# HOT SPOTS SENSITIZATION AND OPTICAL DETONATION MEASUREMENTS OF EMULSION EXPLOSIVES

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Emulsion explosives are non-ideal explosives. The composition, rheological structure and its detonation properties were presented in previous ICT Conferences. Emulsion matrix is only detonable if it is sensitized (generally by hollow microspheres dispersed in the emulsion gelatin phase). Two kinds of spheres were used in experiments – expanded dry micro-spheres of 50 µm mean diameter (DE) and 5 mm expanded polystyrene spheres (EPS). The last big EPS spheres were successively dispersed in emulsion gelatin (emulsion matrix) and in emulsion explosive sensitized with DE microspheres. This sensitization mechanism can be explained by the deformation and collapse of the hollow microspheres, under shock detonation transmission, generating compressed hot gas and very high shear stress conditions in two lateral pockets. These two hot spot zones insure ignition of the outside emulsion matrix, generating, by the progress of reaction, cells and compressed layers, very well recorded in experimental tests, by the printed figures in copper witness plates. The experimental micro dynamic measurements were performed using optical techniques, using optical fibers and fast opto-electronic converters (presented previously in ICT Conferences). Two kinds of optical multimode fibers can be used in these measurements: PMMA and silica fibers. In a similar way, two kinds of opto-electronic receivers were used, as a function of optical range (maximum sensibility at 650 nm and 850 nm). Rise time is less than 50 ns. Silica fibers of 60 µm present more precise experimental measurements, due its reduced diameter and its more difficult ignition behavior with atmospheric air, when shocked from expansion of detonation products. Record signals show clearly the collapse of the EPS sphere. It shows the arrival of shock detonation front in sphere, the disappearing of light during collapse of sphere, and at the end, the very intense light generated by hot spots. Then two reaction progress regions seem to be developed. Their progression generate, sometimes, a central reaction zone, proved by the printed crater at the copper witness plate. This complex phenomena ensure the self-sustained detonation progression.

**Keywords**: emulsion explosives, hollow microspheres sensitization, detonation thermal radiation, optic fiber, opto-electronic converter, micro sphere collapse under detonation front, copper witness plate printed figures.

### 1. INTRODUCTION.

**1.1. Classical phenomenological detonation basis**. In a previous work, the classical physics of detonation was presented and discussed (Campos et al, 2008). It assumes the basic configuration of Chapman-Jouguet mechanisms, with an interaction between shock and reaction zones. Chapman (1899) and Edmund Jouguet (1905) combine the shock and

reaction, respectively in fresh and products mixtures, developing the basic physical model of detonation. The Chapman-Jouguet one-dimensional regime (CJ point) obeys to the assumption of the entire flow to be one-dimensional and the front is assumed as a non thickness discontinuity plane, where conservation laws for shock waves (mass, momentum and energy) are applied. Based in these concepts many developments were done. Later on, it is assumed that (vd. Suceska, 2007) the detonation structure of a condensed explosive can be described, taking as example a pure homogeneous explosive, assuming shock front width, for solid and liquid explosives, followed by the reaction zone. These values prove that delay and thickness dimensions of detonation phenomena. Inside condensed heterogeneous explosives this schema is more complex. In a previous contribution (Campos et al., 2014) it was discussed the contribution, in heterogeneous explosives, of the influence of the thickness of reaction, of the further expansion and recombination of products, in its final composition (describing changes from gas to condensed, and from condensed to coalescent and expanded final species).

The presented assumptions and simplifications must be present, as dimension design basis, to evaluate the perturbation when it is introduced a sensor wire or an optical fiber (external material) to an existing non ideal explosive detonation. The dimension and position of this material, correlated to the cell structure dimension of progressed detonation, is very important to evaluate level of perturbation.

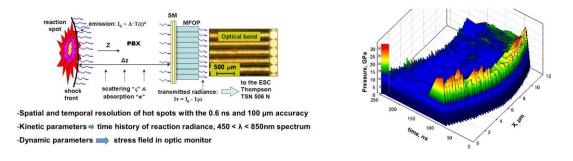
**1.2. Thermochemical prediction method**. The method, here presented, of predicting reaction path and final composition of detonation products, including solids species, uses a thermochemical computer code, named THOR, presented in previous contributions (vd. Campos et al., 2006, 2008). The development of THOR Code started some years ago (Campos, 1991, Durães et al, 1995, 1996). This code are formed by four interactive calculating clusters. These calculations require a large database (THOR DB) that contains the thermochemical properties of the reactants and possible products of reaction. Recently, optimized numerical approaches for the calculation of the Gordon and McBride (G&McB) polynomial coefficients, used in THOR to evaluate the energetic state of the detonation/combustion products, were updated (Matias et al., 2010). For each state and specie, obtained the standard molar heat capacity, enthalpy and entropy, as a function of temperature, were used to calculate the G&McB polynomial coefficients.

**1.3. Emulsion explosives** are industrially made of an aqueous solution of ammonium, sodium and calcium nitrates, emulsified with a hydrocarbon solution of oils, wax and emulsifiers, and sensitised with hollow glass or plastic microballoons. The prediction methods show the influence of the equivalence ratio of ammonium nitrate/fuel mixtures. The sensitization with microballoons is justified in order to reduce final density, allowing a self-sustained detonation regime, with a heterogeneous reaction zone driven by a lot of hot spots ignition points.

**1.4. Simplified classic physical mono-dimension model.** Generally it is always assumed that detonation process observes the Rankine-Hugoniot balance equations, based in a physical model in a P-V plane (Campos et al., 2008). This physical simplified model allow to us to define priorities, in parameter selection, for dynamic detonation measurements. Consequently it was selected the detonation velocity (measured by the recording of an event, or intensive parameter variation pulse, between two points of known position) and the detonation pressure (obtained often by the measurement of induced shock velocity in a known inert material).

**1.5. Thermal radiation of detonation**. In our particular case, the reference parameter in detonation measurement, is light radiation emitted by detonation front or by the shocked material, assuming its temperature jumping during shock compression. Consequently, it must be remembered the black-body radiation spectrum, used to evaluate the spectral intensity of radiation from a black body at temperature *T*. The black-body spectral irradiance was first derived by Max Planck (1900) and it expressed by the unit of  $I(\lambda)$ , in W. m<sup>-2</sup> .nm<sup>-1</sup>. It transduces the power emitted per unit surface area (per m<sup>2</sup>) of the black body per unit wavelength (per nanometer) (vd. Shubert, 2006, Siegel & Howell, 1992). The grey body is assumed as a "quasi" black body with a constant reduced emission. Detonation can be assumed as a grey body reaction plane, moving at fast velocities. Plaksin et al, 2009, discussing this problem, always assumed the main radiation, when it is generated in a PBX heterogeneous explosive by hot reaction spots, can be classified in two parts, one generated by compression of fresh material and the other generated by the products of detonation, and the generated radiation can transverse the fresh material, giving optical information before the arriving of detonation front.

**1.6. Original multifiber recording system.** The possibility of recording an optical signal, before arriving of detonation front, lead us to define configurations of the registration optical method. It was developed in LEDAP, during 1996-2000, an 64 optical fibbers ribbon system (250 µm of diameter each fibber) that was connected to a fast electronic streak camera (vd Mendes et al., 1996, 2001, and Plaksin, I. et. al., 1997, 1998, 2000, 2001). This method allows the simultaneous registration of several detonation parameters in one experiment (detonation wave velocity, detonation pressure, detonation front curvature, shock to detonation transition and collisions of detonation wave (Mach wave formation/ and relaxation process). Later on, the collected signals were analysed and discussed (vd. Plaksin et al., 2009). An example is presented in Figure 1, of the *"Multi-Channel Optical Analyzer instrumented with the Multi-Fiber Optical"* (Plaksin et al., 2009), presenting the scheme of configuration and discussed results of time-and-space-resolved measurements of the oscillating front



**Figure 1**. Scheme configuration of a multi optical fiber and discussed results (Plaksin et al., 2009).

**1.7. Motivation to measurement with single optical fibers.** The results of using multi fibers are clearly proved and the use of strip of fibers is well demonstrated. However, this optical system, needs a very expensive nanosecond fast streak cameras, with a very complex sweep time and trigger devices, implying very accurate and precise start chronometric systems, implying very intensive and interactive analysis, due to the more qualitative than quantitative optical results.

It is a very useful system for phenomenological analysis, but preceding problems cannot be negligible. It also must remembered that the initial systems (Mendes et al., 2001) started with multiple single fibers, connected to a fast oscilloscope, using opto-electronic transducers, allowing not only the quantitative measurement of the radiative detonation phenomena but also flyers velocities of slapper devices (Campos et al., 2002). Consequently, the present work continue the development of this study of single optical fiber measurement technique (Campos et al., 2014).

## 2. DETONATION OF EMULSION EXPLOSIVES

**2.1. THOR thermodynamic prediction** of properties of products of detonation is based in four calculating clusters (vd. Figure 2):

- the conservation equations (mass, atomic species, momentum and energy), being the thermodynamic equilibrium for  $G=G_{min}$  (P,T,x<sub>i</sub>), applying to the condensed phase the model proposed by Tanaka, 1983, or the equivalent function proposed by Gordon and McBride, 1994, or using applied EoS for solids ,Quaresma et al., 2016,

- the thermal equation of state (EoS), using generally the  $H_L$  EoS (Durães et al., 1995), - the energetic equation of state, related to the internal energy  $E = \Sigma x_i e_i(T) + \Delta e, e_i(T)$ being calculated from JANAF Thermochemical Tables, 1971, and polynomial expressions of Gordon and McBride, 1994, - the combustion condition regime, assumed like an isobar or isochor adiabatic combustion, or a Chapman-Jouguet detonation.

The structure, assumptions and simplifications were presented in previous ICT Conferences.

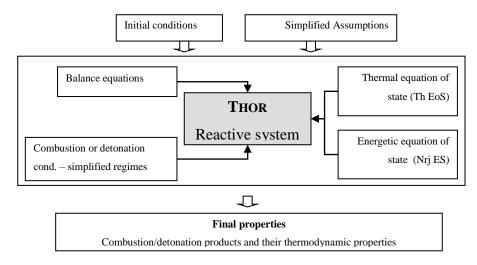


Figure 2. THOR structure of calculation.

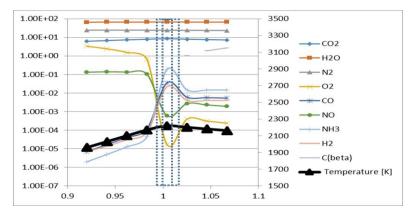
The Chapman-Jouguet detonation condition (mass, momentum and energy balances and  $dp/dV]_{S} = ((P-P_{O}) / (V-V_{O}))$  for the detonation regime, is based on the assumption that the detonation velocity D is obtained adding sound velocity  $a_{o}$  with particular velocity  $u_{p}$  (D =  $a_{o} + u_{p}$ ). Several thermodynamic codes are available and referred in bibliography. However, some of them do not include condensed species or EoS for solids or condensed species. The implementation of numerical approach to include condensed or solid species implies numerical equations and constants that are representative of behaviour of condensed materials in detonation products (vd. Quaresma et al., 2016). Most of preceding constants can be obtained directly from the CEA – Nasa Code (2007). The problem appears when there are components (from existing materials database) without their polynomial form. This is the case of NIST, JANAF Thermochemical Tables (1971) or ICT Database (2005). In order to solve this problem a new database was built – ThorDB – allowing the direct and interactive appliance of ~18000 compounds and species.

#### 2.2. Detonation of ammonium nitrate emulsion explosive

The basic reactive media is a matrix of emulsion explosive, obtained by an aqueous solution of ammonium nitrate emulsified with oil, wax and emulsifiers under the composition was presented in previous contribution (Campos et al., 2014). The numerical

calculation, using THOR code, was performed in order to establish the limits of fuel oil concentration. Assuming as initial reactant start composition [87.35 mass % of AN, 3.77 % of Fuel oil, 0.025 % of AIR and 8.84 % of Water, corresponding to an equivalence ratio of 0.92] and the products of detonation [CO2, N2, O2, H2O, NO2, CO, H2, OH, H, CH2O2, C(alfa), C(beta), C(gas), NH3, NO, O, N components] it was increase the concentration of Fuel from 3.77 until 6.25 %, keeping the mass concentration of other reactants (increasing the equivalence ratio of mixture from 0.92 to 1.07).

The detonation products composition (expressed in mole fraction percent, 1<sup>st</sup> axis) and temperature evolution (2<sup>nd</sup> axis, in K) shows clearly (vd. Figure 3) the temperature evolution as a function of fuel concentration, showing the maximum values close to the stoichiometry. Kinetic mechanisms of interaction, between these detonation products composition, was discussed previously (Campos et al, 2008). It was observed an existing transition zone, close to the stoichiometry, justified by the changes of base reference species, to calculate detonation products composition (Campos et al., 2014).



**Figure 3.** Evolution of detonation products composition and temperature of emulsion explosives, as a function of equivalence ration (function of mass percent of fuel oil in reactants).

The obtained temperture is dependent of water concentration in emulsion explosive. Similar calculation, keeping the same mass concentration of reactants without water (ANFO compositions) show an higher detonation temperature. The temperature differences between these two kinds of AN/FUEL mixtures are ~400 K, due to the water existance (vd. Campos et al., 2014).

#### 2.3. Sensitization of emulsion explosives by hollow micro balloons

Hollow micro balloons present two kinds of contribution in global self-detonation propagation. First, they decrease density of fresh material, allowing sustainability of detonation regime (observing the energy balance equation, referred before in THOR prediction procedures). Second, the existing void or low density heterogeneities, such as

inclusions, cracks, etc. or hollow micro-balloons, enhances the shock sensitivity of explosives by the formation of local additional shock dissipation that creates small regions of high temperature - called hotspots (Lee and Tarver, 1980). Herring et al., 2010. explain that chemical reactions, for heterogeneous explosives, initiated in the hotspots, emit pressure waves that merge with the lead shock and strengthen it, so that further hotspots are generated with more local intensity. This positive feedback is the principal mechanism of the shock-to-detonation transition in inhomogeneous explosives (Menikoff, 2009). While the main contribution of heterogeneities is well known, (vd. Herring et al.2010) the hierarchy and their effective phenomenology is not. In particular, since the details of the growth of reactions from the hotspots are not well understood, it is not known whether hotspots act separately or if the spatial arrangement of hotspots determines their efficacy. Spherical voids are an often-studied, common defect in explosives (Menikoff, 2009, Zukas and Walters, 1998, and the most cited Bowden and Yoffe, 1952). Experimental, theoretical, and numerical studies have sought to explain the sensitivity enhancement caused by voids and inert inclusions. Bourne and Field, 1991, reported results from shocked two-dimensional samples of gelatin or an emulsion explosive that had large cylindrical voids introduced. They observed that voids could shield their downstream neighbors from the lead shock, but that they could also effect the collapse of their neighbors by emitting shock waves when they collapsed (Ribeiro et al., 2003). Gois et al., 2001, Dattelbaum et al., 2009, shocked samples of nitromethane with randomly embedded glass beads or microballoons, observing that their presence decreased the run distance to detonation and the pressure dependence of that distance. The balloons were found to have a greater effect than the beads, and small beads were in turn more effective than large beads. Medvedev et al., 2008, conducted a theoretical analysis of emulsion explosives with microballoons that explained changes in detonation velocity with microballoon concentration, via an ignition and growth model with a constant mass burn rate per hotspot. Mendes et al., 2014, study the influence of different kind of hollow microbubbles in sensitization and progression of detonation of emulsion explosives. Bourne and Milne, 2002, experimentally and computationally, considered a hexagonal lattice of cylindrical voids in an emulsion explosive or nitromethane and observed that the reactions at the hotspots accelerated the shock relative to a comparison with water.

The study of collapsing voids under detonation can be correlated to the cavitation bubble collapse – it can be found in open bibliography many animation and models showing the formation of two parallel peripheral zones (Figure 4) generating intense shear flow and hot spot regions (vd. Blake and Gibson, 1987). The existence of many boundary conditions justify the existing jet, as a non-equilibrium zone discontinuity, projected beyond material. Experimental fast video records show clearly jet overpass the original position of bubble. The collapse of a bubbles, inside a reactive material is amore complex

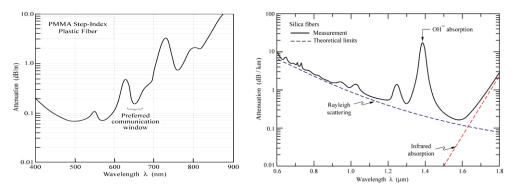
phenomena, because outside detonation front do not stop when bubble collapse, and it is observed contournent in this conditions (Ribeiro et al., 2003).



Figure 4. Typical cavitation bubble collapse simplied model simulations.

### 3. OPTICAL METROLOGY AND RESULTS

**3.1 Components and assembled device equipment.** The main set-up starts by an optic fiber, having one end inserted inside explosive material and the other end connected to a fast fiber opto-electronic analog receiver. This fiber opto-electronic analog receiver must be connected to an fast oscilloscope in order to record received signal. Two kinds of optic fiber can be used: silica and PMMA fibers. Assuming a constant core diameter value, the attenuation of PMMA and silica fibers are not at the same wavelength window (Figure 5, Schubert, 2006). According to preceding measured values of radiation, correlated to bibliographic information of detonation temperature, it is expected PMMA fiber to be more appropriated to our measures than silica fiber. However, the problems of PMMA ignition in air, under a strong shock, makes silica more appropriate for detonation measurements.



**Figure 5.** Attenuation evolution of PMMA and silica fibers, as a function of wavelength window (Schubert, 2006).

Transmitters and receivers are directly function of industry standard selected transmission wavelengths. There two wavelengths possible to our proposes: 650 and 850 nm. Two kinds of analog receivers were selected (HFBR-2505 de 650 nm e o HFBR-2406 de 850 nm from Agilent Technologies), allowing up to 125 MHz acquisition frequencies. It was built, for the presented study, an 8 independent channel opto-

electronic converter device. The receivers were assembled according fabricant instructions, tested and results were discussed (Campos et al., 2014).

**3.2. Experimental base set-up.** Two kinds of typical set-up's are used to measure detonation velocity and pressure of explosives (pressure is obtained by the induced shock pressure measurement, in a disk, cut in a material of known shock polar properties – vd Figure 8 (right)).

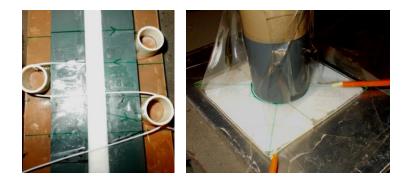


Figure 6. Example of experimental set-up to measure detonation velocity (left) and pressure (right).

## 4. BUBBLE SENSITIZATION EXPERIMENTAL SET – UP AND RESULTS

Emulsion explosive were formed by a gelatine (emulsion matrix) sensitized by low dense bubbles of 50  $\mu$ m (Expanded Dry microspheres DE, from AkzoNobel) and 5 mm (expanded polystyrene spheres - EPS) mean diameter size, as a function of the experimental proposes.

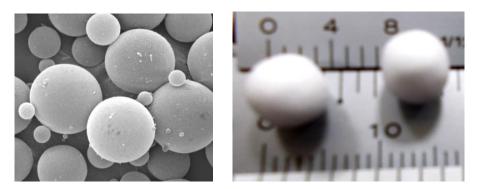
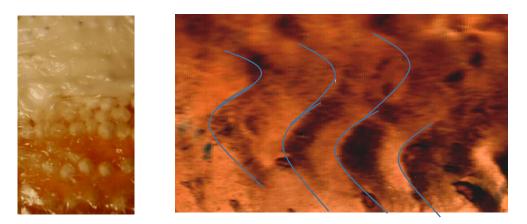


Figure 7. Expanded bubbles DE of 50  $\mu m$  and EPS of 5 mm mean diameter size.

In order to measure the influence of collapse of EPS spheres (5 mm mean diameter), two kinds of reactive configurations were tested (Figure 8), successively inserting EPS spheres directly in the gelatine (emulsion matrix) or inserting them in emulsion gelatine

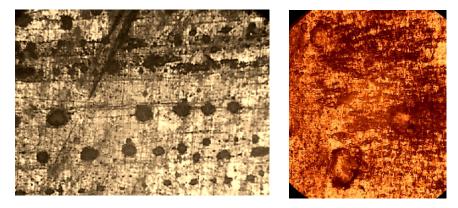
sensitized with the DE microspheres (global  $\rho$ = 1131 kg/m3 and D=5100 m/s). This last composition was used also as donor explosive. All the EPS were placed as a single layer deposed over the copper witness plate (vd Figure 8).



**Figure 8.** EPS spheres inside emulsion gelatine and inside emulsion gelatine DE sensitized (left). Printed waves (right) at copper witness plate (from EPS inserted in emulsion sensitized gelatine).

The obtained result show that EPS spheres (alone) do not sensitize gelatine in order ensure a sustainable detonation. EPS inside donnor explosive, generate printed waves in copper witness plate of a period exactly of 20 mm (4 EPS diameters) (Figure 8, right).

The printed figure at copper withness plate, of a single layer of EPS spheres inside donor emulsion explosive, show crater points (a pair of local points by each EPS sphere (Figure 9 - left). Increasing microscopic scale, it can be seen the craters, generated by a EPS sphere collapse, surrounded by small craters generated by collapses of DE microspheres (Figure 9 – right).



**Figure 9**. Single EPS layer - printed crater points (left). EPS collapse jet craters details (right). The mixture of emulsion donner explosive with EPS, having a global density of  $\rho$ =463 kg/m3, detonates with a D=~1980 m/s.

The obtained results lead us to define a typical experiment, using emulsion donor explosive with 2 silica fibers, having each one 2 EPS spheres, according to the schema presented in Figure 10. The lap of time between the optical fiber A and B gives, for a known distance, allows the calculation of the velocity of detonation. More – the instant when optical fiber B starts, shows if initial EPS sphere finish to collapse or not).

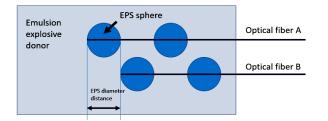


Figure 10. Experimental set-up – 2 parallel fibers with 2 EPS spheres each one.

The obtained opto-electronic record (vd. Figure 11) shows clearly the collapse and the post hot spot light region, showing that collapse of first sphere is more or less at same time of arrival detonation front at the second fiber.

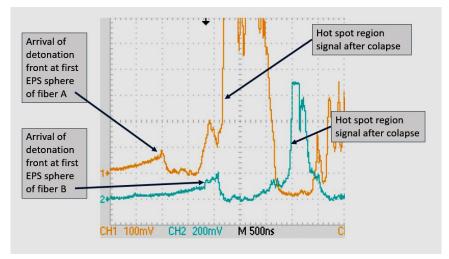


Figure 11. Opto-electronic signal of silica fibers with 2 EPS spheres.

This signals are in a excelent agreement with copper witness plate printed craters (Figure 12 - left). It is very interesting to observe the initial jet originates a reaction (proved by the second printed crater, close to the first one). This results, couple with optical fiber records, seem to induce a reaction scheme described in Figure 12 (right), where the hot spots formation generate a first progression zones. These zones, growing, interact between them, origination a third progression zone (vd Figure 12 - right). When this

progression do not attempt all steps, it is observed simple jets (visible in witness print patterns).

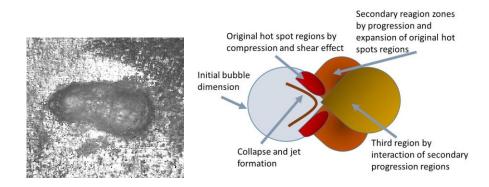


Figure 12. Copper printed crater formation by EPS collapse under donor emulsion explosive detonation (left) and explanation (right).

### 5. CONCLUSIONS.

Emulsion explosives are non-ideal explosives. Two kinds of spheres were used in experiments – expanded dry micro-spheres of 50  $\mu$ m diameter (DE) and 5 mm expanded polystyrene spheres (EPS). The EPS spheres were successively inserted in emulsion gelatin (emulsion matrix) and in emulsion gelatine sensitized with DE microspheres. Silica fibers of 60  $\mu$ m were used in optical detonation measurements. Record signals show clearly the collapse of the EPS sphere. It shows the arrival of shock detonation front in sphere, the disappearing of light during collapse of sphere, and then, the very intense light generated by hot spots. Two reaction progress regions seem to be developed. Their progression generate, sometimes, a central reaction zone, proved by the printed jet in copper witness plate.

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