COMBUSTION INSTRUMENTATION

EXPERIMENTATION AND INSTRUMENTATION

The aim here is to analyse the devices (and their implementation) used to measure and control combustion process, i.e. fuel supply and control, air supply and control, ignition, flame detection, exhaust, emissions detection, overall diagnosis, etc., for proper operation or, in the more demanding case, for investigation of special details, as the internal structure of a flame (including radicals evolution).

Depending on the application of the combustion process, e.g. to heat engines, additional variables and instruments may be of interest (e.g. engine tachometers and dynamometers).

Combustion always involves complex thermo-chemical and fluid-mechanical interactions, and thus, the instrumentation to condition, control and diagnose all the variables of interest may become rather complex. Instrumentation for research on combustion is much more sophisticated than instrumentation for proper operation of a combustor, but they are not unconnected; on one hand, instruments for routine operation were in the past research instruments, and, on the other hand, all experimental research make use of common operation instruments (e.g. flow controls), besides special sophisticated instruments (e.g. laser diagnostics). Aiming at research instrumentation, both aspects are covered.

Experiments are purposely tried events, to check some preconceptions. Instrumentation is the actual set of devices used and their way of implementation to carry out the experiment (experimentation). Experimental techniques for combustion (and in general) may be grouped according to its function of conditioning, stimulation and diagnosing the event. We envelop these techniques, exposed in chronological order of execution, with a previous planning phase and a final analysis phase.

EXPERIMENT PLANNING

Design of experiments (DOE) is a difficult creative endeavour, usually involving sophisticated tools for their implementation. Clearly defined experiment objectives (known target), a mathematical model to use for making predictions (checkable expectations), development of an ordered set of nominal trials to perform (known procedures), a feasibility study to find the weak points and choose the most reliable tools for actual implementation, redundancy in measurement, and planning for eventual malfunctions (experimental robustness), are a guarantee to success, but the key rule may be that, the simpler the experiment, the better (and try it at least twice!).

Experiments must be designed robust, i.e. with a planning insensible to uncontrolled environmental perturbations (Taguchi's experiment-quality approach). One aspect of robustness is redundancy; validation of the results is greatly enhanced by the accumulation of data that demonstrate with a high degree of confidence that the process is repetitive (without the need of lots of trials).

Instrumentation heavily depends on the objective of the test. According to its main objective, instrumentation may be aimed at:
- Normal operation of a working system (reliability and economy are the drivers)
- Experimental research in a test-rig or prototype system (accuracy is the driver).

Instrumentation equipment serves the following purposes:
- Controls, of the initial state and of the boundary conditions (sometimes depending on measurements), that may be split in:
  - Conditioning, i.e. generic controls, usually permanent and passive (not requiring power) to establish a known configuration: structure, reservoirs, piping, test section, power supply, heating, cooling, and other interfaces.

o Stimulation (or actuators), i.e. specific controls, eventful and active (requiring power or a trigger) to force some stimuli or avoid it.

- Diagnostics, i.e. sensors to measure the evolution of selected variables, including the initial state and boundary conditions. Real time diagnosis is required for real-time control of the experiment, but it is most desirable for analysis too (sending samples to a distant laboratory may still be necessary for some special biological or chemical analysis, but fluid physics has always used flow visualization for immediate diagnosis).

Combustion experiments depend on so many intermingled parameters that it has not been possible to carry out scale-model experiments, as for other aerodynamic and hydrodynamic problems, and there is a big jump from academic setups to industrial prototypes.

EXPERIMENT CONDITIONING

Besides instrumentation for diagnostics and control, combustion equipment always includes the basic infrastructure to set up the process, i.e. to condition and control the configuration: combustor body (its structure and supports), feeding ducts and exhaust, access doors and viewing windows, thermal insulation, safety devices, etc.

Experiment conditioning is so application-dependent, that no further details are presented here.

EXPERIMENT STIMULATION AND CONTROL

The key stimuli in combustion is the ignition event (usually an electric spark), but, as for typical thermo-fluid-mechanical experiments, there are other controls for the fluid supply (e.g. fuel pump, air blower), fluid flow-rate (valves), thermal state (heaters, coolers), etc.

Every stimulation requires some sensors to timeline, regulate or control its action, so that sensors should be studied prior to actuators (and the number and variety of sensors in a test-rig is much greater than that of actuators).

We do not extend here on combustion stimuli but on diagnostics, not without recalling that any sensing involves some stimuli on the test-subject (there is no measuring without perturbation).

EXPERIMENT DIAGNOSTICS

Diagnostics means the taking of measurements and samples with the aim at identifying how the system behaves in space and time. Our main aim in this survey of combustion instrumentation and experimental techniques is really here, on measurement techniques.

Diagnostics instrumentation in combustion is based on three main types of variables: flow (velocities), thermal (temperatures) and chemical (species). Most sensors (and actuators) act by direct contact with the object, but non-contact coupling (electromagnetic) is increasingly being used for sensing (and sometimes for actuators).

Besides the transducer itself, both, sensors and actuators, usually require some power and signal conditioning (digital or analogue), nowadays interfacing to a digital computer for automated operation (data acquisition and control, DAQ; digital signal processing, DSP; remote operation; etc.).

When research on combustion is implied, and not merely combustion control (with its inherent diagnostics), very sophisticated techniques are usually applied, most of the time optical (or better radiometric) in nature, since the high temperatures (>1000 K), small scales (<1 mm), and large gradients (10^6 K/m), make intrusive techniques only applicable to intake and exhaust control. The most demanding task is measuring active species within a flame (radicals and ions). Advanced diagnostics rely on spectrometric methods using laser instrumentation and advanced computer modelling codes.

EXPERIMENT ANALYSIS

The analysis of an experiment depends a lot on its purpose, it is very different to check that a certain magnitude is within allowable bounds (e.g. to check the concentration of CO in a vehicle exhaust), that to investigate the formation of soot.

Redundancy is so important to guarantee data validation, that a big problem in data analysis is the reduction of the overwhelming amount of data taken; statistical analysis helps a lot, but the most important is to have a parametric model to fit the data to, i.e. data fitting to models and not just to generic curves and surfaces.

However, as we said before, we deal here only with instrumentation, and leave actual data acquisition and analysis aside.

EXPERIMENTAL TECHNIQUES

Experimental techniques may be classified as above in configuration setups, stimuli (actuators: flow inducers, valves, spark plugs), and diagnostics (sensors: flow, temperature, concentration); it should be recalled that besides the sensor or actuator itself (the transducer), some power supply and conditioning circuit is usually involved.

For combustion instrumentation, experimental techniques could also be classified according to function as: fuel-related, air-related, fuel/air-related, safety-related, emissions-related, and flame-structure-related (only for combustion research), or according to the setup: operating installation, standard test-rig, specially built research set-up. For combustion experimentation, special burners have been devised, starting from the now-standard axisymmetric Bunsen premixed burner in 1855, Wolfhard flat-flame premixed burner in 1939, Wolfhard-Parker twin-slot diffusion burner in 1949, Pandya-Weimberg opposite-jets diffusion burner in 1963, etc.

The traditional classification of experimental techniques is, however, according to the type of variable being measured or controlled: temperature measurement, flow rate or velocity, pressure, composition, etc., sometimes grouped in physical and chemical techniques. But nowadays, and particularly in combustion research, the same experimental technique is used to measure several magnitudes at once, as

when Ràmàn scattering is used to measure composition, concentration and temperature. Here we follow the traditional approach, but a separate presentation of general non-intrusive diagnostics is included below.

Time measurement

Many experiments on combustion deal basically with steady states, but timing is important in any case (all steady states start and end). Timing in combustion is not as critical as in other fields, except when analysing the initial phase of the ignition process.

Geometry, level, edges and particle measurement

Fixed geometry is measured before or after the experiment. Low varying geometries as liquid level, solid-fuel borders, actuator position (for stimuli or for sensor location), and so on, are measured with potentiometers, capacitive sensors, ultrasounds, etc. More subtle edges, as flame fronts, smoke plumes, or the multiplicity of particle sizes (fuel sprays, tracers, soot) usually require optical techniques.

Thermometry

The contact thermometers most used in combustion are thermocouples, thermistors (negative temperature coefficient resistors, NTC) and metallic resistance thermometer devices (RTD), in that order. More advanced non-contact thermometers (sometimes named pyrometers) are dealt with below under Optical techniques.

Velocimetry and flow rating

Flow meters may be global, to know the overall mass flow rate or volume flow rate, or local to know the velocity field. In the first case, tank weighting or level change in condense fuels, or general flow meters as rotameters, calibrated nozzles and diaphragms, turbine-meters and other more sophisticated methods as thermal capacity and Coriolis effect devices, may be used.

Measuring the velocity field is much more cumbersome at least because of the amount of data implied, and sophisticated methods are used, as explained below.

Piezometry

Pressure measurement (piezometry) is not a difficult problem in combustion except in reciprocating engines, where rapid changes and large pressures are involved, and there piezoelectric sensors are used (quartz-crystal transducers develop an electrical charge when compressed; they are bored into the head of the cylinder or adapted within a modified spark-plug). Piezoresistive transducers are semiconductors (doped silicon) that change their electrical resistence upon compression, and are used to follow the high-frequency pulsation at the intake and exhaust ports in reciprocating engines. For small pressure differences the best are silicon-chip capacitance sensors.

Chemical analysis

Different stages in a combustion processes may demand chemical analysis, i.e. qualitative and quantitative finding of the composition in a mixture: intake (fuel and air analysis), inside (radicals formed

for kinetic studies), and exhaust (emissions). The main components in combustion processes are: N_2, O_2, fuel, CO_2 and H_2O; trace components are: CO, NO_x, OH, O, H, CHO, OH_2, e^-, OH^-, CHO^+, etc. In general, analytical techniques may be grouped as:

- Chemical methods of analysis: characteristic reactions, selective absorption, electrochemical techniques, etc.
- Physical methods of chemical analysis, ranging from electrical, thermal or optical, to the most sophisticated spectroscopic techniques.

Many times, a sample of the mixture is analysed off-line and discarded, often through a separation process of chromatography, but most advanced analytical techniques are non-intrusive and on-line.

Chemical sampling may be intrusive, through a quartz tube of some 1 mm or less, followed by chemical analysis (using Orsat selective absorbers, or gas chromatography, or mass spectrometry, or selective solid-electrolyte conductometry), or non-intrusive sampling (radiometric) that may be passive or active, classical or quantum. Sometimes a special chemical type, the electrochemical one, is singled out, since it is widely used to directly measure concentrations of many different gases (CO, NO_2, SO_2, H_2S, HCl, but only in the ppm-range). Before gas chromatography took over in mid XX c., and later radiometry and mass spectrometry, the standard method in exhaust analysis was developed by Orsat in late XIX c. After filtering solid particles and dehumidifying the exhaust sample, the amount of CO_2 was first measured by volume subtraction after passing the gas through a NaOH solution; afterwards, non-saturated hydrocarbons were removed by a KOH and pyrogalic-acid solution, oxygen was removed by a $NH_4Cl/CuCl$ solution, and CO by H_2SO_2.

Note that composition ranges may be different not only for different species, main or trace, but for different function; e.g. for CO-toxicity the maximum human exposure is 50 ppm, whereas for CO-combustion the minimum concentration for ignition is 12.5% (the instruments are different).

Experimental techniques are described below; first those related to the overall control and safety of the process, and later on the more sophisticated diagnostic techniques.

CONTROLS AND SAFETY

The general goal of combustion instrumentation is to procure a safe, energy-efficient and emission-free, combustion process for the intended use: operation or research. To that purpose, electrical, pneumatic and hydraulic actuators, automatically or manually operated, are used to control the process.

FUEL AND AIR SUPPLY

Fuel supply

Most fluid fuels are already available at a supply pressure (either bottled or piped). If not, some pumping should be implemented, as in vehicle engines and coal-fires burners. The usual fuel flowrate control is a solenoid valve (a needle electrovalve). Heating power control is based on fuel-supply control, either on/off or modular. Related to fuel presence and the possibility of uncontrolled combustion is the 'explosimeter', a fuel-gas sensor, usually based on the rapid oxidation of the fuel at room temperature in

the surface of a catalyst (a Pt-wire that gets hot and changes its electrical resistance in the presence of a reactive atmosphere).

Commercial liquid fuels require pumping and filtration, and sometimes also heating systems; solid fuels usually require more handling and preparation; neither of those additional systems are dealt with here,

Air supply

The air supplier may be a variable speed fan (the speed is varied by changing the voltage, the frequency or the wave profile), the engine suction itself, or a natural draught induced by the fuel supply. Manometers (diaphragm, piezoelectric) may be part of the diagnostics. Air flow metering with integrated temperature sensor is fed to the electronic control unit (ECU), if any. Air must be supplied not only for combustion, but for purging purposes to bring the system to known safe conditions.

Air/fuel ratio

Good control of air/fuel ratio is important in premixed flames for efficiency and polution-avoidance, and has become critical for operation of exhaust catalysts. A simple fix regulation may produce unwanted air/fuel ratios due to fluctuations in fuel and/or air supply (pressure or composition), or changes in load condition (e.g. secondary air in a burner does not follows fuel flow rate); for that reason, an O_2-detector in the exhaust is used to control the air/fuel ratio (an oxygen sensor is chosen because x_{O2} monotonically grows. with A-A_0, quasi-linearly (x_{O2}=0..0.1 from λ=1..2), whereas for instance x_{CO2} decreases parabolically with A-A_0, and on both sides! of λ=1, besides the sensor being more expensive).

Several O_2-detectors have been developed since the old λ-probe (Saab/Bosh-1977) that revolutionised electronic ignition and injection control in Otto engines, where stoichiometry is now maintained to λ=1.00±0.01 (domestic water-heaters work with 10..50% excess air). They are electrochemical cells yielding a voltage depending on the difference in oxygen concentration (O^{2-} really) between the exhaust and the ambient air (highly non-linear emf, as seen in Fig. 1), through the electrolyte (a ceramic sheet of ZrO_2). The electrodes are gas-permeable platinum layers. They only work when hot (>300 ºC, that is why they were placed at the exhaust manifold); besides, the output depends on the operating temperature. Since 1990 all λ-probes (in the front and at the rear of the catalyser) are heated to work also when idle and at part throttle. Other resistive semiconductor probes have been tried without too much success (TiO_2, SnO_2).
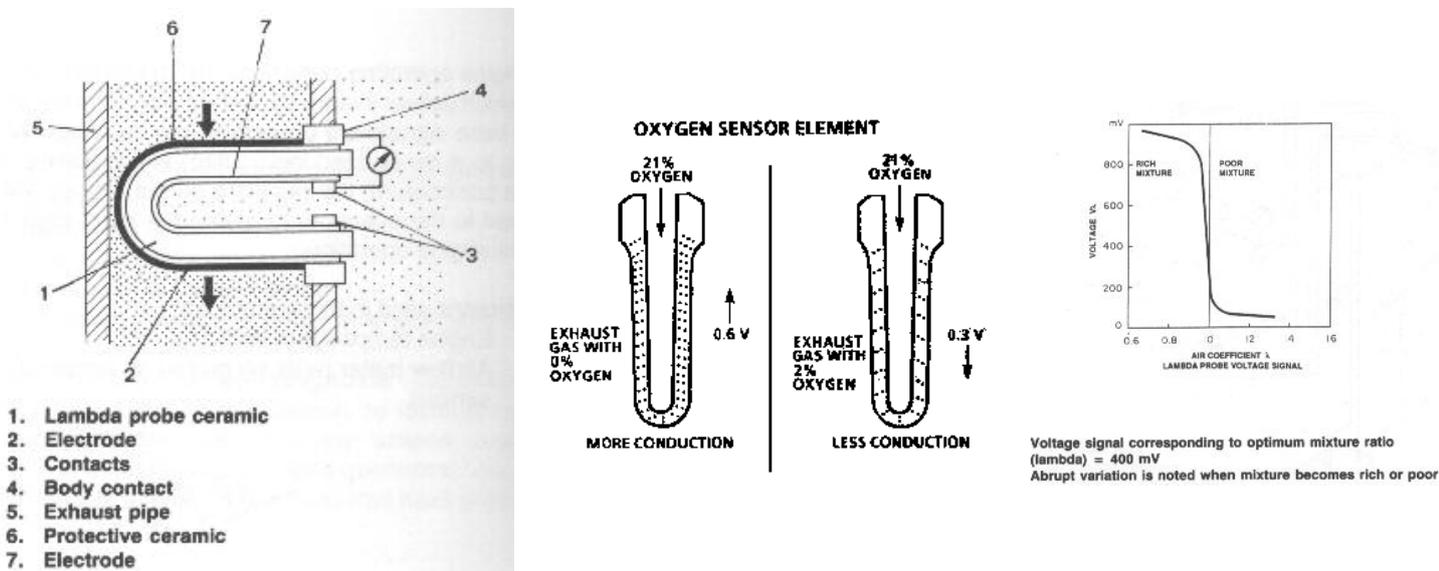
Fig. 1. Functional details of a lambda probe sensor.

1. Lambda probe ceramic
2. Electrode
3. Contacts
4. Body contact
5. Exhaust pipe
6. Protective ceramic
7. Electrode

FLAME DETECTORS

Different types of fire alarms and flame detectors exist; some are good for close-proximity detection and others for overall surveillance (indoors or outdoors):

- Thermal. A probe that changes with temperature (bends a bimetallic strip, bends a burdom-type vapour-pressure phial, etc.). They are pasive devices (no need of power), simple, and fail-safe. Just for safety, a wire that melts may be used to break a contact.

- Thermoelectric. An emf is generated that may power a solenoid to keep both, the main and the pilot fuel supply (a manual start is needed, usually the user holding a push-button while the thermocouple gets hot). It was the standard for small appliances as home water heaters.

- Ionisation. Measures the change in electrical conductivity of air through a flame (a flame is a plasma with some 1 ppm charged-particles). It is very quick and can be automated, being the method presently used in home appliances, in spite of the fact that it is not passive (it requires power).

- Infra-red emission (IR with $\lambda_{CO2}=2.7$ μm or better $\lambda_{CO2}=4.1..4.6$ μm). Solar and lamp radiation may cause false alarms. It is better to use several wavelengths to discard solar radiation reflections. All radiometric methods are expensive, so they are only used in large equipment.

- Ultra-violet emission (UV with $\lambda=0.2..0.3$ μm). Lightning and arc-welding may cause false alarms. A combination of UV/IR sensors is better.

- Chemiluminescence. For known flame types, a photomultiplier tube with an spectral filter may sense characteristic radiation emissions.

EMISSION DETECTORS

- Gas leak detectors and explosimeters. Usually based on the electrical resistance variation of a platinum wire, due to the temperature increase caused by catalytic oxidation of the fuel-air mixture. Portable system with field replaceable measuring cells are in the market capable of sensing minute concentrations of natural gas, butane and propane, either along pipes and combustor (gas leak), or in the ambient (explosimeters). Sometimes, to avoid explosions, it is not enough to have a quantity less than the LEL in a closed room, since very light fuels like H_2 or very heavy gas fuels as diethyl ether ($C_4H_{10}O$) will stratify a lot.

- Flue gas emission analysers. Usually based on selective radiation emission or absorption, or on electrochemical cells. Oxygen in the exhaust is measured to know the air/fuel ratio used, and the most used method is the zirconium-oxide cell explained above. Other O_2 and NO_x sensors are based on wet electrochemical cells, consisting of coated electrodes (sensing, reference, and sometimes a counter electrode too) and a small volume of an acid or base solution (the electrolyte); gases diffuse through orifices on the sensing face to the porous sensing electrode, reaching the electrolyte, and generating a very small electrical current proportional to gas concentration; their response time is low, and they have a consumable counter electrode. Portable system with field replaceable measuring cells are in the market capable of measuring at once flue velocity (0..50 m/s), H_2O (0..30%), O_2 (0..25%), CO (0..10000 ppm), NO (0..1000 ppm), NO_2 (0..1000 ppm), SO_2 (0..1000 ppm), differential temperature (0..1000 K) and CO_2 (0..25%). Excess air and energy efficiency can be easily computed from those measurements.
- Smoke and particulate analysers. Usually based on light transmission, scattering or reflection, or by β-radiation absorption, or by the tribo-electric measuring principle (the tribological probe measures the charge on the particles that strike a metallic rod, which depends on the flow velocity and the concentration of the dust in the flue gas).

Detector must be calibrated from time to time (according to required standards), using certified concentrations of test gases.

FLAME DIAGNOSTICS

The most conspicuous feature of combustion to analyse is the visible flame. Most flames flicker, and it is difficult to have a steady flame to look at: it must be protected from minute air-drafts and fuel-supply perturbations.

Flames tend to get anchored to solid edges because there are ample ranges in temperature and velocities nearby, where stabilisation may take place. When flames cannot stabilise on the rim of the burner, flame holders (flow gutters in I-, V- or H-shape) are placed downstream of the fuel injectors, as in gas turbine combustion chambers where flames are stabilised in streams up to 100 m/s).

Although contact heat devices may be used for flame detection and analysis as said above, optical (non-intrusive) techniques are preferred, either based on classical optical effects (ray geometry and photometry, as intensity absorption, particles, edges, shadowgraphy, interferometry, moiré, speckle, polarisation, etc.), or based on quantum effects (spectral intensity analysis: the frequency identifies the species, and the intensity gives their concentration). A generic overview of optical diagnostic techniques is given below, after flow and chemical instrumentation is presented.

Laminar premixed flame experiments are the standard way to get validation data for combustion chemistry models (e.g. for pollutants and soot formation).

EXHAUST CONTROL

Flow rates are controlled at the intake by virtue of fuel and oxidiser supply systems; the aim here is how to provide a safe exhaust for the operation of combustors. The usual procedure is to get rid of the exhaust gases through a chimney to the atmosphere, far enough to minimise nuisance and danger (overheating, asphyxia, intoxication, deflagration). Sometimes the exhaust is cleaned to minimise emissions, but we focus here on fuel releases.

Normal practice to cope with unavoidable fuel releases through the exhaust, due to lack of appropriate ignition or unexpected extinction, is to force fresh air for a while to dilute the mixture below its ignition limit, before any other trial is performed.

A better procedure in experimental setups is to pass the exhaust through a pilot flame to guarantee that explosive mixtures do not build up.

FLOW MEASUREMENT

Flow measurement is used for accounting and for control. Basic fluid-mechanical instrumentation, as used in other applications to measure liquid tank level, flow velocity fields, turbulence level, pressure, particle size and distribution, etc., are also used in combustion instrumentation: positive-displacement counters (as the domestic gas meter), differential-pressure meters (diaphragm and venturi meters), turbine flow-meters, hot-wire anemometers (HWA), laser doppler velocimetry (LDV), particle image velocimetry (PIV), ultrasonic velocimetry, etc. The most precise flow-rate sensors are the heater-based for gases (thermal capacity) and the Coriolis-based for liquids. Advanced optical techniques are covered below.

As said before, there is an emphasis on particle characterisation in combustion diagnostics, both because of the importance of soot formation and particulate emissions, and because of the importance of spray combustion, what is related to flow measurement (e.g. PIV may be used to measure single-fluid flow by adding tracer particles, or to measure particle velocities in proper two-phase flows: fuel sprays and exhaust soot). Modern high-speed digital cameras are used nowadays to better analyse two-phase flows.

HOT-WIRE ANEMOMETRY (HWA)

In this technique, a very small metal wire is heated and the power dissipated and temperature reached are measured (the electrical resistance depends on temperature); as the amount of cooling depends on the convective velocity of the fluid in which it is immersed, proper calibration provides a measure of fluid velocity in terms of power supply for a constant temperature difference, or in terms of temperature difference for a constant power supply.

The wire is made of platinum or wolfram, and is very delicate, with diameter in the range 0.5..5 μm and lengths from 0.1..2 mm, making HWA only usable for relatively clean gases like ambient air (that is why they are usually named anemometers). Because of the geometry, a single hot wire only yields one component of the velocity field, so that multi-sensor probes are used for three-dimensional fields. Notice that by just measuring the electrical resistance, hot-wires are also thermometers.

PARTICLE IMAGE VELOCIMETRY (PIV)

In this technique the fluid is seeded with small particles (of order of 10 μm or larger), of the same density as the fluid, in an amount such that there are as many as possible without overlapping in the image.

In two-dimensional PIV, a thin powerful sheet of light is shed on the object (preferably by a pulsed laser, to avoid heating by absorption), and the light scattered by the individual particles is focused in an image plane, where their positions are tracked to compute velocity vectors from consecutive images. Stereographic and photogrametric and holographic techniques are being used to provide direct three-dimensional measurements.

This technique best applies to quasi-steady, quasi-planar flows. Refractive index gradients could distort the image. Collimated light may be used to avoid parallax distortions.

SPECKLE VELOCIMETRY (SV)

Similar to PIV but with many more and smaller particles (of order of 1 μm) whose individual images overlap, forming speckles (a small granulation) without any meaning to direct observation; but if the speckle pattern is illuminated with a coherent light beam, Young interference fringes appear, proportional to the average particle separation, that, if subtracted from a reference speckle image, gives the apparent velocity field.

LASER DOPPLER VELOCIMETRY (LDV)

In this technique, the time tracers take to cross along consecutive fringes formed at the intersection of two nearly-parallel beams from a laser, is measured in a scattered-light detector. As this simple setup only yields the speed modulus in one direction (along the in-plane counter-bisect), timing shifts and multiple laser beam-pairs are used to measure the whole velocity vector at a point.

In spite of its fast and precise response and non-intrusive character (most of the times there is no need to add the tracers, as common fluids as air and water always carry fine particles in suspension), LDV has the strong handicap of being a one-point sampling technique (i.e. zero-dimensional, when most velocity fields are three-dimensional).

TEMPERATURE MEASUREMENT

Combustion thermometry usually focuses onto the gas phase; measurement of surface temperatures in conducts and combustor walls is also important, but not so demanding (except perhaps at moving surfaces as gas-turbine rotor blades). Measuring high temperatures in a gas is a difficult subject since thermal conductance from gas to probe is poor, heat conduction through the lead wires and the metallic sheath is important, and radiative coupling between probe and walls is high. Moreover, in thin, fluctuating, reacting, hot regions (as within a flame), there is response-time problem, probe micro-vibrations problems, materials-resistance problems, and basic non-equilibrium ill-defined-temperature problems.

For high spatial and temporal resolutions, fine-gauge thermocouples are the best; platinum-resistance probes (Pt-100) are too large, and thermistors (NTC) have a small temperature range. Accuracy and

response time are key issues. As for any contact thermometer, one assumes that thermal equilibrium of the sensor and the object is established, but with low-conducting gases at high temperatures, losses through the probe support and to the walls are large and only a steady state is reached. The response time, of the order of 1 s for 1 mm size thermocouples, may be lowered down to 10^{-2} s for the smallest 10 μm wide thermocouples (at the expense of durability and accuracy). For high temperature measurement in combustion, thermocouples must be sheath with SiO_2 to avoid metal oxidation and catalytic effects.

But intrusive thermometers as the thermocouple cannot provide whole field temperatures in fluctuating flows, as in reciprocating internal combustion engines; knowledge of the spatial distribution of gas temperature prior to ignition is needed for modelling engine combustion with stratified load and/or exhaust gas recirculation, since large temperature inhomogeneities are involved.

Non-intrusive radiometric temperature measurement is preferred for advanced multidimensional analysis, the physical probes been used for calibration. Thermometry based on gas density measured by the refractive index is easy, but resolution is low at high temperatures. Infrared thermometry based on CO_2 and H_2O bands, an ultraviolet radiometry at the 0.309 μm band due to OH are also used.

ANALYTICAL TECHNIQUES

Analytical techniques refer here to chemical-composition measurement. A short description of some of the traditional analytical techniques used in combustion follows, with the more advanced optical diagnostics being covered under the *Optical Diagnostic Techniques* heading, below. Sometimes the main products in the combustion of organic matter are separated before further exhaust analysis; water vapour is traditionally absorbed and weighted in phosphorus-oxide, $P_4O_{10}(s)+6H_2O(v,l)=4H_3PO_4(s)$, and carbon dioxide in sodium hydroxide, $NaOH(s)+CO_2(g)=NaHCO_3(s)$.

GAS CHROMATOGRAPHY

Chromatographic techniques (gas or liquid) are based, as for other means of mixture separation, in the natural segregation of species between two immiscible phases. In gas chromatography, a small sample is diluted in an inert gas carrier (He, H_2, Ar, N_2...) and forced to flow through a porous media or a stationary liquid, what introduces a selective speed-lag (dependent on size and affinity of species to plug material) that allows selective elution for collection or discarding, i.e. a fractioning, as when a drop of mixed dyes spread over a tissue (the origin of chromatography).

After separation, traditional chemical analysis may be performed, but, with proper calibration with known samples at standard temperature and pressure, a catalogue of retention index can be prepared, and qualitative (just by a look-at table) and quantitative (e.g. by light absorption) measures can be obtained.

Taking physical samples in combustion processes is not simple because the probe may induce catalytic reactions at those high temperatures. The probe is typically a narrow SiO_2 tube (down to 0.1 mm in diameter have been achieved)

MASS SPECTROMETRY

In mass spectrometry, a small sample is put into vacuum and bombarded with an electron beam to produce a stream of charged fragments in different proportions and mass-to-charge ratios (ion source generation), that, when subjected to a magnetic or electric field, produces a proportional deviation, i.e. a fractioning of different mass-to-charge ratios, with the intensity at the target being proportional to concentration.

(RADIATION) SPECTROMETRY

Note. Spectrometry is understood to refer only to electromagnetic spectrometry, i.e. spectro-photometry or spectro-radiometry; no mass spectrometry. Sometimes, spectrometry is also used as a generic name for all quantum-optical techniques.

Three types of radiation effects are used in spectrometry: emission, absorption, and scattering. Either the wavelength, λ, or the wavenumber $k=1/\lambda$, or the frequency, $v=c/\lambda$ (with c the speed of light), is measured (only the frequency is independent of the refractive index). Details are covered below.

Although high-resolution microwave and infrared spectroscopy are applied to optical diagnostics in combustion, visual laser spectroscopy is the most common advanced technique for flame analysis: Raman spectroscopy, Coherent Anti Raman Spectrometry (CARS), Laser Induced Fluorescence (LIF).

OPTICAL DIAGNOSTIC TECHNIQUES

APPLICATIONS

Optical diagnostic techniques are commonly used in all kinds of fluid diagnostics, not only in combustion, but they are specially critical to flame diagnostics because of the hardship of the environment and small size.

Optical diagnostic techniques may serve to study:
- Geometry: fluid boundaries, moving objects, moving fluid-interface fronts, etc.
- Particle size and distribution: nephelometry, Rayleigh scattering.
- Velocimetry: particle image velocimetry (PIV), laser doppler velocimetry.
- Thermometry: disappearing-filament pyrometry, sodium-line reversal.
- Concentrations: refractometry (schlieren, moiré).

The fact that most novel diagnostic techniques, both for physical analysis and for chemical analysis, are based on electromagnetic radiations, is because of the following advantages of optical systems:
- They are non-intrusive
- Whole field sensing possibility
- Immediate qualitative diagnosis with possibility of quantitative analysis
- Digital video processing automation possibility

Optical diagnostic techniques naturally started by photographic recording of what the observer's eye was watching; it first developed along classical optics, but most applications nowadays are based on quantum optical effects (spectrometry).

Optical techniques have some disadvantages, however. First of all, the radiation coming from the object may go through a complex path (windows, intermediate fluids, environmental air, and so on), with optical properties not well known or controllable. Second, the optical setups used to be most delicate and expensive, but introduction of fibre-optics and afocal optics has alleviated some of these problems. Third, optical techniques need to be calibrated in a similar configuration (a laminar premixed flame is usually used as a standard).

FUNDAMENTALS

Electromagnetic radiation can best be understood at the microscopic level in the particle-sense (from the wave/particle dualism), as a photon gas; a photon has zero mass at rest and an energy proportional to its frequency, $E=h\nu$. The interaction of electromagnetic radiation with a material substance may be small, giving rise to thermal interactions (energy redistribution without molecular breakings; only translational, rotational, vibrational and electronic energy level rearrangement), or the interaction may be stronger (giving rise to loss of electrons in the molecule, i.e. ionisation and chemical reactions, or even causing nuclear reactions).

Energy jumps in the interaction matter-radiation are widely separated in the spectra and are studied independently (the experimental techniques and equipment are different): nuclear transitions yield γ-rays independently of atomic and molecular structure, atomic (electronic) transitions yield X-ray and UV-ray (fine spectral lines) independently of nuclear and molecular structure, and molecular transitions (vibration and rotation) yield visible and IR bands (thick lines) independently of nuclear and atomic structure (e.g. ions radiate the same as neutral atoms in the visible and IR).

Radiation is constantly emitted by matter at equilibrium due to the continuous energy transitions associated to the prevailing temperature. A system out of equilibrium may emit additional radiation depending on the reactions taking place inside. Most optical techniques, however, rely on the analysis of stimulated emission due to an apply coherent radiation source (laser), although the stimuli may also be a diffuse light, an electron beam, an electric or magnetic field, a spark, an arc, a flame, etc.).

Stimulated radiation due to a coherent radiation source may cause:
- Optical resonance when the source frequency coincides with some natural frequency of the system, and the molecules radiate at the same frequency and in all directions, without lag.
- Chemiluminescence emission, when the source frequency coincides with some natural frequency of the system, and the molecules radiate at a lower frequency and in all directions, with a lag that may be $>10^{-4}$ s (and then it is called phosphorescence), or $<10^{-4}$ s (and it is called fluorescence).
- Elastic scattering (Rayleigh scattering), when the system is excited with any frequency and emits at the same frequency with a small amplitude (some 10^{-3} the intensity of the source, larger at larger frequencies), and non-isotropic but lobular and polarised.

- Inelastic scattering (Raman scattering, 1928), when the system is excited with any frequency and emits at the several different frequencies with a very small amplitude (some 10^{-6} the intensity of the source) and non-isotropic but lobular and polarised. The frequency differences with the source depends on the type of molecules, and they may be below the exciting one (called Stokes lines) or above it (called anti-Stokes lines).

CLASSIFICATION

Three main items must be considered in optical diagnostics: the object, the radiation source, and the radiation sensor.

OBJECT TYPES

According to the object, optical diagnostic techniques may be classified in two main groups:
- Heterogeneous systems, basically particles (isolated or forming a mist) in a fluid matrix, and fluid boundaries.
- Homogeneous systems, basically smoothly changing fluid volumes.

RADIATION SOURCES

According to the radiation source, optical diagnostic techniques may be classified as follows:
- Own radiation emission by the object (thermal radiation and chemiluminescent radiation)
- External radiation source
  - Visible white light, usually from incandescent lamps, but sometimes from special luminaries (Nernst, Globar, Hg, Na).
  - Monochromatic light, mostly from lasers (monochromators with white light yield much lower spectral power). Although it depends on temperature, typical He-Ne lasers have 10 mW and λ=0.633 μm (the iodine stabilized Helium-Neon laser has λ=0.6329914 μm), cheap diode lasers may have 10 mW and λ~0.8 μm (AlGaAs) or λ=0.65 μm (AlGaInP), powerful NdYAG lasers have 100 W and λ=1.064 μm (invisible near infrared), and the most powerful, CO_2 lasers have >1 kW and λ=10.600 μm (invisible far infrared). Excimer lasers are pulsed gas discharge lasers which produce optical output in the ultraviolet (the actual wavelength can be changed by changing the gas mixture).

DETECTOR TYPES

According to what is detected (image), optical diagnostic techniques may be classified as follows:
- Radiation origin: object own emission (by its temperature or internal processes), source own emission outlined by the object, source reflection on the object and surroundings (typical eye-sight), source refraction along the object, object emission stimulated by the external source (dispersion, luminescence).
- Image information: ray intensity (bright, contrast, colour and saturation of typical eye-sight imaging), ray deflection refractometry (moiré, schlieren, shadowgraphy), interferometry (radiation phase measurement), and holography (intensity and phase measurement).
- Spatial dimensionality: point sampling, line of sight, bidimensional, tridimensional.
- Spectral dimensionality: white light, monochromatic.
- Spectral frequency band: visible, infrared, ultraviolet, microwave, etc.

- Image sensor: photochemical or photoelectric. The traditional chemical photogram (Gr. *photo*, light, *gram*, message) was much used since its development by Daguerre in 1839 until the end of the XX c., that was substituted by digital photography. Photoelectric sensors may be onedimensional (photodiodes, photomultipliers, field-effect transistors) or twodimensional (vidicon tubes, charge coupled devices, CCD, are the most used). Image sensors (eye, photographic plate, CCD) are two-dimensional.

RADIATION EMISSION AND RADIATION ABSORPTION

The own emitted radiation from a sample can be used to diagnose hot or excited objects as flames. The radiation absorbed by an object from a known wide-band light, can be used to diagnose cold and hot objects. Molecules absorb light only at certain characteristic wavelengths called its spectrum. At infrared wavelengths, the spectrum results from vibrations of the atoms in the molecule, while at visible and ultraviolet wavelengths the spectrum is caused by the electrons orbiting the molecule. The spectrum can be calculated from quantum mechanics or measured in the laboratory

Condense matter yields a continuous spectrum dependent on its temperature (it approaches blackbody radiation for dielectric materials like soot), but gases yield clearly separated spectral bands (they would require optical sizes of many metres to approach blackbody radiation).

Radiation emission may be used to measure temperature or to analyse species and concentrations. If the object is cold, it can be raised to high temperatures by contact with a hot wire or by exposure to a flame (e.g. by dipping an inert platinum wire in the solution or powder sample and putting it on a Bunsen flame).

Light is often said to have a colour temperature (not the real temperature of the emitting body, but the one of a blackbody that would give the same colour sensation); the colour temperature of some common light sources are: 2000 K for a candle flame, 3000 for an incandescent lamp, 4000 K for a carbon-arc or a magnesium-flash light, 6000 for direct sunlight and 10 000 K for open sky.

The colloquial usage of "red hot," "white hot," and so on, is part of the colour sequence black, red, orange, yellow, white, and bluish white, seen as an object is heated to successively higher temperatures.

RADIATION SCATTERING

When electromagnetic radiation impinges on a material particle, some part is re-radiated at different angles from incidence (what is known as scatter). Scattering radiation depends on the size of the particle and radiation-source wavelength. The interaction may be described as follows:
- For large particles (say >1 mm that is the typical minimum width of a collimated beam, very much larger than the source wavelength, λ), some part of it is absorbed, some part is reflected (both specularly and diffusively), and the rest is refracted according to the transmissivity of the particle. The geometrical optics approximation applies (i.e. ray tracing), without any influence of λ.
- For medium-size particles (say from 1 μm to 1 mm) the bending of outlining light-rays, i.e. edge diffraction, has to be considered. Diffraction is the name given to any deviation from the laws of

geometrical optics; it was modelled by Fresnel in 1818. Diffraction in the far zone, the Fraunhofer approximation, is good enough in this size range, instead of the full Mie scattering theory.

- For small particles (say 1 μm, comparable to the wavelength of the light used, λ), some part of the incident radiation is scattered in a non-isotropic, non-symmetric lobular pattern with intensity independent of frequency, larger to the foreword, what is known as Mie scattering. Tyndall effect is due to Mie scattering, as well as other whitish effects are: clouds, dispersion opalescence, foams, mists, and dust haze (they scatter all wavelengths equally).

- For very small particles (<1/10 μm, or better λ/10), some part of the incident radiation is scattered in a non-isotropic, symmetric lobular pattern with intensity proportional to the fourth power of the frequency, what is known as Rayleigh scattering. The blue colour of sky and the reddish colours at sunset and sunrise result from Rayleigh scattering. Scattered light intensity depends on species concentration and on species cross-section, so that if one is known, the other may be found (this simple technique is widely used in combustion, and named Rayleigh line scattering, RLS). Most scattered radiation has the same frequency as the incoming radiation (Rayleigh scattering), but some parts have different frequencies and are given the specific name of Raman scattering.

Radiation dispersed from a monochromatic source is the best mean to diagnose flames. Scattered radiation is fractioned in a tuneable monochromator (a prism or better a grating) and focused onto a detector (visual or infrared), for measuring the actual species (based on wavelength) and its concentration (based on intensity detection).

HOLOGRAPHY

Holography, developed by Gabor in 1947, is the method for recording and reconstruction the whole information of an optical scene (intensity and phase of the light waves). In traditional imaging, light scattered by the object in the scene is focused by a lens onto a 2D-plate, building a planar projection of the scene proportional to light intensity without any phase information. For reconstruction, i.e. for viewing, we just look at the image with any light and we see a 2D-frozen image of the original scene, irrespective of the eye position.

To build a hologram, the procedure is to split a coherent light beam, shine one part on the scene, and store the interference of the reference beam with the beam scattered by the object. For reconstruction, i.e. for viewing, the recorded hologram is places in a beam of the same coherent light used for recording, what sheds a virtual replica of the initial scene 'as if the objects were there'; the recording plate acts like a window through which the observer can peep through.

Holography requires a large temporal coherence of the source light (a very monochromatic laser) to produce sharp interferences, and large spatial coherence of the source light (a very planar beam) to enable reconstruction by any similar laser and not necessarily the recording one. Holography can be used to visualise objects and particles, or to produce interferograms.

TOMOGRAPHY

Tomography is an image synthesis technique based on building a three-dimensional visualisation by juxtaposition of many two-dimensional adjacent images (sectional views). Tomography is used in flame structure research to avoid the accumulated contribution from all the scene; flame sheets are diagnosed at a time, and later the three-dimensional structure is built with computer aid.

INTERFEROMETRY

Interferometry is the measuring of the refractive index field of an object phase by means of the interference pattern formed when a reference light beam is combined with the beam going through the object. Both beams have to be spatial and time coherent, and that is why a single laser beam, split in two, is used.

The refractive index field is directly related to the density of the fluid object, what depends on temperature and composition, allowing to resolve one of them if the other is known.

Many different types of interferometers have been developed, most of them requiring an optical bench (a very rigid setup, isolated from external noise) to work.

SPECKLE INTERFEROMETRY (SI)

A coherent light is split in two collimated beams; one beam passes through the object, then through a scattering solid, then it is combined with the other beam, and the combination is focused on an image recorder, generating a speckle pattern (a blurred image to the eye). When two such speckle images are subtracted (optically or digitally), a clear image of the refractive index field appears. Usually a reference speckle is shot ob the object before the flow starts, so that continuous subtraction to the subsequent speckles yields a sequence of refractive maps.

MOIRE DEFLECTOMETRY (MD)

A collimated light beam (preferably coherent) goes through an object phase, then through two separated parallel gratings and is finally focused on an image plane. The superposition of the two fine linear gratings yields an X-pattern of intensity fringes (moiré pattern) that is distorted by the ray deflection within the object path. The sensitivity of the instrument can be tuned by adjusting the distance between gratings and their relative rotation.

RAMAN SCATTERING (RS)

Raman scattering is the most used spectrometric technique of analysis. It measures the inelastic radiation scattered by molecules when a strong laser light is shone on them (the signal is weak). The shifting in wavelength from the source depends on the type of molecules, whose concentration depends on the line intensities, whereas the relative intensities amongst the lines for a single species is related to the local temperature (in reality, to the vibrational-rotational temperature of the molecules, that may be different to the translational temperature if there is no local equilibrium).

Coherent anti-Stokes Raman Scattering (CARS)

In this technique, two lasers are focused on the sample (a point, sheet or volume) at the same time, with their frequencies tuned to enhance the response of one type of molecule by the process of stimulated emission. Coherent anti-Stokes Raman scattering is being used for accurate temperature measurements in research on turbine combustors.

LASER INDUCED FLUORESCENCE (LIF)

In this technique, a laser of appropriate wavelength is focused on the sample so as to excite the electronic energy levels in some type of molecules, notably hydroxyl radicals (OH), polycyclic aromatic hydrocarbons (PAH), or formaldehyde (CH_2O), which, upon subsequent relaxation, yield a characteristic emission. LIF has been successfully used, for instance, on optically accessible engines to measure two-dimensional temperature fields and fuel concentration fields, before and after ignition, using excimer lasers at 248 nm and 308 nm tracer molecules (3-pentanone, 10%wt) added to the fuel (iso-octane). LIF-OH is used for the hottest part of flames, whereas LIF-CH_2O is used in the coldest part of flames.

LASER INDUCED INCANDESCENCE (LII)

This technique is used for detailed analysis of soot formation. Visible radiation from a sooting flame comes from all regions of the flame, giving only an overall picture of a three-dimensional and often unsteady phenomena; with a powerful laser, however, a line or sheet is shed onto the flame and only soot incandescence in that part is sampled, since the natural emission is much dimmer.

(back to Combustion)