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Explosion properties of iron dust in pure oxygen at elevated pressure

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Abstract

A 50 L explosion vessel was purposely designed and manufactured to investigate the explosion properties of iron dust in oxygen at elevated pressure. Pressurized oxygen was injected into the vessel through a perforated tube to blow the iron dust deposited at the bottom of the vessel. This technique has been calibrated to create a homogenous and turbulent flammable dust cloud. The flammable cloud was ignited with a pyrotechnic match delivering 60 J. During the first series of tests, the influence of the initial O₂ pressure and mass of iron were investigated. As expected, the maximum pressure and maximum rate of pressure rise increase when the initial pressure or the ratio mass Fe / mass O₂ increases. However, when the iron mass increased, a spontaneous ignition occurred at the beginning of oxygen injection, resulting in a violent jet fire that perforated the 25 mm thick wall of the vessel. Further investigations are required to explain this observation. The spontaneous ignition could be caused by friction between the iron particles and the steel walls or by electrostatics. The influence of ferric oxide Fe₂O₃ mixed with iron dust was also studied. It resulted in rapid combustion with a high pressure increase quite different from the one caused by a dust explosion.

Keywords: *dust explosion, self-heating, iron, oxygen*

1. Introduction

The use of oxygen is widespread in many industrial applications like combustion, steel production, aerospace industry... Oxygen is usually stored and transported at high pressure. Such conditions promote fast oxidation reactions of metallic and non-metallic materials, which can result in unwanted ignition and severe accidents. Many accidents involving fires and explosions in oxygen-enriched atmosphere were reported in the past (Dicker & al., 1988), sometimes resulting in catastrophic failure and devastating consequences (Saha & al., 2011; Chowdhury, 2011).

Some of them involve metal particle deposits which may be blown in pipework and equipment conveying oxygen and create a flammable dust cloud. The most common ignition mechanisms in such situations are mechanical friction between mechanical parts or between particles, impact of metallic particles on walls, adiabatic compression, electrical arcing, electrostatics (Benson, 2015). Especially friction and impact can generate hot spots with very high temperature which may exceed the auto-ignition temperature of the material. Professional safety rules are implemented by industrial operators (EIGA, 2020) to prevent the risk of ignition. Prevention is usually based on the selection of materials (metallic and non-metallic) compatible with oxygen, geometrical design (to avoid sharp angles) and limitation of the conveying velocity. The American Society for the Testing of Materials (ASTM) have developed many guidelines and standardized test methods to characterize the ignition sensitivity of metallic and non-metallic materials in enriched-oxygen atmospheres (ASTM G63, G72, G74, G86, G88, G94, G124) which can be used for the design of high-pressure oxygen installations. In addition, if one wants to design protection barriers to mitigate the explosion effects, like an explosion resistant enclosure, it is required to characterize the explosion pressure and pressure rise

during such events. Whereas explosion properties of metal dusts in air have been extensively investigated (Cashdollar, 2007), very limited data are available concerning the explosion properties of iron dust in pure oxygen.

The present work aims at producing experimental data on the explosion properties of iron dust in oxygen at elevated pressure. The testing conditions are selected to represent the pressure, velocity and turbulence level which are present in some oxygen production unit equipment like filters where iron dust can accumulate in operation. Tests are conducted in a similar way than the standardized tests usually performed to determine maximum explosion pressure and maximum rate of pressure rise (EN 14034-1 and -2). The influence of the initial pressure and iron dust concentration are investigated, as well as the effect of the addition of iron oxide dust to the iron dust. A secondary objective is to observe if the test conditions may result in a spontaneous ignition.

2. Experiments

2.1. Description of the experimental setup

An explosion vessel was purposely designed and manufactured for the project (Fig. 1). The vessel is a 50 L steel cylinder made of a 25 mm thick tube welded at the bottom to a steel cap and closed on the top by a steel flange. It is designed to withstand at least 150 bar. Two 5 L reservoirs pressurized with oxygen are connected through the upper flange to a vertical perforated tube into the vessel. The iron dust is placed in an aluminum cup at the bottom of the vessel. Due to the high combustion temperature, the aluminum cup melts and burns during the test and must be replaced at each test. It is assumed that the combustion of this aluminum cup does not interact with the dust cloud explosion as the combustion durations are not of the same order.

A fast-acting valve is mounted on the oxygen injection line. When it opens, oxygen is discharged into the vessel through the multiple holes of the perforated tube and blows the dust to create a flammable dust cloud. The flammable cloud is ignited close to the top flange by a pyrotechnic igniter delivering 60 J. The air initially present in the vessel is not evacuated before oxygen injection, so a small volume of nitrogen (about 40 L at atmospheric pressure) remains in the vessel during the test. When the initial pressure equals 30 bar, this represents about 2,5% of the total gas volume in the vessel. It is assumed to be negligible compared to the quantity of oxygen injected.

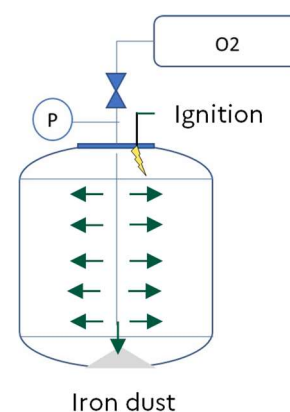


Fig. 1. View and scheme of the experimental setup of the explosion vessel

Attention is paid to the design of the perforated tube. It is inspired from the perforated ring used for standardized explosion tests described in EN 14034-1. This injection technique produces several high-speed jets at each hole of the tube. When the jets impact the vessel walls, the flow velocity is converted into recirculation velocity which allows both homogenous mixing of the dust and production of isotropic turbulence (Dyduch & al., 2016). Actually, the turbulence parameters in the

vessel mostly depend on the number and diameter of the holes (Proust et al., 2007). An additional issue comes from the high density of the iron dust.

An engineering tool based on the jet theory (Proust et al., 2009) is used to calculate the number and the diameter of holes on the perforated tube which are required to produce the desired turbulence intensity.

There is not any available technique to measure turbulence in the explosion vessel in oxygen-enriched atmosphere at high pressure. Consequently, to calibrate the system and validate the design of the perforated tube, it is first tested at smaller scale in a 7 L transparent vessel with pressurized nitrogen injection (Fig. 2) The transparent vessel is illuminated by a laser sheet and filmed. Video post processing is used to assess the turbulent velocity in the vessel (Bozier and Veyssi re, 2005) and compare the results to the calculations (Fig. 3). As expected, the turbulence intensity is not constant during the gas injection. It drops with the discharge pressure and falls to 0 very quickly after the end of injection. Therefore, the initial injection pressure and the final injection pressure are set so that the turbulence intensity at the end of injection is about 2 m/s as it is in the standard 1 m³ vessel (Proust et al., 2007). A good agreement is achieved between prediction and measurements. Then it is assumed that the design rules can be upscaled to the 50 L explosion vessel without any further verification.

Finally, the design parameters of the perforated tube are: tube diameter 4 mm, with one 2 mm axial hole at the bottom and fourteen (2 x 7) 1 mm radial holes homogenously distributed along the tube length. The initial pressure in the O₂ reservoirs depends on the expected initial pressure in the explosion vessel. The typical duration of oxygen injection is 2 s.

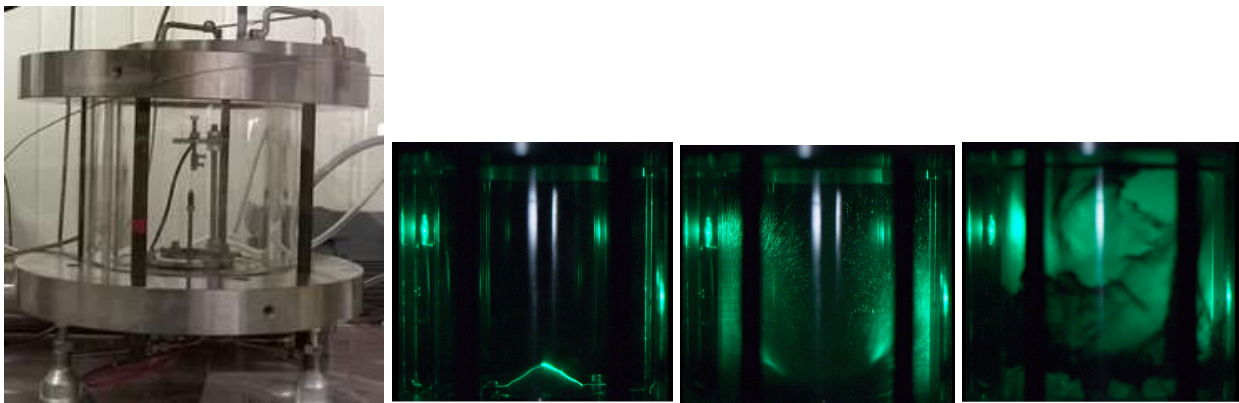


Fig. 2. 7 L transparent vessel used for calibration of the dust injection system setup

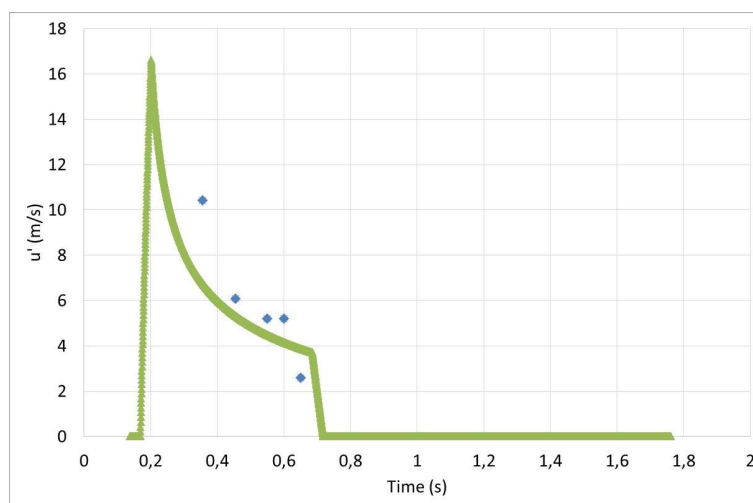


Fig. 3. Turbulence intensity in the transparent 7L calibration vessel – comparison of calculation (green curve) and measurements (blue dots) – Nitrogen injection pressure 130 bar

Ignition and injection valve closing are triggered simultaneously, so that there is no jet velocity anymore, whereas the turbulence is still present.

The dust is selected so that it can be easily dispersed, and it remains in suspension during the combustion. The fluidized bed theory (Davidson and Harrison, 1985) mentions that the first requirement is fulfilled when the particle diameter is larger than 20 to 40 μm . At the opposite, the free fall speed must be lower than the turbulent velocity assumed to rank a few m/s: for dense particles like iron, this happens when particle diameter remains below 100 μm . Finally, tests are conducted with iron dust (purity 99%) with a maximum particle size of 60 μm (Table 1).

Table 1. Particle size distribution (in μm)

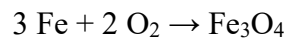
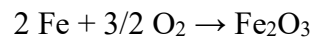
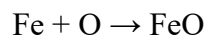
Dust	Dv(10)	Dv(50)	Dv(90)
Fe	20.8	34.9	55.6
Fe ₂ O ₃	1.1	9.8	29.4

The explosion pressure is measured with a piezoresistive pressure sensor Kistler 0-200 bar. The sensor is remotely connected to the injection pipe rather than directly to the vessel, so that it is protected from the very high combustion temperature in the vessel. A few calibration tests are first carried out with a reference case (hydrogen-air explosion) in order to check that the pressure measurement is not disturbed by any acoustic effect in the injection tube.

All the tests are remotely controlled and monitored with a HD camera to detect any unexpected events.

2.2. Theoretical predictions

Preliminary calculations are performed with the CIRCE software (Liu & al.) to estimate the explosion pressure depending on the initial ratio of iron mass and oxygen mass. A Monte-Carlo method is used to calculate the final equilibrium by minimization of the Gibbs energy. The reactants are solid iron (Fe) and oxygen (O₂). The remaining species after combustion are Fe (solid, liquid and gas), O, O₂ (gas), and stable solid iron oxides FeO, Fe₂O₃, Fe₃O₄ which result from the following reactions:



Note that two mechanisms are in competition during the combustion process: on the one hand, the gaseous oxygen is consumed and converted into solid oxides, so that in the event of complete combustion there would be no more gas after combustion and therefore no pressure in the vessel. On the opposite, the very high combustion temperature partially vaporizes iron and heats the gases, which increases the pressure. Finally, the maximum explosion pressure (about 166 bar) would be reached when there is an excess of oxygen compared to the initial mass of iron (Table 2) i.e. when the molar ratio equals 15% mol Fe/mol O₂. The corresponding mass ratio is about 0.3, much lower than the stoichiometric concentration. The final composition of iron oxides depends on the initial molar ratio of iron and oxygen.

Table 2. Explosion properties calculated with CIRCE (initial conditions 30 bar, 10°C)

Molar ratio (%mol Fe / mol O ₂)	Mass ratio (kg Fe / kg O ₂)	Maximum adiabatic temperature (°C)	Maximum explosion pressure (bar)	% Solid after explosion (mol/mol)	Combustion products
60	1.05	3662	57	80	FeO
50	0.87	3284	95	66	FeO
40	0.7	2758	127	49	FeO
30	0.52	2226	143	34	FeO
20	0.35	2041	157	17	Fe ₂ O ₃ + FeO
15	0.26	1901	166	9	Fe ₂ O ₃
10	0.17	1292	134	5.5	Fe ₂ O ₃

3. Results and discussion

Twelve tests are performed in total. The first series (tests n° 1 to 4) aims at investigating the influence of the initial pressure, from 5 bar to 30 bar which is the common service pressure in oxygen operating units. The second test series (tests n°5 to 8) addresses the influence of the iron/oxygen mass ratio. Finally, the third test series addresses the influence of the addition of ferric oxide Fe₂O₃ to the iron dust. Tests conditions and main results are presented in Table 3.

Table 3. Test conditions and main results

Test n°	Initial pressure P _{init} (bar)	Mass Fe (g)	Mass ratio Fe ₂ O ₃ (g) / Fe (g)	Concentration (kg Fe / kg O ₂)	Maximum explosion pressure P _{max} (bar)	P _{max} /P _{init}	Maximum rate of pressure rise (dP/dt)
1	5	114	0	0.3	16.5	3.2	38
2	10	228	0	0.3	32.3	3.2	149
3	20	456	0	0.3	67	3.3	182
4	30	683	0	0.3	76.7	2.6	78
5	30	341	0	0.15	41.7	1.4	8
6	30	1366	0	0.6	107.5	3.6	213
7	30	2730	0	1.2	136.6	4.6	490
8	30	5640	0	2.4	<i>Spontaneous ignition</i>		
9	30	341	1	0.15	35.7	1.2	0.3
10	30	1366	1	0.6	38	1.3	0.6
11	30	1366	0.5	0.6	64	2.1	6.4
12	30	1366	0.25	0.6	66.5	2.2	14.5

Some pictures of a typical test (Test 7) are shown on Fig. 4. After all tests a rust deposit is visible on the internal vessel walls and on the injection tube. This color is characteristic from Fe₂O₃ production. There is no visible trace of the other iron oxides (FeO, Fe₃O₄) in any test. The Fe₂O₃ deposit can be easily cleaned before each new test at the beginning, but after many tests there is always a thin oxide layer which sticks to the wall. Moreover, melt iron which does not participate to the combustion also accumulates in the bottom of the vessel after test. Unlike iron oxide it is almost impossible to remove this iron layer before each new test because it is “welded” to the vessel.

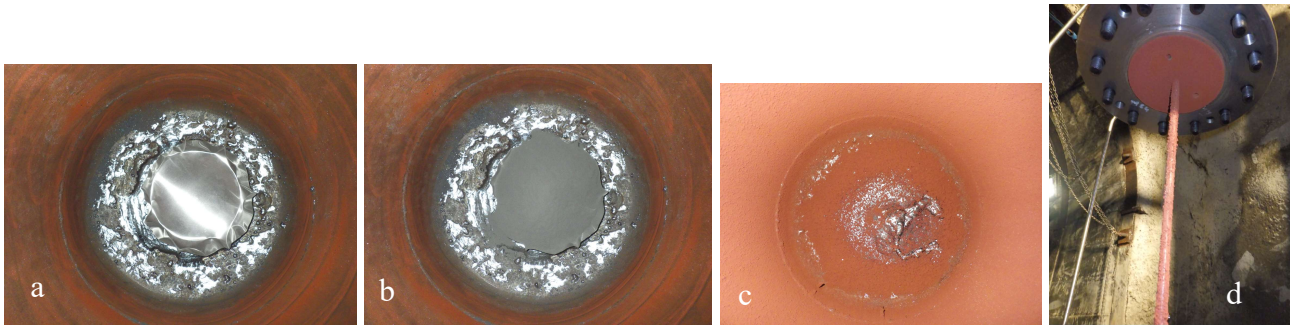


Fig. 4: Pictures of Test 7 – a, b, c : bottom of the vessel – a: aluminum cup (empty); b: cup filled with iron dust ; c: iron oxide dust (from combustion, after the test); d: iron oxide deposit on the injection tube

3.1. Influence of the initial pressure

A first series of tests is performed to investigate the influence of the initial oxygen pressure from 5 bar to 30 bar. Pressure curves are plotted on Fig. 5. They are typical of a dust explosion with a pressure rise duration lower than 500 ms. Tests are carried out at the theoretical optimum mass ratio which should result in the maximum P_{\max} . As expected, the maximum explosion pressure P_{\max} increases when the initial pressure P_{init} increases as well, and the ratio P_{\max}/P_{init} is constant (Fig.6). However, the P_{\max} value is much lower than the calculated value (see Table 2): with $P_{\text{init}} = 30$ bar one gets only $P_{\max} = 76.7$ bar, whereas the expected value is about 160 bar.

Cashdollar & Zlochower (2007) performed an extensive testing program with metal dusts in the 20- L sphere at atmospheric conditions. They also observed a large difference between the experimental P_{\max} and the theoretical predictions in adiabatic conditions. For two different iron dust samples with different particle size distribution, the measured P_{\max} was 40% to 60% lower than the adiabatic P_{\max} . The same observation was made for the maximum measured temperature T_{\max} which was also 40% lower than the calculated adiabatic T_{\max} . They noticed that the prediction was better for very fine particles (mean diameter $\sim 4 \mu\text{m}$) than for coarser ones (mean diameter $\sim 45 \mu\text{m}$) because the former are totally vaporized, whereas the latter volatilize only partially.

Then, the gap between the experimental and calculated values of P_{\max} may come from the adiabatic hypothesis (the explosion vessel is not adiabatic), the particle size which influences the volatilized fraction of dust, and last but not least, the efficiency of the dust dispersion, especially at high concentration.

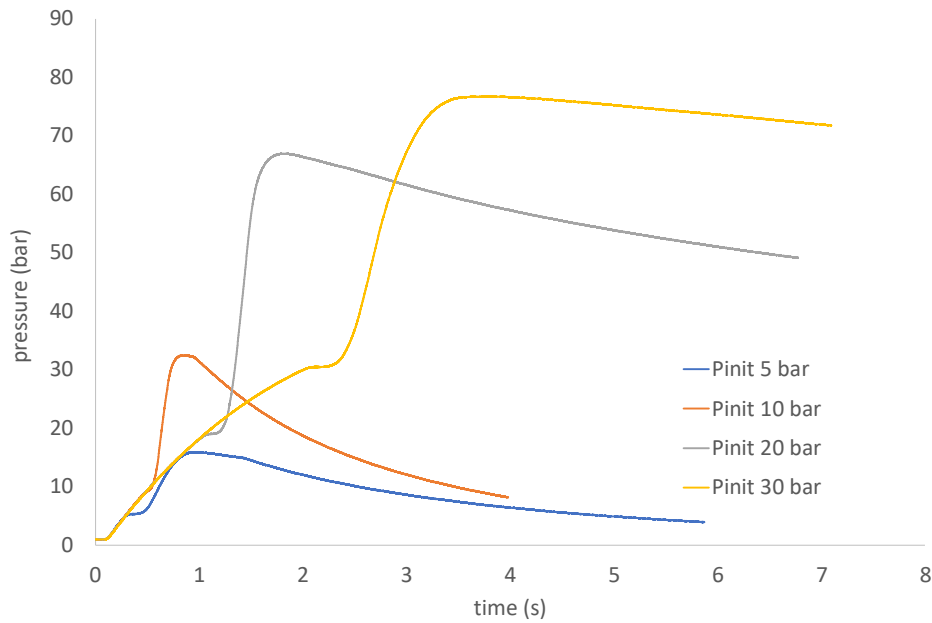


Fig. 5: Pressure-time curves for Tests 1 to 4 – Influence of P_{init} . Oxygen injection starts at $t=0$ – Injection duration, and therefore ignition time, vary with P_{init}

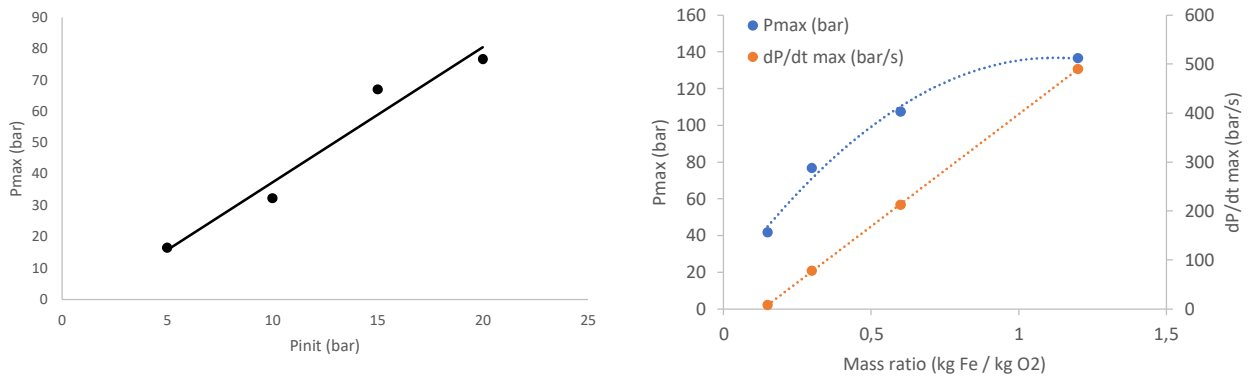


Fig. 6: Left : Dependency of P_{max} on P_{init} for $kgFe/kgO_2=0.3$ – Tests 1 to 4
Right: Dependency of P_{max} and dP/dt_{max} on mass ratio – Tests 4 to 7

3.2. Influence of iron mass

The next series (Tests 4 to 8) is performed at $P_{init} = 30$ bar with variation of the mass ratio Fe/O_2 . Pressure-time curves are plotted on Fig. 7, and the influence of the mass ratio Fe/O_2 on P_{max} and dP/dt is plotted on Fig. 6. In all the tests O_2 injection starts at $t=0$ and ignition occurs at $t = 2.1$ s. At 0.15 kg $Fe/kg O_2$ (Test 5), the dust cloud does not ignite immediately, and the very low pressure increase is attributed to a delayed combustion of the dust. Test 8, with a mass ratio closed to stoichiometry, is discussed in the next chapter. It seems that the maximum pressure during Test 7 ($P_{max} = 136.6$ bar) with a mass ratio of 1.2 kg $Fe/kg O_2$ is closed to the calculated worst case ($P_{max} = 166$ bar – see Table 1), with. The corresponding K_{st} value, calculated as $(dP/dt) \cdot V^{1/3}$ with the vessel volume, reaches 180 bar.m/s. If we compare this value to the K_{st} of iron dust in air at atmospheric conditions, the iron dust looks much more reactive in oxygen than in air. The available databases (Cashdollar & Zlochower; 2007; Staubex database) mention that iron is classified as a ST1 dust with a typical K_{st} value of 30 to 40 bar.m/s. The initial elevated pressure also plays a role.

However, if we focus on the mass ratio which results in the maximum explosion pressure, the experimental mass ratio (1.2 kg $Fe/kg O_2$) is much higher than the calculated value (0.3 kg $Fe/kg O_2$).

Again, we suspect that the adiabatic hypothesis does not fully apply, but also that the dust dispersion is not fully efficient when the dust mass exceeds $\sim 1\text{kg}$ in the vessel, and that a significant part of the dust may not contribute to the explosion. According to Cashdollar & Zlochower (2007), it could be highly dependent on the fraction of dust which vaporizes during the combustion. This assumption is difficult to verify in our experimental setup. It can only be noticed that at high concentration, part of the iron melts and accumulates at the bottom of the vessel.

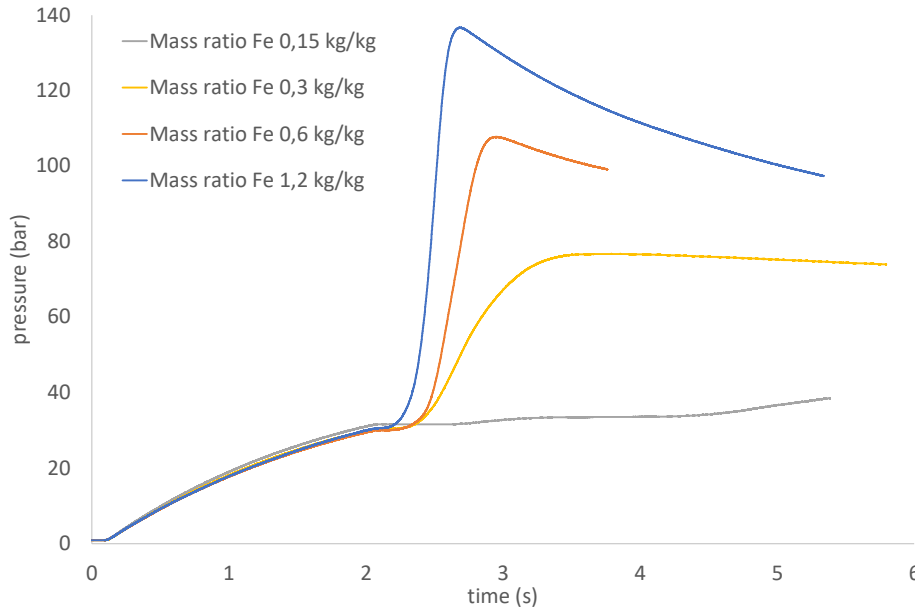


Fig. 7: Pressure-time curves for Tests 4 to 7 – Influence of mass ratio Fe/O_2 . Oxygen injection starts at $t=0$ – Ignition at $t = 2.1\text{ s}$

3.3. Spontaneous ignition

Test n°8 was performed with an increased mass of iron (5460 g) and an oxygen initial pressure of 30 bar. This test condition (mass ratio 2.4 kg Fe / kg O_2) is very close to the stoichiometric conditions since only Fe_2O_3 is formed (mass ratio 2.3 kg Fe / kg O_2). However, a spontaneous ignition occurred 250 ms after oxygen began to fill the vessel. The pressure started to increase immediately like in a dust explosion, but 100 ms later the 25 mm thick wall of the vessel was perforated. These events are visible on the pressure-time curve of Fig. 9. The test resulted in a violent phenomenon characterized by a flame jet outside the vessel (Fig. 8). The jet stopped at the end of oxygen injection. The oxygen injection tube completely disappeared during the test, and a 2 cm diameter hole was created in the vessel wall.

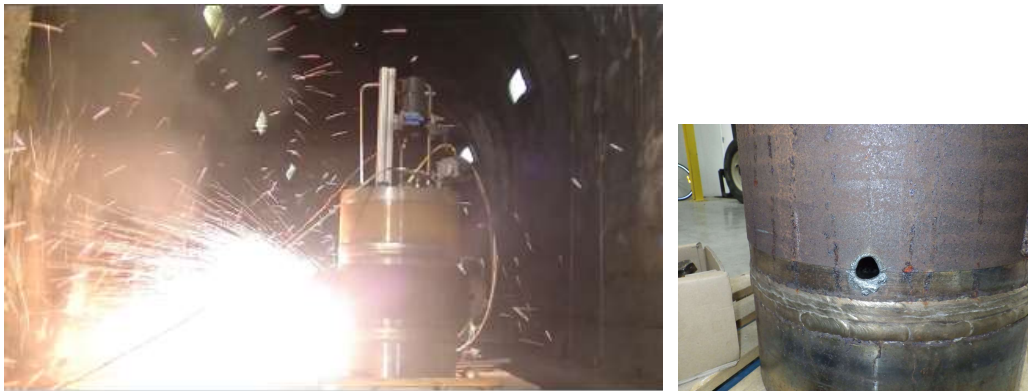


Fig. 8. View of test 8 after the vessel has been perforated

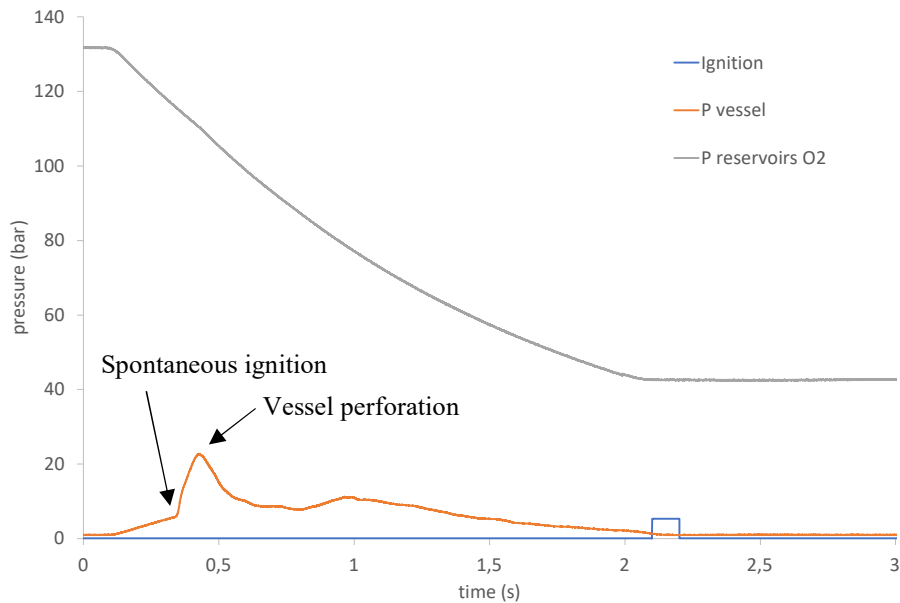


Fig. 9. Pressure-time curve in Test 8 - spontaneous ignition

There is no evidence about the ignition mechanism involved. The dust ignites spontaneously, before the ignition source is activated. This test is characterized by a large quantity of iron particles, which promotes friction and many shocks between particles and many impacts of particles on the internal steel walls. Mechanical impact or friction can generate very hot spots, especially at the beginning of oxygen injection, when the particles momentum is the highest.

Adiabatic compression can be excluded because the ignition occurs when the oxygen pressure is only 6 bar in the explosion vessel, and the corresponding temperature increase is not sufficient to cause auto-ignition.

The last explanation could be an electrostatic discharge. The iron dust is conductive so it cannot accumulate charges, but iron oxide particles are present on the vessel walls from the previous tests and may be blown from the walls and accumulate electric charges. Then electrical arcing could occur and ignite the dust cloud. Further investigations are required to understand the spontaneous ignition mechanism and in which conditions it occurs, as the present experimental setup has only a very limited instrumentation.

The mechanism of perforation of metallic pipes was investigated by Dieguez & al (1988) who set-up an experiment to investigate the risk of perforation of a “hollow vessel” containing an oxygen flow. The experiment aimed at assessing the resistance to ignition of metals and alloys used in oxygen pipes and equipment. It was carried out as follows: a constant flow of oxygen at a given speed (5 cm/s), pressure and temperature is supplied to a section of pipe (the so-called “hollow vessel”, 1 m long and 80 mm diameter). A capsule containing a small quantity of iron dust (less than 200 g) and an electrical igniter is placed on the wall of the pipe. When the igniter is triggered, it causes the combustion of the iron dust on the internal surface of the wall. More than 600 parametric tests were performed. Depending on the initial conditions (oxygen purity, pressure, temperature), pipe material and pipe wall thickness, the combustion can propagate and burn more or less the pipe wall. Sometimes the pipe is totally perforated. Then the pipe is analyzed, the hole is characterized and the time between ignition and perforation of the wall is measured. Many interesting results were obtained. First of all, for a given initial pressure and wall thickness, the probability of perforation depends on the mass of iron dust in the capsule. For instance at 30 bar, a mass of iron dust of 150g is required to perforate a 10 mm thick carbon steel pipe, and the perforation takes about 25 s. Secondly, the iron dust capsule ignited spontaneously when the oxygen temperature was higher than 300°C.

In our experiment, the perforation went much faster and made a hole in 100 ms after ignition. This observation is not consistent with the results from Dieguez & al. However, our experiment is characterized by a high flow velocity in the vessel, which was not the case in the Dieguez & al experiment. Therefore, the hole in the vessel may result from the impingement of a large oxygen jet produced by the degradation of the injection tube. In such case, the ignition might have occurred close to the injection pipe, rather than on the vessel wall.

3.4. Influence of iron oxide

After Test 8 the vessel was repaired and a new injection tube was manufactured. Then the last test series was performed with addition of fine iron oxide dust Fe_2O_3 mixed to the iron dust. Tests 10 to 12 were carried out with the same mass ratio Fe/O_2 (0.6 kg Fe / kg O_2) and the mass ratio $\text{Fe}_2\text{O}_3/\text{Fe}$ varying from 0.25 to 1. The pressure time curves are plotted on Fig. 10 together with the result of Test 6 which was performed with 0.6 kg Fe / kg O_2 but without any oxide addition. When oxide is added, a very different behavior is observed with a low but continuous pressure rise, which takes several seconds to reach the maximum pressure. This is definitely not a dust explosion. It looks like a rapid combustion with a high temperature rise which heats the gaseous phase and increases the pressure, similarly to what happens during a self-heating phenomenon.

This observation questions the way the combustion begins in the vessel. Obviously, even if the dust cloud is ignited by the pyrotechnic igniter, the flame does not propagate in the cloud because it is inerted by the presence of iron oxide dust. Then the ignition does not turn into an explosion. However, the combustion reaction starts. Is it due to the presence of very hot particles produced by the igniter which falls on the iron deposit? Or is it spontaneous ignition? It would be interesting to repeat the same tests without any igniter.

Unsurprisingly, the maximum pressure and the pressure rise increase when the amount of oxide dust decreases. Different mechanisms are in competition: on one side, oxygen is consumed so that gas is converted into solid oxide. On the other side, the very high temperature (about 2000 K) heats the excess of oxygen and may also vaporize part of the iron, which is responsible for the pressure increase. Once watching at the monitoring video, one can hear distinctly a “boiling noise” which could be attributed to a liquid pool of iron boiling at the bottom of the vessel.

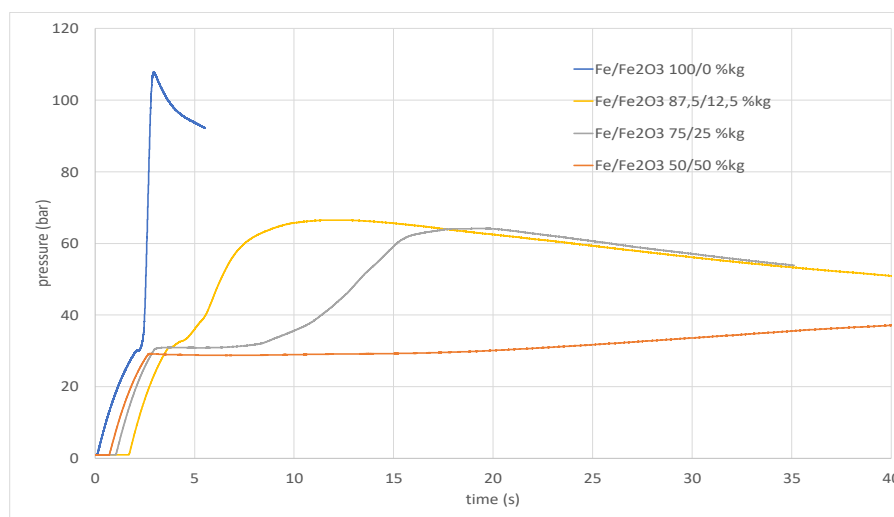


Fig. 10. Pressure-time curves for Tests 4 to 7 – Influence of iron oxide concentration

4. Conclusions

This work highlights three different behaviors when iron dust is dispersed in oxygen at elevated pressure:

- A classical “dust explosion” behavior but with an increased reactivity of iron dust compared to atmospheric conditions in air;
- A rapid combustion behavior similar to self-heating, which cannot be considered as an explosion, but can generate significant overpressure and very hot temperatures.
- Spontaneous ignition which might be promoted by mechanical friction and shocks between particles, and/or electrostatic discharges. We assume that spontaneous ignition could occur either in a dust cloud or in a dust deposit, and could be activated more easily when iron oxide was present.

This work gives interesting perspectives for the safety of oxygen operation units. First, the iron dust looks much more reactive in oxygen than in air. These new data must be considered for designing an explosion resistant equipment. Secondly, the rapid combustion phenomenon is probably correlated with a high and long temperature increase of the vessel walls. It could cause at the same time a loss of mechanical resistance of the walls, and a significant pressure rise in the vessel. Finally, the spontaneous ignition must be investigated further to be prevented because if it happens, it could result very quickly in a catastrophic failure, with very limited possibilities to mitigate the consequences.

Acknowledgements

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