Appendix B

Experimental equipment and procedures

B.1 Spark generator details

The detailed schematic layout for generation of high-voltage pulses used in the spark generator described in [29] is shown in Figure B-1. A triggering pulse of 5 V is used to discharge the primary capacitor through a high-voltage coil. This triggering is done with a computer using a digital I/O plug-in board (*National Instruments PCI-6503*).

Figure B-2 shows the spark generator and the explosion chamber, in addition to pictures of the probes used for spark measurement and the surface-mounted resistors. The standard ground loops ("pigtails") of the scope probes are not used, ensuring less influence of noise from the spark discharge. The reader is referred to [29] for additional details on the spark generator and the spark voltage and current measurement system.

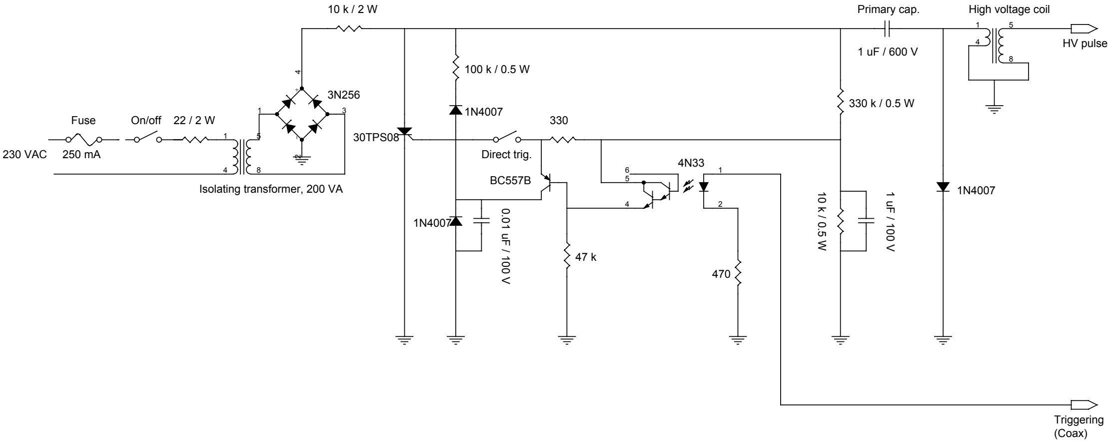
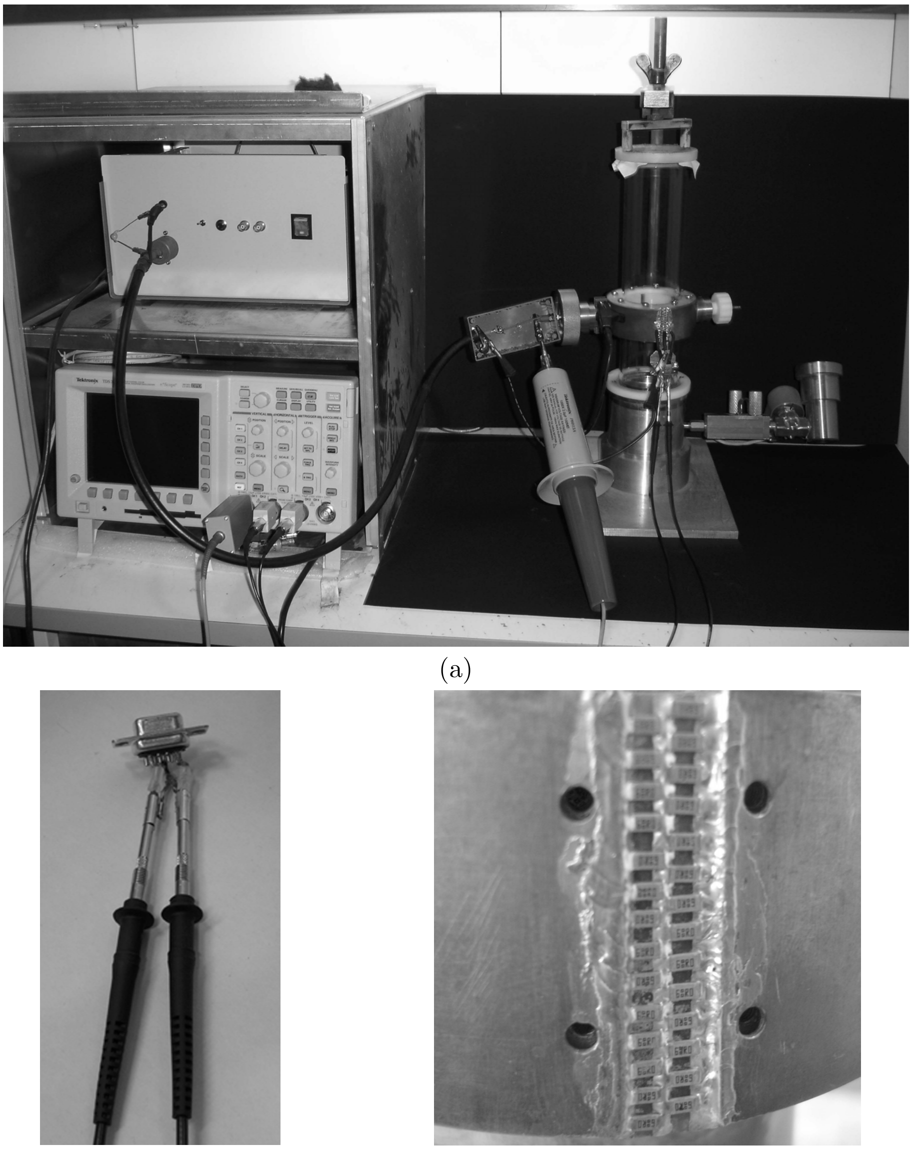


Figure B-1. Schematic layout of the system for generation of high-voltage pulses.



(a)

(b)

(c)

Figure B-2. (a) The spark generator, the measurement system and dust explosion chamber. (b) The current measurement probes, where the ground loops are reduced to a minimum. (c) The surface-mounted symmetry resistors, integrated in the shield around the explosion chamber.

B.2 Spark energy calculation

The oscilloscope traces from the high-voltage probe and the two conventional probes used for current measurements are imported into a spreadsheet, and the spark energy is calculated according to Equation (3.6).

However, when calculating spark power and integrated energy, some intermediate calculations are also made:

1. The signal from the high-voltage probe is delayed 9.0 ns compared to the signal from the scope probes [29]. This is taken into account when calculating circuit power by ensuring synchronisation of current and voltage.
2. The voltages from the scope probes are interpreted as currents through the measurement resistors with an effective resistance of 2.5 Ω each (due to the 10 Ω symmetry resistor in parallel).
3. The balanced (i.e. mean value when taking polarity into account) current signal is calculated. Figure 3-9 illustrates the effect of using balanced current measurements with regards to reduction of noise.
4. Adjustment for any DC offset of the resulting current signal is made.
5. Spark power is calculated.
6. Resistor power (total resistance of 5 Ω) losses are subtracted.
7. The time scale is defined by setting the time $t = 0$ at first peak of the damped oscillating current trace. Energy integration is made between $t = -20$ and 100 ns.
8. Both cumulative circuit energy and net spark energy are calculated.

A fundamental condition for this approach is that the cumulative spark energy stabilises after some time, typically about 100 ns after breakdown. If the charging resistor were too small, the spark energy would continue to increase beyond the time frame of the oscilloscope measurement, and the measurement of spark voltage and current would be of little value.

B.3 Experimental procedures

The experimental procedures are mainly based on the documentation for Kühner's MIKE apparatus [55], with some adjustments. The following properties are similar to MIKE:

- Dispersion pressure 7 bar(g).
- 50 cm^3 pressurised dispersion chamber.
- Identical explosion chamber volume (1.2 litre), shape and bottom (cup).

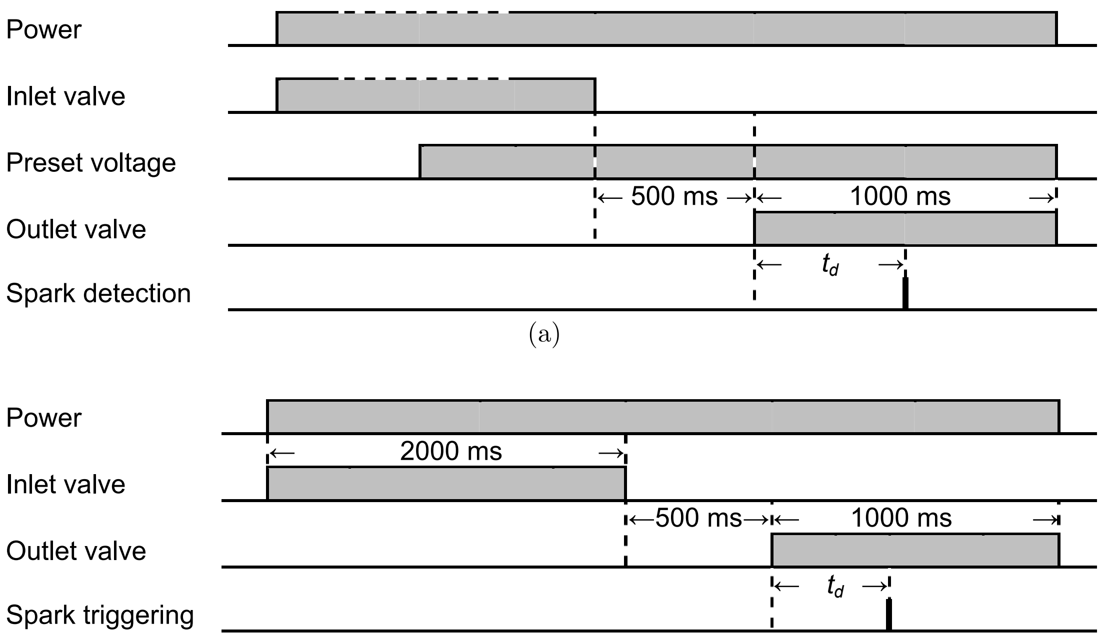
- Electrode diameter (2 mm) and electrode material (tungsten).

The following properties of the explosion testing system are different from MIKE:

- The method of generating sparks, both when triggering the sparks by dust dispersion and when using electronic synchronisation between dust dispersion and sparkover, is different. Schematic circuit drawings are given in Figure 3-7 and Figure 3-8.
- The dispersion nozzle is modified (larger orifices than used in the MIKE apparatus) in order to allow the dust to pass through it without clogging.
- Most dusts were dispersed by forcing the dust sample through the nozzle (exception: the metal dusts Ti, TiH_2, ZrH_2 and Al flakes which were, due to clogging, placed in the explosion chamber cup in the same way as when using the MIKE apparatus).
- When triggering the sparks by dust dispersion, rounded off electrodes were used to avoid corona leakage.

Figure B-3 shows the sequence for ignition testing using (a) the dust cloud itself to trigger the sparkover and (b) electronic synchronisation between dust dispersion and sparkover. The term ‘inlet valve’ is used for the valve upstream of the 50 cm^3 dispersion chamber, and the ‘outlet valve’ is downstream. The delay between outlet valve opening and spark discharge is labelled t_d. The procedure is programmed in LabView, and a digital I/O PCI plug-in board (*National Instruments PCI-6503*) is used to control power onset, valves and spark triggering.

The schematics of the gas mixing equipment used when testing the MIE of propane is shown in Figure B-4. The propane concentration inside the explosion chamber was considered known when the concentration of the outflow was equal to the concentration of the inflow. A propane analyser (*Servomex 1400*) was employed to measure the concentration.



(a)

(b)

Figure B-3. (a) Sequence for ignition testing when using the dust cloud itself to trigger spark discharge. The stochastic delay between dust dispersion and sparkover is t_d. The discharge circuit is shown in Figure 3-7. (b) Sequence for ignition testing when using electronic synchronisation between dust dispersion and sparkover with a preset delay t_d. The discharge circuit is shown in Figure 3-8.

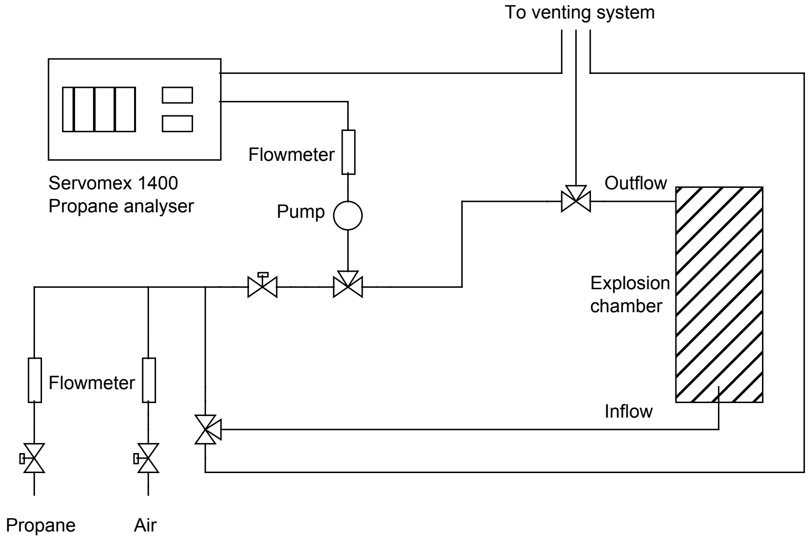


Figure B-4. Schematics of the gas mixing equipment.

When performing MIE tests, the procedure when using the dust cloud itself to trigger the spark [30], is as follows. At first, the voltage at which the electrode gap is preset is found by trial and error, ensuring stable spark triggering for the electrode gap and dust in question. The spark energy is estimated by the gross capacitor energy prior to breakdown ($\frac{1}{2}CV^2$). Ten ignition trials are performed at each energy level, enabling plots of frequency of ignition as a function of capacitor energy.

When synchronised sparks are used [33], a different approach is taken. Because the spark energy is measured for each trial and this energy is somewhat unpredictable, even though the discharge capacitor and electrode gap are held constant, the frequency of ignition can only be presented as histograms (Figure 5 in [33]). Alternatively, the method of presentation shown in Figure 4 and 6 in [33] can be chosen. In this case, the region of ignition uncertainty is illustrated by the overlap of points indicating ignition and the points indicating no ignition.

For both methods, a certain variation in some key parameters is made in order to achieve the lowest possible ignition energies. This applies to e.g. dust concentration, electrode gap length, electrode shape and delay between dust dispersion and sparkover; the latter only applies when using synchronised sparks. However, to keep the number of trials at a reasonable level, the degree of variation has to be limited.

An example of a test procedure involving the use of the dust cloud itself to trigger the spark is shown in Table B-1. For synchronised sparks the equivalent procedure is shown in Table B-2.

Table B-1. Test procedure for determination of MIE of dust clouds, using the dust cloud itself to trigger the spark.

1. Ensure that the dispersion pressure is 7 bar(g).
2. Start with a 0.9 g sample of powder and put it in the dust chamber (exception: dusts that clog the dispersion pipe and nozzle should be placed directly in the explosion chamber cup).
3. Determine the lowest electrode voltage at which the spark is triggered for the chosen electrode gap configuration.
4. Start with a relatively large discharge capacitor, e.g. 3000 pF.
5. Perform 10 ignition trials with successful spark triggering and record the frequency of ignition at this energy level. Also, record the delay between dust dispersion and sparkover t_d (see Figure B-3a).
6. Lower the spark energy by a factor of ~2 and repeat the ignition trials until the dust cloud no longer ignites in 10 successive tests.
7. Vary some of the key parameters in order to achieve optimal conditions for ignition, e.g. dust concentration, electrode gap length and shape, and perform 10 new tests at the spark energy level that yielded no ignition.

The entire procedure should be performed twice for every dust: Once *with* an inductance and once *without* an inductance (if a series inductance can be added in the discharge circuit).

Table B-2. Test procedure for determination of MIE of dust clouds, using sparks that are synchronised with the dust dispersion.

1. Ensure that the dispersion pressure is 7 bar(g).
2. Start with a 0.9 g sample of powder and put it in the dust chamber (same exception as in Table B-1).
3. Choose an ignition delay time (t_d) of 120 ms (see Figure B-3b).
4. Ensure that the charging resistor matches the discharge capacitor by manually triggering the spark and importing the oscilloscope traces into the computer. The integrated spark energy should stabilise within ~100 ns after breakdown, otherwise a larger charging resistor must be chosen (the time constant RC should be $>$ 1 µs).
5. Perform 10 ignition trials at each discharge capacitance level, starting at a relatively high energy level. Record whether the dust cloud is ignited or not. In every trial, the spark energy must be calculated from the oscilloscope traces.
6. Lower the spark energy in steps until no ignition occurs for 10 ignition trials, or until the spark energy cannot be reduced any further.
7. Vary some of the key parameters in order to achieve optimal conditions for ignition, e.g. dust concentration, ignition delay time, electrode gap length and shape, and perform 10 new tests at the capacitance level that yielded no ignition.
8. Represent the ignition data as histograms, showing the probability of ignition within given energy ranges.