

# Auto-ignition Conditions of Iron Micro Powders in Heated Oxygen

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## 1 Introduction

Information about ignitibility of metal particles is important for variety of practical applications associated with propulsion, military and industrial safety. In particular, metal particles can be used as catalytic additives that improve the combustion efficiency of conventional fuels [1] and suppression of detrimental emissions from stationary combustion sources [2]. Numerous publications were devoted to the study of explosion characteristics of metal and nonmetal dusts suspended in air [3-5]. The other interesting for fundamental science and important for practical applications case is auto-ignition of particles resided in layer. Local ignition can cause slow and smoldering combustion of the layer. Simulating this process is a comprehensive physical-chemical problem that combines both kinetic oxidation processes of individual particles and heat transfer processes in particle-particle and particle-gas systems. Development and checking the adequacy of the corresponding models therefore requires input data such as critical parameters of a gas medium, at which auto-ignition occurs, their dependence on particle material and size as well as information about ignition delay times and their dependence on oxygen temperature and pressure. The sufficient amount of experimental data was obtained in this study by means of rapid compression machine (RCM), which was used for generating heated oxygen atmosphere for wide ranges of pressures and temperatures. Critical conditions at which auto-ignition of iron micro particles resided in layer occur were determined in dependence of particles sizes. Also some characteristics of ignition and combustion processes like ignition delay time and combustion temperature were obtained.

## 2 Experiments

A high compression ratio of used RCM (up to 80) provided both high pressure and high temperature at the end of compression stroke. Fig. 1 shows the general layout of the compression cylinder and measurement setup. The test chamber (2) is equipped with a high-temperature quartz pressure sensor Kistler 6031U18 (4) combined with a Kistler 5015A charge amplifier (11), a gas inlet/outlet valve (5) and quartz window (6) to the mandrel of which the bifurcated optical fiber light guide was connected for recording the luminescence appeared in test volume at ignition and combustion. The neutral filter (optical density  $D=3.74$ ) was installed before photocathode of one of

photomultipliers. This allowed detecting both weak luminescence at local auto-ignitions of individual particles and strong luminescence at combustion of whole particle layer. An optical reflection probe (8) was used for measuring positions of the compression piston and hence the current gas volume during the compression stroke.

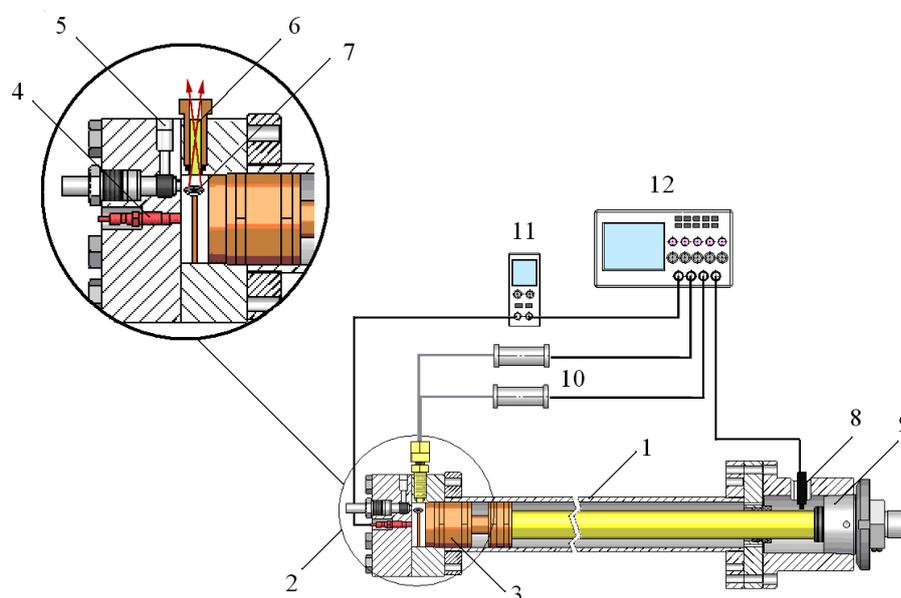


Figure 1. The layout of the compression cylinder and measurement system: 1 – compression cylinder, 2 – test chamber, 3 – compression piston, 4 – pressure sensor, 5 – inlet gas and vacuum valve, 6 – quartz window, 7 – ceramic cup with particle sample, 8 – optical reflection probe, 9 – conical stopper, 10 – photomultipliers, pyrometer and spectrometer, 11 – charge amplifier, 12 – digital oscilloscope.

The experiments has been performed for five iron powders: two metallurgical ultra fine iron powders with main particles fraction 1-3  $\mu\text{m}$  and 1-5  $\mu\text{m}$ , respectively, and three powders separated by sieves 45  $\mu\text{m}$ , 56  $\mu\text{m}$ , 63  $\mu\text{m}$ , 80  $\mu\text{m}$ , and 125  $\mu\text{m}$ . All powders were examined by certified picture scanner “Mini-Magiscan”. The results of accurate granulometric analysis are presented in Table 1. It was found that the main fraction of powders consisted of the particles with size ranges lightly different from the sizes of the sieves meshes. Therefore we used the main fraction sizes (the size range of particles generally presented by quantity in powder) as identification of each powder.

Table 1. Results of granulometric analysis for studied powders

Powder	Size range (min-max), $\mu\text{m}$	Main fraction			average by quantity size (length/weight), $\mu\text{m}$	average by mass size, $\mu\text{m}$	
		by quantity		by mass, $\mu\text{m}$			
		horizontal projection, $\mu\text{m}$	vertical projection, $\mu\text{m}$				
metallurgical ultra fine iron powders	0.2-9.2	1-3	1-3	2-4	2.59/2.16	3.4	
	0.2-16.4	1-5	1-3	4-5	4.01/3.08	4.7	
powders separated by sieves	45 $\mu\text{m}$	9.0-98.1	20-40	30-40	30-60	42.92/30.56	43.8
	56 $\mu\text{m}$	6.7-180.3	60-90	70-90	70-90	82.76/59.01	80.6
	63 $\mu\text{m}$						
	80 $\mu\text{m}$	32.8-268.1	110-140	100-110	130-160	155.48/114.8	140.2
125 $\mu\text{m}$							

The particle samples (mass of 0.05 g) were placed in a small ceramic cup (Fig. 1 (7)) mounted in the test chamber of RCM.

Temperature of burning particles was measured with time resolution of 4  $\mu$ s by photoemission method based on analysis of photoelectron energy distribution. The essence of this technique has been extensively described in [6, 7]. The applicability of this method was proved by observation of emission spectrum during ignition and combustion. The registered spectrum in 300–600 nm was continuous and similar to emission spectrum of solids. It allowed us to use the preliminary temperature lamps calibration for determining current temperatures during particles auto-ignitions and burning. Moreover the measured temperature well agrees with color temperature calculated from spectrum by method described in [8]. The advantage of used method is a possibility of temperature measurements with temporal resolution up to 1  $\mu$ s.

### 3 Results

The results of all experimental runs are plotted on Fig. 2 in terms of oxygen pressures and temperatures generated by RCM. Conditions at which auto-ignition and combustion was observed marked by filled symbols. The critical parameters of oxygen needed for auto-ignition are connected by color lines on Fig. 2. It is seen that critical temperature strongly depends on oxygen pressure and this dependence intensifies with decreasing particle sizes. Therefore the kinetic characteristics like dependence of ignition delay time on temperature must be determined at similar pressures.

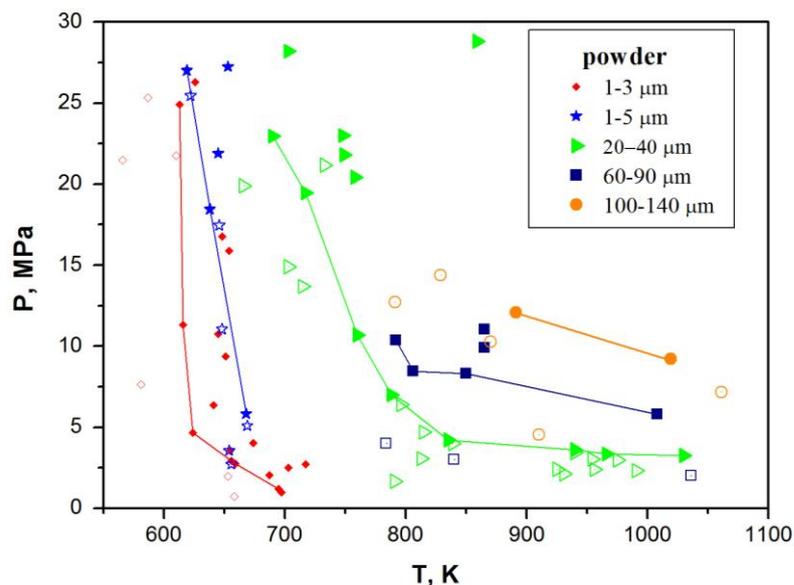


Figure 2. Pressures and temperatures of oxygen at which auto-ignitions of particle samples were observed (filled symbols) and were not observed (empty symbols).

The ignition delay time was defined as the time difference between pressure peak at the end of compression stroke and the onset of emission (5% of maximal rise) registered by photomultiplier with neutral filter. The example of pressure, luminescence and pyrometer's signals recorded during compression, ignition and combustion of is presented on Fig 3. The powder 1-3  $\mu$ m was tested in this experiment and post compression oxygen temperature was 625 K. Measured in this test ignition delay time is indicated on graph. A weak luminescence was always observed at the end of compression event for post compression oxygen temperatures higher then 600 K. This light (marked on Fig. 3 by orange ellipse) is not connected with ignition of particle samples. It corresponds to ignition of submicron particle ejected from piston's seals due to friction on cylinder surface during compression

stroke. This conclusion was confirmed by experimental runs without any particle samples. The measured temperature of burning particles is also presented at Fig. 3 (line 4). It was calculated by pyrometer's signal processing. The detected temperatures near the onset of particles auto-ignition are varied from  $2450 \pm 50$  K to  $3100 \pm 50$  K and then slow down to the value of  $1850 \pm 50$  K which is close to melting temperatures of iron (1812 K) and iron oxides. The maximal temperature at auto-ignitions of iron powder (1-3  $\mu\text{m}$ ) didn't exceed value of 3100 K, which is close to iron boiling temperature. Thus, probably the iron boiling limits the maximal temperature, due to high latent vaporization heat of iron.

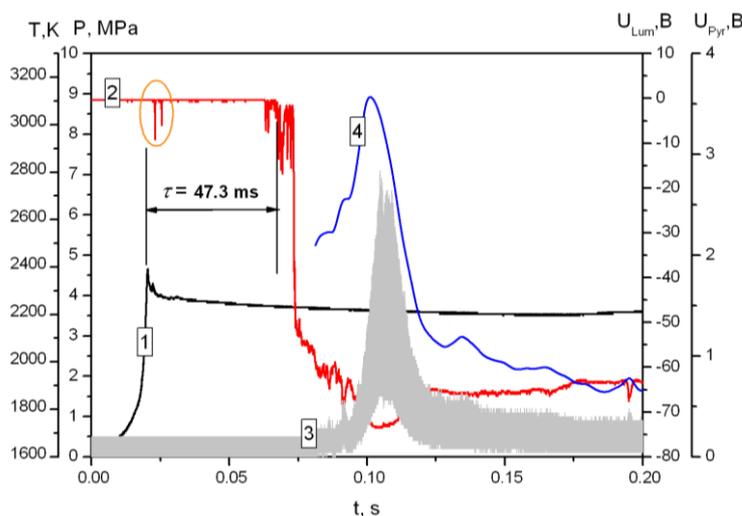


Figure 3. Signal records during oxygen compression and particles auto-ignition: 1 – oxygen pressure, 2 – luminescence in test volume, 3 – signal of pyrometer and 4 – calculated temperature history.

Ignition times of iron particles are strongly depended not only on oxygen temperature but also on oxygen pressure (Fig. 2). In order to point out this dependence we collected auto-ignition data on powder 1-3  $\mu\text{m}$  particles for the same temperatures and different pressures (Fig. 4). It was found that at oxygen temperature  $650 \pm 10$  K ignition time strongly increases with reducing oxygen pressures to values less than 4 MPa. For higher temperature ( $700 \pm 15$ ) the considerable effect of oxygen pressure on auto-ignition time was observed at pressures less than 2 MPa.

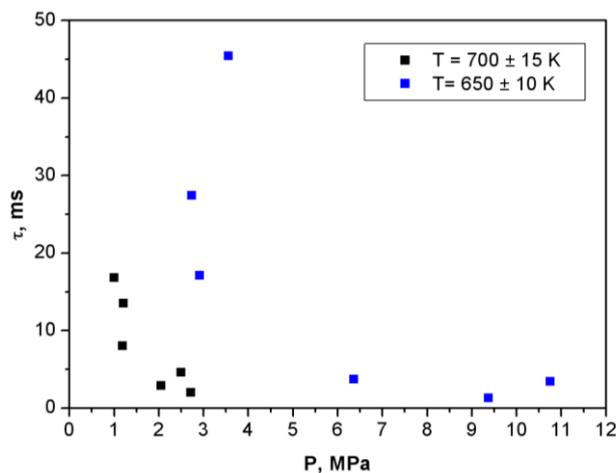


Figure 4. Pressure dependence of ignition times of iron powder 1-3  $\mu\text{m}$  in oxygen atmosphere at different temperatures

For different particles, the pressure behaviour of ignition times is presented in the Fig.5. The lines on this plot correspond to linear approximations of measured ignition times for specified powder at certain oxygen pressure range. It is seen that lines have similar angle that is why pressure influence on ignition time can be presented by introduction of some coefficient. The plotted dependences are rather coarse and have a big scatter but nevertheless are useful for validation of mathematical models describing auto-ignition of iron particles in heated oxygen. Moreover, this data is useful for safety evaluation of technological processes during which iron micro particles and heated oxygen atmosphere can meet at the same time.

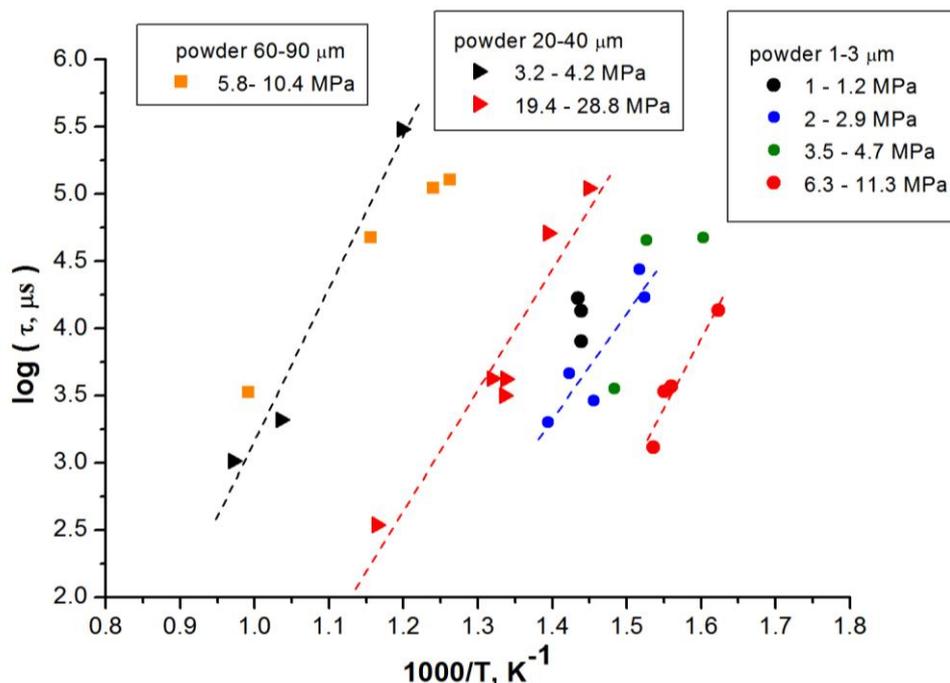


Figure 5. Ignition times vs. oxygen temperature for studied iron powders and oxygen pressures.

## 4 Conclusion

The auto-ignition and combustion of iron micro particles in heated oxygen has been studied by means of rapid compression machine for oxygen pressures varied from 0.5 to 28 MPa and temperatures varied from 550 to 1100 K. The critical conditions that can provoke auto-ignition and combustion were determined for five powders with particle sizes from 1 μm to 140 μm. It was found that iron micro particles can easily ignited in rapidly heated oxygen atmosphere at temperatures much lower than iron boiling temperature. Moreover this critical temperature decreases significantly with increasing oxygen pressure. The influence of oxygen pressure and temperature as well as particle size on ignition delay time was established. The received dependences can be easily applied for the prediction of iron micro powders behaviour in the rapidly heated oxygen atmosphere. The variation of temperature developing during iron combustion of powder 1-3 μm was measured with temporal resolution of 4 μs. It was found that the temperature can shortly arise up to 3100 K. The collected data is important for development of new technological processes during which iron micro particles and heated oxygen atmosphere can appear together. Moreover, obtained database is useful for development and validation of mathematical models describing auto-ignition and combustion phenomena of iron particles in heated oxygen.

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