the container.

After a dust sample is placed into the storage container and pair of igniters is fixed to the electrodes, the container is pressurized to 21 bar abs of dry compressed air. Then the sphere is pre-evacuated prior to test to 0.4 bar abs that is controlled with an additional static pressure gauge installed at the vacuum pipe. A test starts with opening of the dust outlet valve, and a short blast of air disperses the dust sample via the rebound nozzle inside the sphere. After 90 ms the dust valve is closed, and 10 ms later the igniters are activated to ignite the dust cloud.

A control unit runs a test triggering the measurements, activating the dust outlet valve to open at test start and to close at 90 ms later, and activating the chemical igniters. The pressure evolution in the course of explosion is measured with two fast pressure transducers to ensure correct measurements and allow for self checking. The pressure transducers are piezoelectric PCB113A and piezoresistive Kulite KE-375 (500 and 160 kHz natural frequency, 1 and 1.5 % uncertainty, 0.368 and 0.112 V/bar sensitivity, respecti-vely). The piezoelectric pressure transducer is heat-sensitive, so its face is protected by silicon lubricate. The piezoresistive transducer is 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.
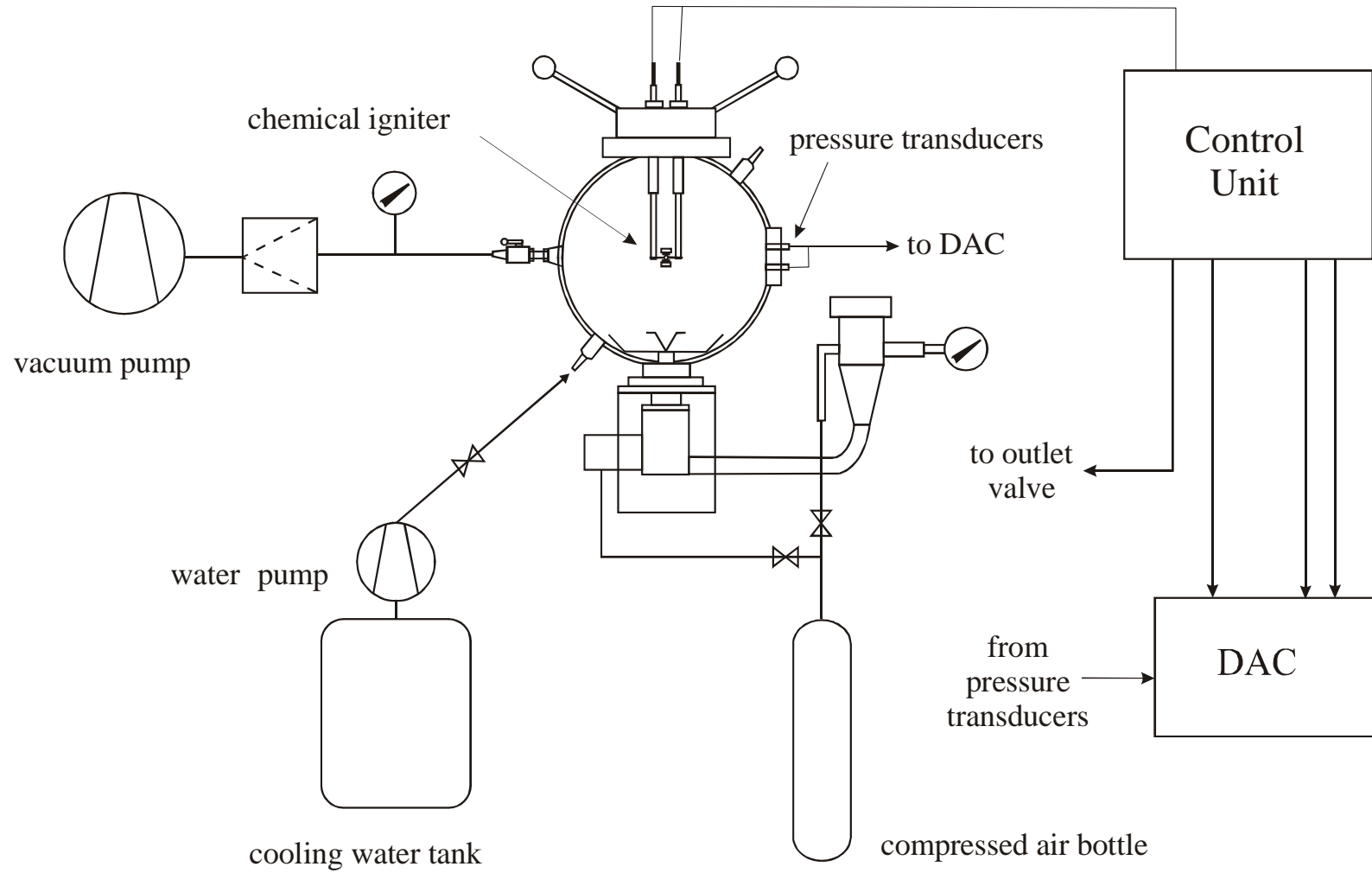
chemical igniter

pressure transducers

to DAC

Control Unit

vacuum pump

water pump

to outlet valve

from pressure transducers

DAC

cooling water tank

compressed air bottle

Figure 3. Scheme of DUSTEX facility

4. TESTING PROCEDURE

The measured explosion indices reported here are maximum overpressure, maximum rate of pressure rise, and lower explosion concentration limit. In addition, explosibility of the tested dusts has been evaluated. All the reported dust concentrations are the mass of a dust sample charged to dust storage container divided by the sphere volume (20 l). Some characteristics of the measurement procedures are described below following the recommendations in Ref. [18].

Test conditions. 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 is activated to open (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 (the 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.

Chemical igniters. The ignition sources used in the tests are electrically activated pyrotechnical igniters manufactured by Fr. Sobbe GmbH, Germany. The igniters are available of various ignition energies from 250 to 5000 kJ. The igniters are composed of 40 wt% zirconium, 30 wt% barium nitrate, and 30 wt% barium peroxide. When activated, the igniter releases a dense cloud of very hot particles and little gas [22]. In the measurements of the maximum explosion overpressures and rates of pressure rise, and of the lower explosive concentrations two igniters of 5 kJ release energy each are used in a test. In the tests to evaluate the dust explosibility, two igniters in a test are used; either of 1 kJ or 500 J release energy each.

Maximum overpressure and rate of pressure rise. Generally, these parameters are measured in three series; the exclusions are due to lack of tested dust. A series consists of a number of tests with step-wise increase in dust concentration. The maximum overpressure and maximum rate of pressure rise reported for each concentration are the averages over all the series. A test is repeated if the measured value of maximum overpressure deviates more than 10 % of the average value or the measured value of the maximum rate of pressure rise deviate more than 30 % of the average in the range from 0 to 185 bar/s, 20 % of the average in the range 185-370 bar/s, and 10 % of the average exceeding 370 bar/s.

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

$$P_m = 0.775 \cdot P_{ex}^{1.15}$$

where P_{ex} is maximum overpressure recorded for the test.

The second one is the additional pressure generated by the chemical igniters.. The correction of this effect is made as follows:

$$P_m = 5.5 \cdot (P_{ex} - P_{ci}) / (5.5 - P_{ci}) \text{ (bar)}$$

where P_{ci} is pressure generated by the chemical igniters themselves, i.e. in a blind test with no dust. It is stated in the manual to the 20-l-sphere [18] that 10 kJ igniters give 1.6 bar overpressure, and $P_{ci}=1.6\bullet IE/10,000$ (bar); IE is igniters release energy in joules. Our blind tests show that the Fr. Sobbe's igniters produce 1.1 bar overpressure; this measured value is used in the processing of our measurement results.

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 $(dP/dt)_m$ is derived at least 50 ms after ignition.

The maximum overpressure and rate of pressure rise measured for a dust over a wide range of concentrations C_{dust}, i.e. maxima of $P_m(C_{dust})$ and $dP/dt_m(C_{dust})$, are the explosion indices P_{max} and dP/dt_{max}.

Lower explosive concentration limit. A test series is initiated at a definitely non-explosive concentration and then continued with a systematic increase in dust concentration (50 g/m^3 for pure tungsten dust and 10 g/m^3 for graphite/tungsten dust mixtures) until ignition is observed. The ignition is assumed to occur if maximum overpressure (corrected) exceeds 0.4 bar (the recorded overpressure P_{ex} > 2 bar). Then the test is repeated with the dust concentration step lower, and the series is continued with step-wise reduced concentration in further tests until a concentration is reached at which no ignition of the dust/air mixture is observed in three successive tests.

The sphere, dust storage container, and dust outlet valve are cleaned thoroughly before each test. After cleaning a blind test (igniters only, no dust) is executed to burn out residues.

Dust explosibility. A dust is classified as not explosible if it cannot be induced to explode in a wide range of concentrations with 2 kJ chemical igniters [17]. Dust explosion is presumed to observe in a test if the corrected maximum overpressure exceeds 0.2 bar (the recorded overpressure P_{ex} > 0.5 bar).

5. CALIBRATION TEST

According to international standards, test equipment must be calibrated at intervals by comparison with a standard or a calibrated testing apparatus. This calibration also applies to the 20-l-apparatus and the 1-m^3-vessel for the determination of P_{max} and K_{max}.

Up to now there are neither internationally recognized reference samples nor reference equipment available for the determination of these explosion characteristics. Therefore the following calibration method has been proposed by Kühner AG, Switzerland (a producer of standard 20-l-apparatus) and carried out successfully: A dust has been selected, prepared and supplied to 53 test laboratories all over the world. The mean values of the explosion indices, measured by the participating laboratories, have been calculated as reference values. The participants were 46 laboratories using 20-l-apparatus and 3 laboratories using 1-m^3-vessels.

A test report has been published [23].This report presents the results of this calibration method and describes the evaluation procedures. For correct calibration the CaRo 03 test sample was milled, homogenized and shipped to the participants in an air tight package; so the sample was tested „as delivered". The test dust was **Niacin USP** (Nicotinic acid) with 10 % of the dust less than 6 μm, 50 % less than 22 μm, and 90 % less than 65 μm. The reference values for the test dust were P_{max} = 8.1±10% (7.3…8.9) bar and K_{max} = 232±10% (209…256) bar m/s.

The facility has been calibrated in the frame of the round-robin test CaRo03. The received test sample was tested as delivered according to the test procedure described above. The measured values are P_{max} = 8.2bar and K_{max} = 250 bar m/s. Like the other testing laboratories, the FZK's has been informed about the evaluation with a certificate (see next page).

Kühner

Certificate of Calibration

Kalibrier - Zertifikat

P_{max} | K_{max}

CaRo 03

Certificate No. Zertifikat Nr.	**PK03 - 47**	Enclosure Beilage	Final Report Schlussbericht	CaRo 03 CaRo 03

Customer Kunde	**Forschungszentrum Karlsruhe GmbH**
Object Gegenstand	**20-l-Apparatur**
Manufacturer Hersteller	-

	P_{max} [bar]	K_{max} [bar·m/s]
Reference values Referenzwerte	$8.1 \pm 10\%$ (7.3 ... 8.9)	$232 \pm 10\%$ (209 ... 256)
Measured values Messwerte	8.2	250

Date of calibration Kalibrierdatum	**26. Dec. 2003**
Remarks Bemerkungen	- -

Birsfelden, Januar 2004

Adolf Kühner AG
Christoph Cesana

P052_136

16.01.04

6. TESTED DUSTS

Three pure tungsten dusts (99.9 %) are tested: 12 µm, 5 µm, and 0.6-0.9 µm characteristic particle size. TEM images of the finest and the coarsest of the tested tungsten dusts are presented in Fig. 4. The dusts are tested as delivered with no pre-test treatment. To form graphite-tungsten dust mixtures, a fine graphite dust of 4 µm characteristic particle size is used; its TEM image is shown in Fig. 5.
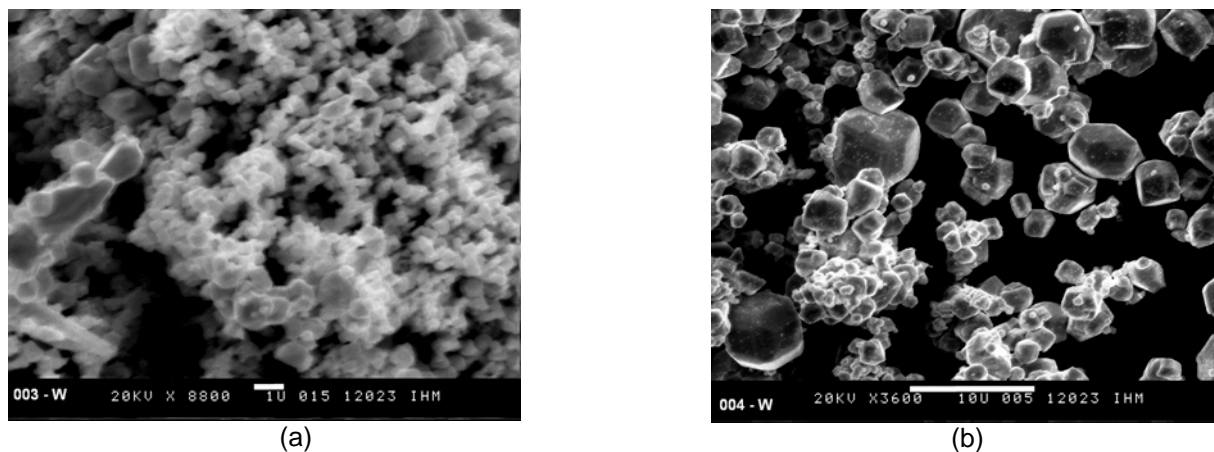

(a)   (b)

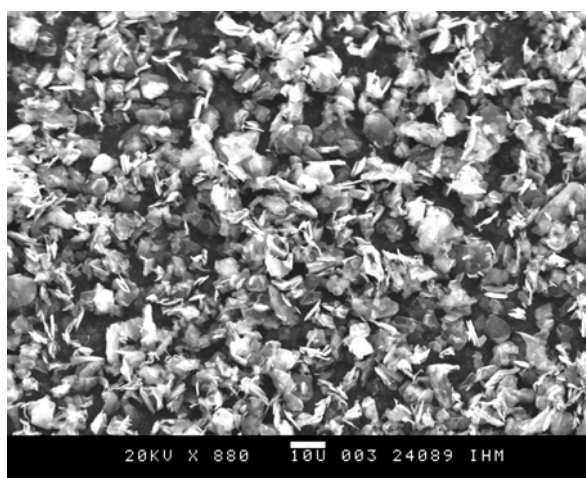Figure 4. TEM photos of the tested tungsten dusts: (a) 0.6-0.9 µm, (b) 12 µm.

Figure 5. TEM photo of 4 µm graphite dust.

The graphite dust is analyzed on hydrogen content; its value is below the detection limit of 0.005%. The graphite dust is dried just before testing at 190 °C during 15 min in SARTORIUS moisture analyzer MA 45; the moisture content was less than 1%. A dust sample to test is weighed with SARTORIUS CP2202S balances (0.01 g accuracy). All the mass concentrations reported here are the sample mass divided by the sphere volume (20 l).

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7. STANDARD TEST RESULTS

The results of the tests made in 20-l-apparatus under standard conditions are summarized in Table 1. Here the test names contain the symbol for the constituent dust (W and/or C) and its characteristic particle size; the values in the column 'Dust size' denote the characteristic particle size (0.6-0.9 μm tungsten dust is indicated as 1 μm-sized); mixture composition is either pure tungsten (pure W) or the tungsten-to-graphite molar ratio of the dust; dust concentrations, which are the mass of the dust sample divided by the sphere volume, are generally averaged corresponding to its accuracy; the values in brackets in columns P_m (maximum overpressure) and dP/dt_m (maximum rate of pressure rise) are the corrections to 1 bar initial pressure of the measured values if they differ (see explanation below). The values "< 0.1" in the P_m column denotes the cases where the measured pressure-time curve do not differ from that measured in a blank test, i.e. in the test with igniters only and without dust. The mark "n/d" in the column dP/dt_m denote a case where the measured maximum rate of pressure rise is due to the igniters effect, and the effect of the dust cannot be determined.

Table 1. Results of the tests with tungsten dusts and graphite/tungsten dust mixtures.

Test name	Dust size	Mixture composition W/C mol	Cdust	Ignition energy	Pm	dP/dtm
	μm	-	g/m³	kJ	Bar	bar/s
W5_01	5(W)	pure W	350	10	< 0.1	n/d
W5_02	5(W)	pure W	400	10	< 0.1	n/d
W5_03	5(W)	pure W	450	10	< 0.1	n/d
W5_04	5(W)	pure W	500	10	< 0.1	n/d
W5_05	5(W)	pure W	750	10	< 0.1	n/d
W5_06	5(W)	pure W	1000	10	< 0.1	n/d
W5_07	5(W)	pure W	1500	10	0.15	n/d
W5_08	5(W)	pure W	2000	10	< 0.1	n/d
W5_09	5(W)	pure W	4000	10	< 0.1	n/d
W5_10	5(W)	pure W	7500	10	< 0.1	n/d
W12_01	12(W)	Pure W	250	10	< 0.1	n/d
W12_02	12(W)	Pure W	500	10	0.1	n/d
W12_03	12(W)	Pure W	1000	10	< 0.1	n/d
W12_04	12(W)	Pure W	1500	10	0.1	n/d
W12_05	12(W)	Pure W	3000	10	< 0.1	n/d
W12_06	12(W)	Pure W	6000	10	< 0.1	n/d
W1_16	1(W)	Pure W	350	10	0.2	n/d
W1_17, 19, 20, 21	1(W)	Pure W	400	10	0.2	17
W1_18	1(W)	Pure W	450	10	0.3	27
W1_01, 02	1(W)	Pure W	500	10	0.6	41
W1_03	1(W)	Pure W	1000	10	3.3	72
W1_05	1(W)	Pure W	1500	10	3.6	67
W1_06	1(W)	Pure W	2000	10	3.9	113
W1_11	1(W)	Pure W	3000	10	4.2(4.7)	134(150)
W1_12,15	1(W)	Pure W	5000	10	4.7(5.7)	259(307)
W1_13, 14	1(W)	Pure W	7500	10	4.5(5.2)	237(277)
C4W1_12, 14	1(W)+4(C)	3/1	3300	10	6(6.5)	260(308)
C4W1_11, 15	1(W)+4(C)	1/1	2300	10	6.7(6.8)	360(368)
C4W1_09, 16	1(W)+4(C)	1/4	1100	10	6.7	252
C4W1_13, 08	1(W)+4(C)	1/30	410	10	6.8	245
C4W1_20	1(W)+4(C)	1/1	270	10	0.1	n/d
C4W1_21, 23, 24	1(W)+4(C)	1/1	320	10	0.2	n/d
C4W1_22	1(W)+4(C)	1/1	370	10	0.65	n/d
C4W1_19	1(W)+4(C)	1/1	420	10	2.1	34
C4W1_27	1(W)+4(C)	1/4	170	10	0.1	n/d
C4W1_28, 30, 31	1(W)+4(C)	1/4	190	10	0.3	n/d
C4W1_29	1(W)+4(C)	1/4	220	10	0.6	n/d
C4W1_26	1(W)+4(C)	1/4	250	10	0.8	n/d
C4W1_32, 33	1(W)+4(C)	1/1	2300	2	5.9	162
C4W1_34, 35	1(W)+4(C)	1/1	2300	1	0.19	n/d

Maximum overpressures and rates of pressure rise

Tungsten dusts. The dusts are tested as described above. The coarser tungsten dusts, of 12 μm and 5 μm characteristic particle size, do not appear to be explosible in the concentration ranges from 250 to 6000 g/m^3 (12 μm dust) and 350 to 7500 g/m^3 (5 μm dust). The explosion overpressure measured do not exceed 0.5 bar overpressure in the tested concentration range. It should be noted that if the mass of dust sample charged into dust container is about 100 g and larger (dust concentrations higher than 5000 g/m^3), an appreciable amount of injecting air remains in the container while almost all the dust is still delivered into the sphere. As a result, the pressure P_i in the sphere just after the dust valve closing, which must equal to 1 bar, appears to be lower (down to 0.9 bar depending on dust sample mass). First, it means that the oxygen amount is lower than normally that impedes ignition. It can also cause a lower turbulence level. Then, tungsten is almost 10 times denser than graphite, and the large particles of tungsten dust are difficult to disperse. This is deepened by lower velocities of the dispersing air flow at lower pressure difference, which might make the dispersion process inefficient. However, increase in the dispersing air pressure could make the test conditions beyond the standards. It should be noted that the tests with tungsten appeared to be rather destructive. The moving parts of the dust outlet valve were damaged after several shots with high-mass samples and have must be repaired. Finally, they were replaced by new ones to make the calibration tests.

The finer 1 μm tungsten dust is tested in the range of dust concentrations from 250 g/m^3 to 7500 g/m^3. The same effect of lower initial pressure at ignition moment is observed. However, the finer dust can be ignited in a wide range of concentrations even at lower initial pressures P_i. It is known that P_m and dP/dt_m are linearly proportional to initial pressure [18], and a software is available to take into account the influence of the initial pressure on maximum overpressure and rate of pressure rise [26]. The measurement results of the maximum overpressure and maximum rate of pressure rise and their corrected values are plotted versus dust concentration in Figs. 6 and 7. Both the dependences of P_m and dP/dt_m on dust concentration are typical for the test method: the overpressure rises with concentration from the lower explosive concentration, reaches its maximum of 4.7 bar, and then decreases slightly. The rate of pressure rise also increases with concentration and reach the maximum of 260 bar/s at the same concentration (5000 g/m^3) and then decreases. Note that the stoichiometric concentration for the reaction of tungsten oxidation ranges from 1250 g/m3 to 1730 g/m3 depending on the tungsten oxidation rate. It is characteristic for dust combustion that the maximum overpressure is observed at the dust concentrations several times higher than the stoichiometric one [19, 20, 24, 25]. An incomplete dispersion can contribute to this effect not so much for the amount of dust retaining in the dust container is negligible as compared to the dust sample mass. The main contribution is due to incomplete dust combustion. As a dust particle does not burn out during the characteristic combustion time, the dust at stoichiometric concentration burning in air does not consume all the oxygen. Depending on the dust-fuel consumption rate the oxygen is consumed at the "optimal" concentrations which are up to 5 times higher than the stoichiometric one [25]. With further rise of the dust concentration the excessive fuel acts as a heat sink causing some increase in the overpressure and rate of pressure rise.
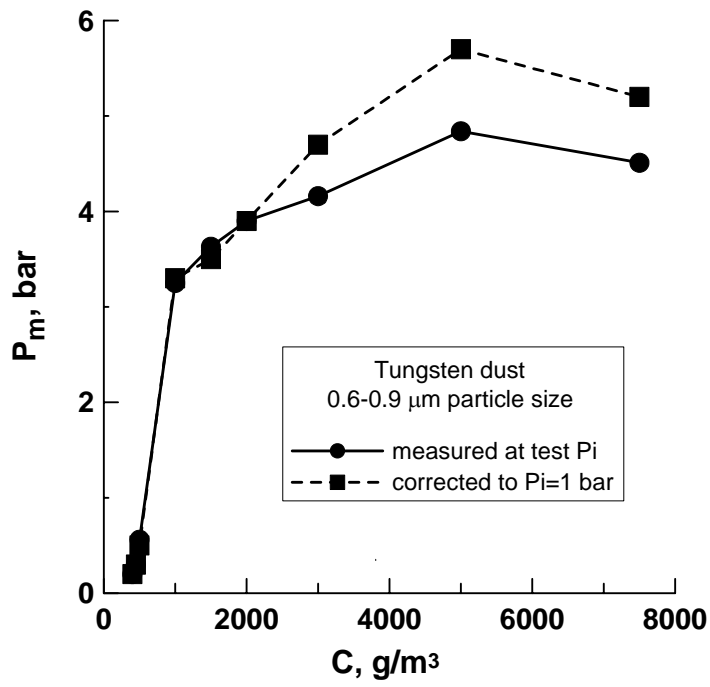
Figure 6. Maximum overpressures measured for 1 µm tungsten dust/air mixture versus dust concentration. Ignition energy is 10 kJ.
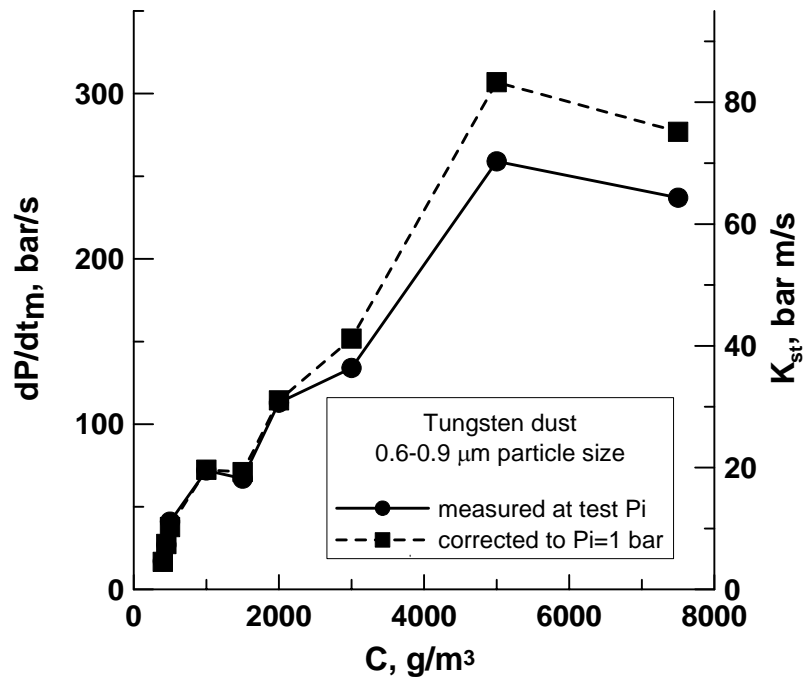

Figure 7. Maximum rates of pressure rise measured for 1 µm tungsten dust/air mixture versus dust concentration. Ignition energy is 10 kJ.

Graphite/tungsten dust mixtures. The tests with C/W dust mixtures are aimed at obtaining the conservative evaluation of the explosion indices possible with the available graphite and tungsten dusts. With this purpose the finest graphite (4 μm) and tungsten (1 μm) are used to form the test mixtures. Four graphite/tungsten mixtures are tested: of α=1/30, 1/4, 1/1, and 3/1 W/C molar ratio. The mass composition M_W/M_C of these mixtures relates to the molar one as follows:

$$M_W/M_C = \alpha\ \mu_W/\mu_C,$$

where μ_W=184 g/mol and μ_C=12 g/mol are molar weights of tungsten and carbon, respectively. Each of the mixtures is tested at one dust-in-air mass concentration only. The concentrations are chosen so that to evaluate the highest explosion overpressures and rates of pressure rise for the mixtures (P_{max} and dP/dt_{max}). These concentrations are assumed to be the minimal ones at which the oxygen is fully consumed during the dust combustion, as well as in the case of the pure dusts. Then suppose that, if mixed, the graphite and tungsten constituents burn independently in air. Consider for simplicity the combustion process of a graphite/tungsten mixture in 1 m³ volume to refer to the dust masses rather than concentrations. So if the graphite fraction of mass $M_C < M_C^{opt}$ (the minimal graphite dust mass/concentration at which the oxygen is fully consumed) is burnt there, it consumes M_C/M_C^{opt} part of the oxygen in the volume. Similarly, the burnt tungsten fraction of mass $M_W < M_W^{opt}$ consumes M_W/M_W^{opt} part of the oxygen. If M_W is the minimal mass of the tungsten fraction necessary to burn out the part of the oxygen amount $1 - M_C/M_C^{opt}$ remaining after graphite has been burnt, one has:

$$M_W/M_W^{opt} = 1 - M_C/M_C^{opt},$$

that gives for the constituents of the "optimal" sample of a graphite/tungsten dust mixture of molar ratio $W/C = \alpha$ the masses:

$$M_C = M_C^{opt}\ M_W^{opt}/(\ M_C^{opt}\ \alpha\ \mu_W/\ \mu_C + M_W^{opt})$$

$$M_W = \alpha\ M_C^{opt}\ M_W^{opt}/(\ \alpha\ M_C^{opt} + M_W^{opt}\ \mu_C/\ \mu_W)$$

Optimal concentrations of dust in air are usually not equal to the stoichiometric ones for the reaction of the dust substance oxidation; e.g. for coal dusts they are two to three times higher [19, 20, 24,25]. Assuming the effective "stoichiometric" coefficient to be 2.5 for graphite and tungsten dust oxidation reactions:

$$2.5\ C + O_2 \Rightarrow \text{products without free oxygen}$$

$$2.5\ W + O_2 \Rightarrow \text{products without free oxygen}$$

gives M_C= 280 g and M_W= 4300 g, which are close to those observed in our tests (250 g and 5000 g, respectively).

Using these formulas with the correction for 20 l volume, i.e. having values 50 times less, the following set of graphite/tungsten mixture samples are prepared for testing (maximum overpressure and rate of pressure rise):

Table 2. Graphite/tungsten dust mixture compositions.

α, W/C (mol)	M_C, g	M_W, g	M_C+M_W, g	C_{opt}, g/m^3
1/30	5.44	2.78	8.22	410
1/4	4.5	17.25	21.75	1100
1/1	2.81	43.13	45.94	2300
3/1	1.41	64.69	66.10	3300

Here **C_{opt}** is the dust mass concentration (the sample mass divided by 20 l). Each concentration is tested twice; the averaged results of maximum overpressures and rates of pressure rise measurements and their corrections with respect to initial pressure at ignition are presented in Figs. 8 and 9. The corresponding results for pure 4-µm graphite and 1-µm tungsten dusts are also given in the figures. X-line represents the molar percentage of the tungsten content in the mixture. The maximum overpressures decrease monotonically with carbon content. However, the dependence is not linear as it might be expected in case the graphite and tungsten fractions burn independently. In this view the dependence of maximum rates of pressure rise on tungsten content is remarkable: the graphite/tungsten dust mixture with W/C=1 (mol) burns much faster than pure graphite or tungsten dusts constituting the mixture: 360 bar/s for the "optimum" mixture against 250 bar/s for pure graphite and 260 bar/s for pure tungsten. Whether the reason of chemical matter or not, remains to be seen.
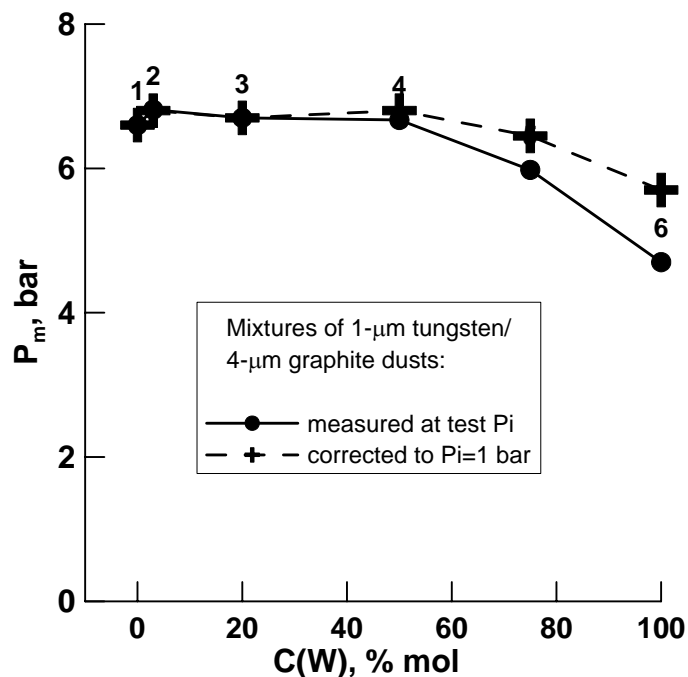

Figure 8. Maximum overpressure versus tungsten content in graphite/tungsten dust mixture. Ignition energy is 10 kJ. Mixtures: **1** – pure graphite dust; **2** – W/C = 1/30; **3** – W/C = 1/4; **4** – W/C = 1/1; **5** – W/C = 3/1; **6** – pure tungsten dust.
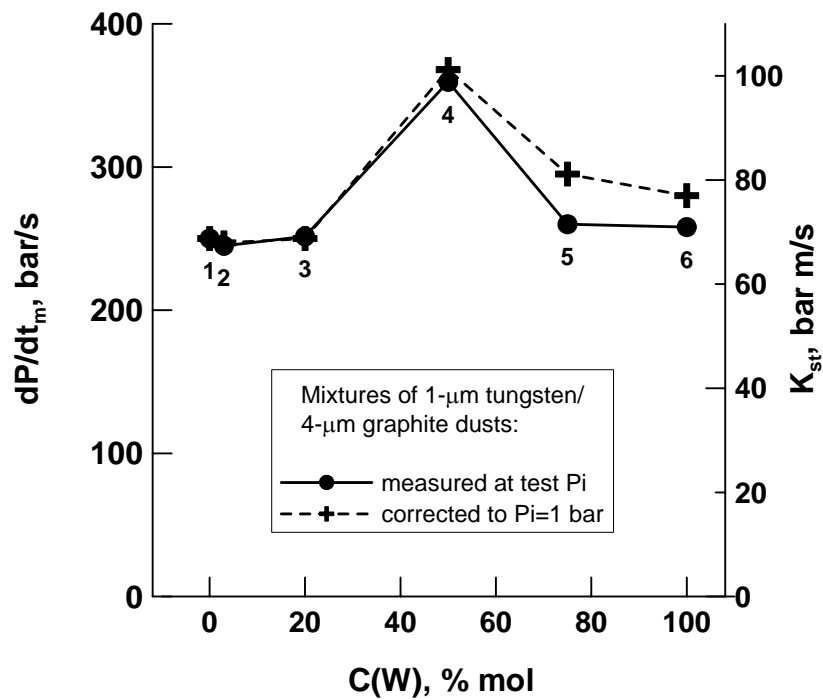
Figure 9. Maximum rate of pressure rise versus tungsten content in graphite/tungsten dust mixture. Igniti-
on energy is 10 kJ. Mixtures: **1** – pure graphite dust; **2** – W/C = 1/30;
3 – W/C = 1/4; **4** – W/C = 1/1; **5** – W/C = 3/1; **6** – pure tungsten dust.

Lower explosion limits

The lower explosion concentration limits are measured for the 1 μm tungsten dust and two graphi-
te/tungsten dust mixtures with W/C=1/1 and 1/4. The results are presented in Table 3, where the lower
explosion limit of 4 μm graphite dust is added. Here M_W/M_C mass ratio of tungsten and graphite consti-
tuents; C_{LEL} – lower explosion concentration limit – is presented in two forms: as mass concentration and
molar concentration. Figure 10 shows the dependence of lower explosion concentration (molar) on the
tungsten molar fraction of the graphite/tungsten mixture; the data point labels in the plot correspond the
mixture number in the table. The lower explosive concentration of graphite/tungsten dust mixture moles
decreases with increasing tungsten fraction; being expressed in the mass form, it increases with tungsten
content due to high density of tungsten

Table 3. Lower explosion concentration limits of graphite/tungsten dust mixtures.

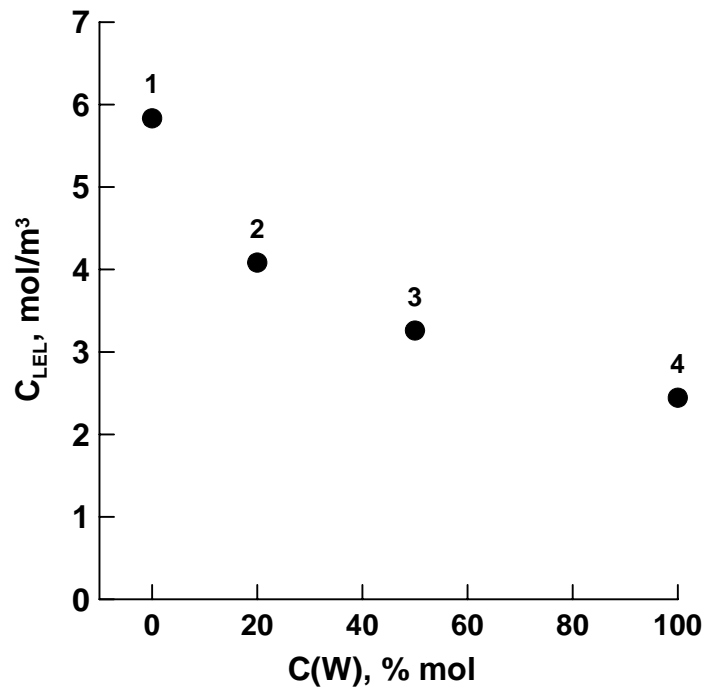| Mixture No.	α, W/C (mol)	M_W/M_C	C_{LEL}, g/m^3	C_{LEL}, mol/m^3
1	C (pure)	0	70	5.8
2	W/C=1/4	3.83	190	4.1
3	W/C=1/1	15.3	320	3.3
4	W (pure)	-	450	2.4

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Figure 10. Lower explosion concentration (molar) of the graphite/tungsten dust mixtures versus the tungsten molar fraction. Ignition energy is 10 kJ.

Explosibility

The explosibility of the 'most dangerous' graphite/tungsten dust mixture, with molar ratio W/C=1, is tested at optimum concentration to make a conservative evaluation. The tests are performed using 10, 2, and 1 kJ release energy chemical igniters; the measured maximum overpressures and rates of pressure rise are presented in Fig. 11 and 12, respectively. To compare the explosibility of the pure graphite dust and its mixture with tungsten, the figures also contain the corresponded values measured for 4 µm graphite dust [14]. Like for the graphite dust, the graphite/tungsten dust mixture is also explosible at 2 kJ ignition energy and is not induced to explode with 1 kJ igniters. However, the maximum overpressure of the graphite/tungsten dust mixture measured at 1 kJ ignition energy is 0.19 bar, i.e. very close to the "explosive - non-explosive overpressure" definition of 0.2 bar, while for the graphite mixture it is 0.1 bar. This difference can be seen more clearly from the data measured at 2 kJ ignition energy: the graphite/tungsten dust mixture generates 5.9 bar maximum overpressure and 160 bar/s maximum rate of pressure rise (6.8 bar and 370 bar/s at 10 kJ), while the graphite dust explosion tests give 5.1 bar and 40 bar/s at 2 kJ and 6.6 bar and 250 bar/s at 10 kJ.
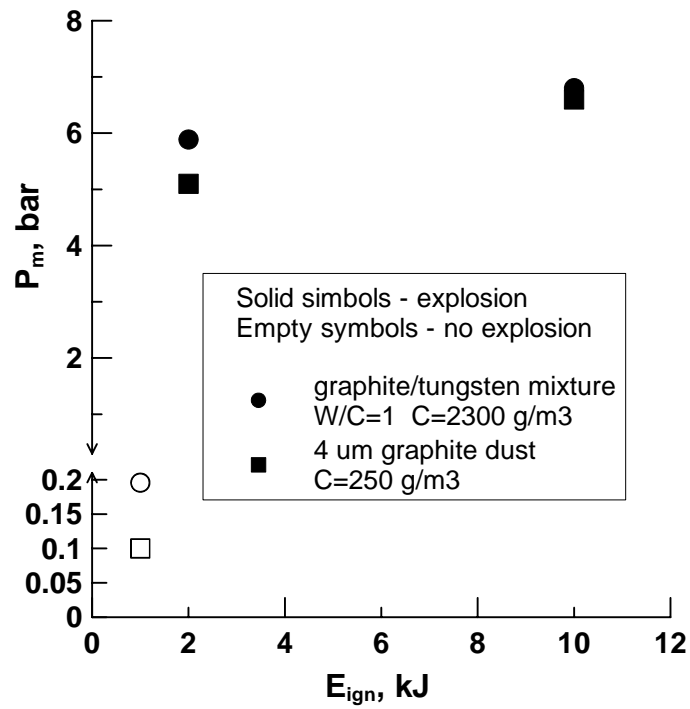
Figure 11. Maximum overpressure of 4 μm graphite dust (quadrants) and the graphite/tungsten dust mixture (circles) versus ignition energy. The tests are defined to be explosible if maximum overpressure exceeds 0.2 bar.
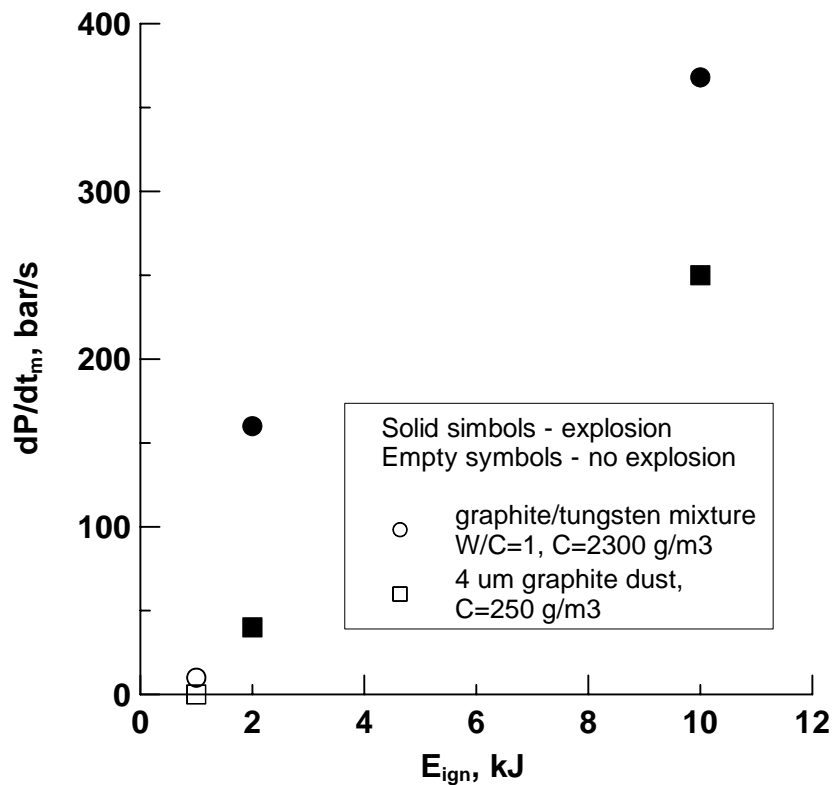

Figure 12. Maximum rate of pressure rise of 4 μm graphite dust (quadrants) and the graphite/tungsten dust mixture (circles) versus ignition energy.

8.   BURNING VELOCITY MEASUREMENTS

The results presented here and reported in Ref. [14] show that the dusts possibly accumulated inside the ITER vacuum vessel are capable to explode generating an appreciable pressure loads. Actually, the o-verpressures observed in 20-l-apparatus are close to P_{aicc} – pressure of adiabatic isochoric complete combustion. However, depending on dust cloud geometry, boundary conditions, dust density space dist-ribution, etc., a development of the combustion wave under certain conditions could reach even a detona-tion regime [27]. To model all the possible LOCA or LOVA scenarios in ITER involving dust mobilization and following combustion in actual-scale experiments seems hardly ever realistic; so a reasonable alter-native is a computer simulation. A computer code capable to model a dust explosion "from first principles" including gas/dust flow calculations, detailed combustion chemistry description, accounting for heat los-ses, etc, seems impossible even at the level of the required computer resources. A successful approach is known in simulating gaseous combustion basing on detailed gasdynamic modeling and a semi-phenomenological combustion model (COM3D, DET3D, and FLAME3D for hydrogen explosion simulati-on).

A similar code looks attractive to address the dust hazard problem in ITER. An approach has been cho-sen to modify FLAME3D code with this purpose. FLAME3D is based on full 3D gasdynamic solver and effective burning rate model. Its gasdynamic part has been extensively validated; good performance has been shown for hydrogen explosions. Matching the effective burning rate model of FLAME3D to dust fuels requires the corresponding experimental data on ITER-relevant dusts. To measure their effective burning rates or burning velocities depending on the dust particle size and dust concentration, a dust Bunsen burner is attempted to use. The idea is to form a spatially-homogeneous dust/air laminar flow flowing in a cylinder with a velocity close to the laminar burning velocity in the dust/air mixture. The flow is ignited by a cylindrical flame holder heated at the dust ignition temperature. The flame propagating from the flame holder to the flow axis forms a cone, which top angle gives the value of the burning velocity if the flow axial velocity is known.

A dust burner is fabricated (see photos in Fig. 13) consisting of a cylinder *1* (20 mm inner diameter, 200 mm length), a dust bunker *2* with auger-type dust feeder *3* supplying a dust at calibrated rate from the bunker to the cylinder bottom. The dust/air flow is formed with an air jet from an air-feeding pipe *4* direc-ted downward inside the cylinder near its bottom. The bottom *5* made of Teflon covered with copper foil is rotated at about10 rpm to spread the oncoming dust over the air-blown area. A short radial baffle *6* over-lapping half-radius of the cylinder deflects the air jet to homogenize the dust air flow. The air is fed into the cylinder via a flow-meter. With the air flow rate and the cylinder cross-section given one gets the velo-city of the air flow moving upward in the cylinder. A flame holder *7* is fixed at the entrance end of the cy-linder 2-3 mm apart. The flame holder is made of a ferronickel alloy ribbon 2 mm wide and 0.5 mm thick. The ribbon is heated electrically.

A series of tests are made with graphite dusts with characteristic particle size from 4 to 50 µm forming air flow inside the cylinder with axial velocities ranging from 10 to 20 cm/s and dust density from 100 to 350 g/m^3. Up to the flame holder temperatures of about 800 °C the air flow remains stable, but the dust is not ignited. At higher temperatures the convection due to the flame holder heat destroys the air/dust flow

making it unstable and impossible to get a measurable flame cone, and even in this case the dust flow is not ignited. So the first attempts are not successful. Nevertheless, they will be continued with elevated oxygen content in the feeding air or with hydrogen added to the flow to facilitate ignition. The dependences of the burning velocities, if the burning will obtain, on the oxygen (hydrogen) content in the flow are planned to measure with the aim to extrapolate the dependences to normal oxygen or zero hydrogen content.
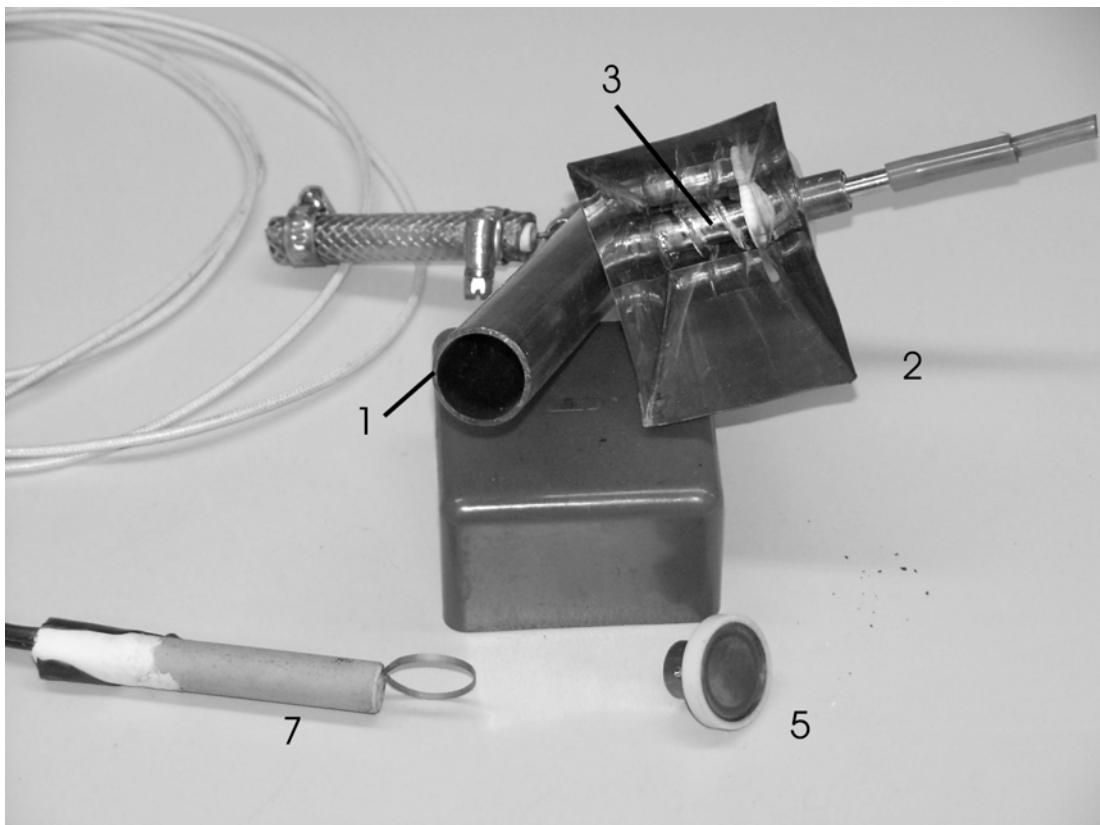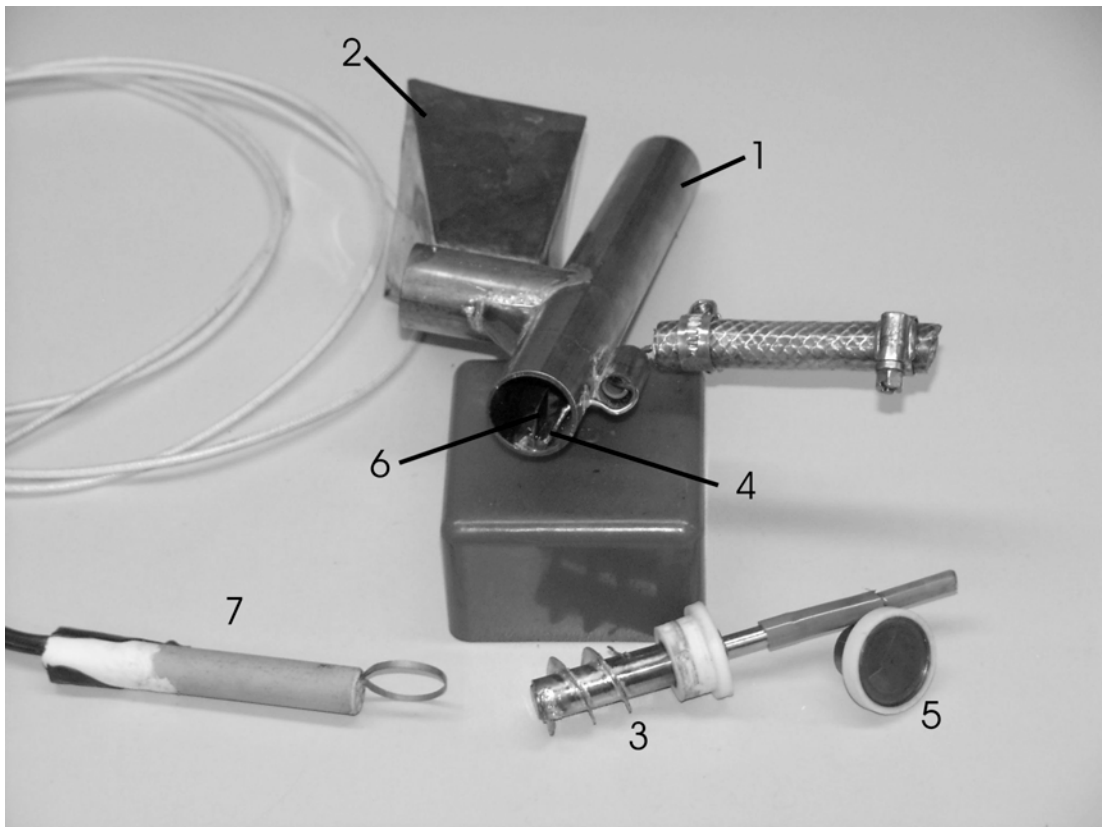
Figure 13. Dust Bunsen burner. 1 – flow former; 2 – dust bunker; 3 – dust feeding auger;
4 – air feeding pipe; 5 – rotational bottom; 6 – dispersing baffle; 7 – flame holder.

9. SUMMARY

Using a standard method of 20 l sphere, explosion indices of tungsten dusts of 0.6-0.9 µm, 5 µm, and 12 µm characteristic particle size are tested. The coarser dusts do not appear to explode under standard conditions in the tested concentration ranges (250 to 6000 g/m^3 for 12- µm dust and 350 to 7500 g/m^3 for 5- µm dust). The finest tested tungsten dust is able to explode in the concentration range from 450 g/m^3 (lower explosion limit) to 7500 g/m^3. The maximum overpressure and rate of pressure rise are 5.7 bar and 300 bar/s, respectively. Graphite/tungsten dust mixtures differing in the mixture composition are tested to measure their maximum overpressure, maximum rate of pressure rise, and lower explosive concentrations. The tested mixtures are composed of 4-µm graphite and 1-µm tungsten dust having the tungsten-to-carbon molar ratios W/C=1/30; 1/4; 1/1; and 3/1. With increasing tungsten content the maximum overpressure decreases from 6.6 bar (pure graphite) to at least 4.7 bar (pure tungsten), while the maximum rate of pressure rise as a function of tungsten content has a pronounced peak of 360 bar/s at W/C=1/1. The maximum rate of pressure rise measured for this mixture is 1.5 times higher than for pure graphite and tungsten dust alone, i.e. graphite/tungsten dust mixtures are able to explode faster than its constituents. This observation is confirmed by explosibility evaluation tests. Both the graphite dust and the graphite/tungsten dust mixture can be induced to explode by 2 kJ igniters; however, the maximum rate of pressure rise measured for the mixture in this case is appreciably higher than that for the pure graphite dust. The tested graphite/tungsten dust mixture does not explode at 1 kJ ignition energy, but the measured maximum overpressure is only slightly lower than the evaluation pressure threshold of 0.2 bar.

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