# Experimental Methods

**ISBN 978-606-95069-8-1**

**Dr. BENOUDJAFER Imane & Dr. BENOUDJAFER Ibtissam**

**Technium Press**
**2024**

# Experimental Methods

Pedagogical Course Handout

TECHNIUM
BOOKS

Dr. BENOUDJAFER Imane & Dr. BENOUDJAFER Ibtissam

EDITORIAL OFFICE
TECHNIUM PRESS
E.U.

For citation purposes, cite as indicated below:

BENOUDJAFER Imane, BENOUDJAFER Ibtissam , Experimental Methods; ISBN 978-606-95069-8-1, TECHNIUM PRESS, 2024..

**FIRST EDITION 2024**

**ISBN 978-606-95069-8-1 (PDF)**

**Cover image courtesy of DALL-E 2.**



# Ministry Of Higher Education and Scientific Research

## The University of TAHRI Mohamed Bechar

## Faculty of technology

## Department of civil engineering and hydraulics

**Pedagogical Course Handout**

**Title:**

# " Experimental Methods"

**Level: Master**

**Field of study: Civil Engineering**

**Specialty: Civil Engineering**

**Prepared by the teacher:**

**Dr. BENOUDJAFER Imane& Dr. BENOUDJAFER Ibtissam**

**Academic year: 2023/2024**

# Preface

This handout is the written support of the course "Experimental Methods", intended for students enrolled in the first year of the master system LMD, Civil Engineering (Materials), second semester of the academic year. It has been written with the aim of mastering the experimental methods and to have a working and reference means covering the required knowledge.

The content of this handout, in accordance with the official program of the subject taught, is composed of three chapters:

- ✓ Classical methods;
- ✓ Physical and physicochemical methods;
- ✓ Mechanical methods.

In order to allow the student to better understand, assimilate, deepen and visualize or shed light on the practical side of certain theoretical notions presented, we have provided several examples of application.

**Dr. Imane BENOUDJAFER**
**The University of Tahri Mohamed Bechar**
**Bechar, Algeria**

# Table of contents

*INTRODUCTION* ..... *1*

## CHAPTER I: Classical methods, description and application

***I.1. GRAVIMETRY*** ..... ***2***

*I.1.1. Definition* ..... *4*

*I.1.2.Principal* ..... *4*

*I.1.3. Apparatus /Equipment.* ..... *5*

*I.1.4 Application* ..... *8*

***I.2. VOLUME*** ..... ***12***

*I.2.1. Definition* ..... *12*

*I.2.2. principle* ..... *12*

*I.2.3. Apparatus* ..... *13*

*I.2.4. Application* ..... *14*

***I.3. DIFFERENTIAL THERMAL ANALYSIS*** ..... ***15***

*I.3.1. Definition* ..... *15*

*I.3.2. Principle* ..... *16*

*I.3.3. Apparatus* ..... *17*

*I.3.4. Application* ..... *18*

***I.4 CALORIMETRIC ANALYSIS*** ..... ***21***

*I.4.1. Definition* ..... *21*

*I.4.2. Principle.* ..... *21*

*1.4.3. Apparatus* ..... *23*

*I.4.4. Application..* ..... *24*

## CHAPTER II: Physical and physicochemical methods, description and Application


***II.1. EMISSION AND X-RAY FLUORESCENCE SPECTROSCOPY ..... 28***

*II.1.1.Definition ..... 28*

*II.1.2. X-ray production in X-ray fluorescence spectrometry ..... 28*

*II.1.2.1. X-ray tubes ..... 28*

*II.1.2.2. Radioactivesources ..... 29*

*II.1.3. Description ..... 30*

*II.1.3.1. Principle ..... 30*

*II.1.3.2. Measurement methods ..... 32*

*II.1.4. Apparatus ..... 33*

*II.1.4.1. Primary radiation source ..... 33*

*II.1.4.2. WDS and EDS spectrometer ..... 34*

*II.1.5. Application ..... 37*

***II.2. X-RAY DIFFRACTION ..... 39***

*II.2.1. Definition Primary radiation source ..... 39*

*II.2.2. Principle ..... 39*

*II.2.3 Description ..... 40*

*II.2.3.1. X-ray production ..... 40*

*II.2.3.2. Line spectrum ..... 42*

*II.2.3.3. Lines' intensity ..... 42*

*II.2.3.4. Measurement methods ..... 43*

*II.2.3.5. Structure of the minerals ..... 43*

*II.2.4. Apparatus ..... 45*

*II.2.5. Application ..... 46*

***II.3. SCANNING ELECTRON MICROSCOPY ..... 49***

*II.3.1. Definition ..... 49*

*II.3.2. Description and application ..... 49*

*II.3.2.1. Principle ..... 49*

*II.3.2.2. Apparatus ..... 51*

*II.3.2.3. Electron types* ..... *51*
*II.3.2.4 SEM image* ..... *54*
*II.3.2.5. Contrast of the SEM image* ..... *55*
*II.3.2.6. Magnification of the SEM image* ..... *56*

## CHAPTER III: MECHANICAL METHODS

***III.1. BASIC NOTIONS*** ..... ***60***
*II.1.1. Representative volume* ..... *60*
*II.1.2. Strain rate* ..... *61*

***III.2. STATIC TESTS*** ..... ***62***

*II.2.1. Tensile test* ..... *62*
*III.2.1.1. Definition* ..... *62*
*III.2.1.2. Principle of the test.* ..... *62*
*III.2.1.3. Apparatus* ..... *62*
*II.2.2. Compression test* ..... *62*
*III.2.2.1. Definition* ..... *62*
*III.2.2.2. Principle of the test* ..... *62*
*III.2.2.3. Apparatus* ..... *63*

***III.3 DYNAMIC TESTS*** ..... ***71***
*III.3.1. High speed tensile test* ..... *71*
*III.3.1.1. Definition* ..... *71*
*III.3.1.2. Principle* ..... *72*
*III.3.1.3. Apparatus* ..... *72*
*III.3.2. Hopkinson compression tests* ..... *74*
*III.3.2.1. Definition* ..... *74*
*III.3.2.2. Principle* ..... *74*
*III.3.2.3. Apparatus* ..... *75*

***III.4. MEASUREMENT OF DEFORMATIONS ..... 76***
*III.4.1. Contact measurements ..... 76*
*III.4.1.1. Strain gauges ..... 76*
*III.4.1.2. Vibrating string extensometers ..... 78*
*III.4.2 Non-contact measurements ..... 78*
*III.4.2.1. Optical fiber ..... 81*
*III.4.2.2 Laser interferometry ..... 84*

# INTRODUCTION

In the field of civil engineering, experimentation is an essential component of the research process. It is often the starting point for research on a given topic. An efficient research, leading to different applications, usually includes experimental aspects, modelling aspects and aspects of validation of theories and models (theory-experiment comparison), which are developed in close connection. The success of the research is generally linked to a good balance between these different components.

Indeed, the knowledge of experimental methods is of particular importance in experimentation, for the preparation of the experimental protocol, the planning of the tests, the loading systems, the instrumentation, the data acquisition and their processing.

In this context, this handout aims to introduce students to the principles of experimental methods in some subfields of civil engineering, to explain the situation in the development of a research, to describe the objectives to be achieved, to present the different principles on which these experimental methods are based, illustrated on different cases of application and to give the essential rules to be respected in order to ensure the success of these methods.

This handout is designed to introduce students to experimental methods in order to ensure mastery of the varieties of advanced experimental study techniques. The course covers mainly some aspects of static and dynamic structural problems. The aim is to give general descriptions with definitions and applications for each of the methods covered, without going into too much detail, as each method could be the subject of one or more books on its own.

This manuscript consists of three chapters.

**The first chapter** presents a descriptive synthesis of the classical methods and their applications. First, the gravimetric and volumetric methods are presented with their applications.Then, the Differential Thermal Analysis which represents the first physical method used for the research and the detection of the phase changes during the evolution of a

chemical system by temperature variation, will be approached with each different category of compound. Finally, the technique used to study the thermal transitions of a material with its applications, will be exposed. This is the Differential Scanning Calorimetry, in which the heat flow (thermal power) to a sample is measured as a function of time or temperature while the temperature of the sample is programmed, in a controlled atmosphere.

**The second chapter** describes a general synthesis of physical and physicochemical methods used in the field of Civil Engineering as experimental methods. X-ray fluorescence spectroscopy (XRF) will be presented first. Then, the main testing techniques used, to deduce the elemental composition of the sample in a qualitative and quantitative way, will be briefly exposed. It concerns the ways of analysis of the spectrum and the methods of measurement that can be carried out to determine the nature and the quantity of the atoms that are present in the sample. Finally, the main testing techniques used and their applications, to test a sample, will be briefly explained. It concerns X-ray diffraction and scanning electron microscopy.

**The last chapter** is devoted to the presentation of mechanical methods that represent the essential step to access the characteristic quantities of materials, from Young's modulus to the yield strength, through the toughness or the resistance, and this under variable conditions. In order to do so, some basic notions will first be described in order to facilitate the understanding of the chapter content. Then, the approach of the mechanical tests followed in experimental methods, will be explained, with its two categories of regime (static and dynamic). As for the first category, we will limit our presentation to the most commonly used mechanical tests. On the other hand, the second one concerns a summarized presentation of a set of dynamic tests allowing to characterize well the materials and their complex behaviors is analyzed. Finally, the methods used to measure the deformations are briefly exposed. They include contact and non-contact measurement methods.

# CHAPTER I

## I.1. GRAVIMETRY

### I.1.1. Definition

Gravimetry is a technique that allows the detection of density variations (according to the composition of the soil) from the measurement of the intensity of the gravity field "g" compared to a reference value, based on the Laws of Universal Attraction (Newton's Laws), the gravitational potential and the gravitational field [PAN 06]. Values of the magnetic field that differ from the reference value are called anomalies.

### I.1.2. principle

Gravimetry allows to determine the geoid, which is defined by the equipotential surface corresponding to the surface of the oceans at rest [SCI 02]. By definition, the geoid is everywhere perpendicular to the vertical as indicated by the plumb line. It is a gravimetric and not a geometric representation of the Earth, as shown in Figure I.1.

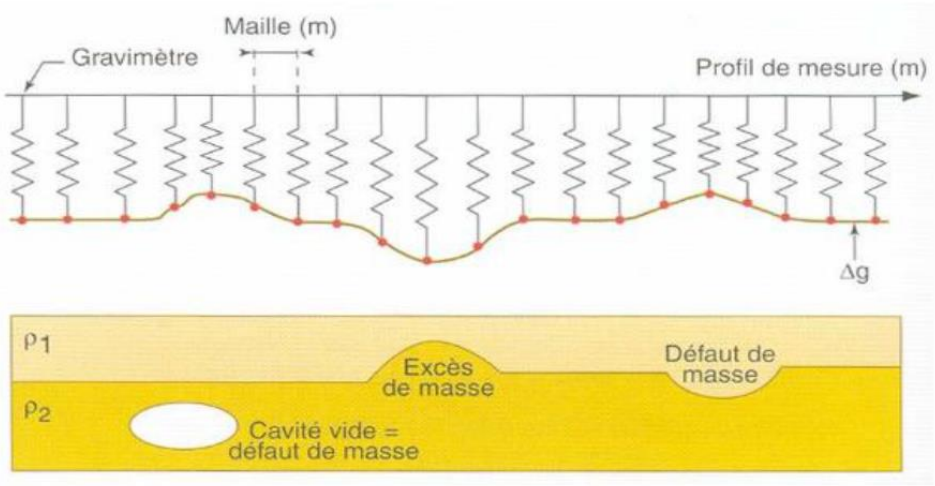



**Figure I.1:***Principle of gravimetry [PAN 06]*

The gravity field is the resultant of two forces:

- ✓ The universal attraction, directly related to the mass of the bodies (density);
- ✓ The centrifugal force, due to the rotation of the Earth (maximum at the equator and zero at the poles).

In order to obtain the variations of the gravitational field due to geological causes, it is necessary to correct our readings of all the other external causes that can influence them (drift of the device, tide, ellipticity of the Earth, ...) [PAN 06].

### I.1.3. Apparatus

The device used for the gravimetric analysis is called a gravimeter. In fact, it is an accelerometer, because according to the principle of equivalence, it measures the same thing [NF EN 13284].

However, a gravimeter is specialized in the measurement of a vertical acceleration close to the earth's gravity referred to as normal, and with a high accuracy around this value [NF X44- 052].

There are three types of gravimeters: **absolute atomic gravimeters, LaCoste-Romberg astatic gravimeters and superconducting gravimeters** [NF EN 13284], [NF X44-052] and [NF EN 13284-2].

**Absolute atomic gravimeters**: this type of gravimeter allows to measure the gravity of the Earth by Arnaud Landragin, CNRS Researcher at the Paris-Meudon Observatory [PAN 06]. The gravimeters are devices of the utmost importance for geophysics and in particular for the study of the internal structures of the Earth and their evolution. The "absolute" gravimeters have in particular the capacity to give the local acceleration of gravity in an exact manner, thus allowing to measure its evolution in space and/or time [SCI 02], as shown in Figure I.2.

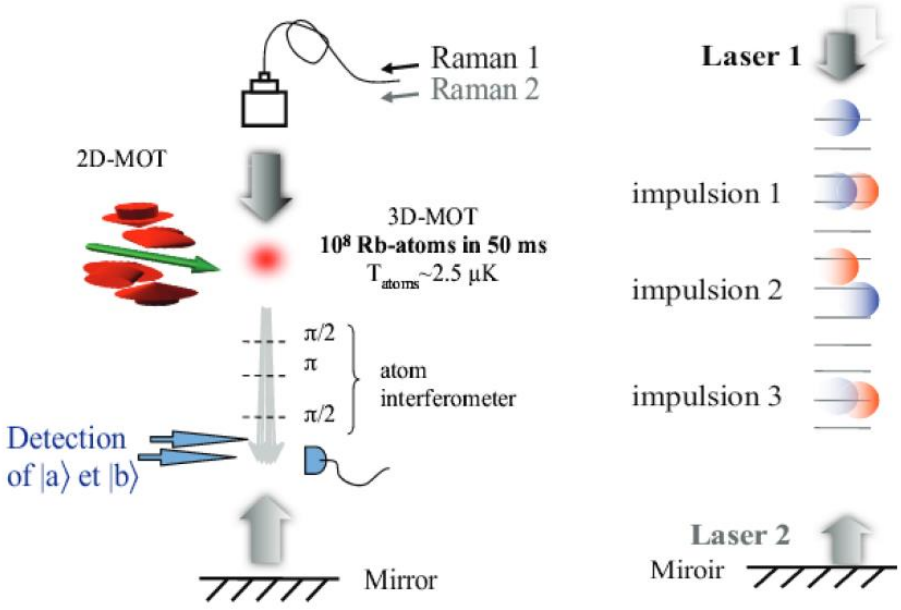



**Figure I.2**: *Schematic diagram of the atomic gravimeter [SCI 02]*

**a) LaCoste-Romberg astatic gravimeters**:

Another type of gravimeter is the La Coste-Romberg astatic gravimeter. It provides a higher measuring sensitivity. The main difference is that the mass is carried by a horizontal rod that can pivot [MEN 99]. This rod is also held by a spring [ROS 84]. When there is a variation in gravity, the rod pivots and the spring extends. The angle formed by the spring and the rod decreases [COM 09]. In order to be able to read the variation, we restore the rod to its horizontal starting position with a micrometer screw, as shown in Figure I.3.

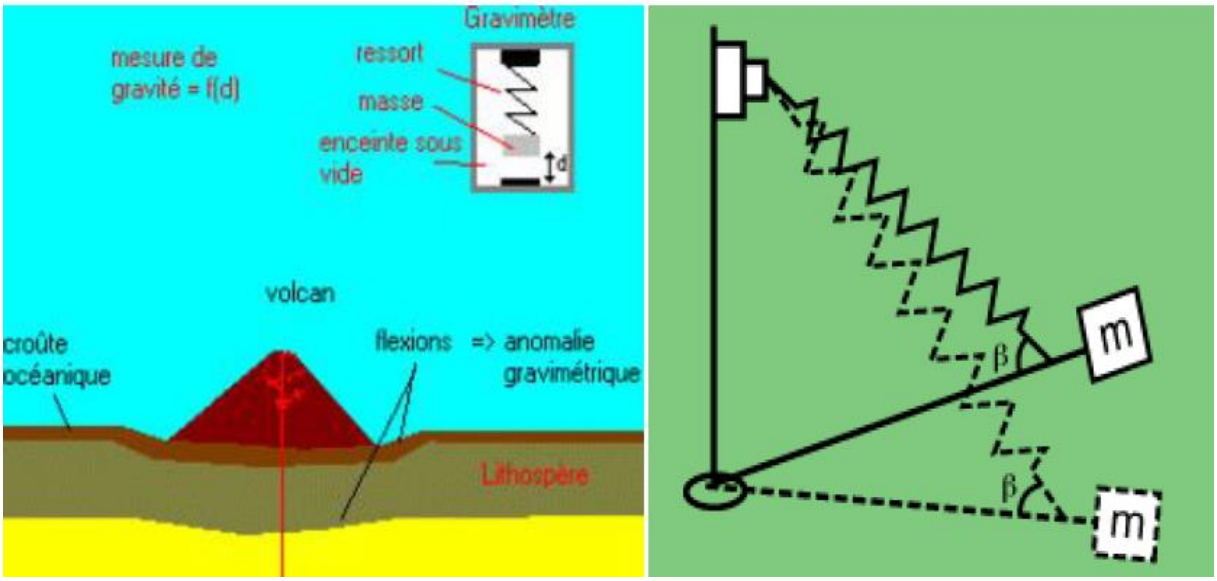



**Figure I.3:***Principle of operation of an astatic gravimeter LaCoste-Romberg [COM 09]*

Following the interferometer configuration, the phase shift depends on: a rotation speed and a length difference, Figure I.4.

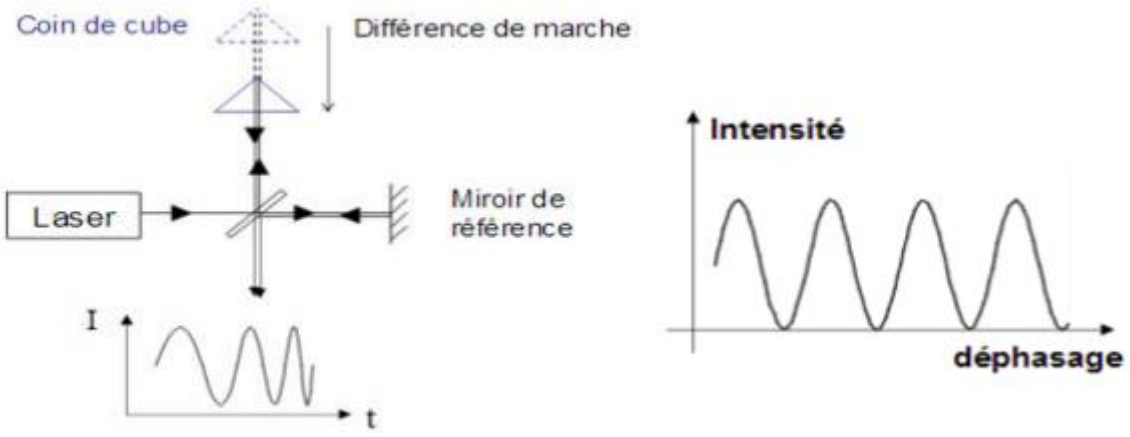



**Figure I.4:***Schematic diagram of the optical gravimeter [PED 77]*

**b) Superconducting gravimeters:** Superconducting gravimeters are characterized by a level of technicality largely superior to that of their counterparts with springs. The principle of this type of gravimeter consists in relying on the magnetic levitation created by two coils [BAR 53].

It is worth noting that there is a necessary condition to observe the magnetic field [DUP 04]: the coils and the sphere must be in the superconducting state (Figure I.5), which is achieved by a liquid Helium bath. Even if the latter is very well thermally isolated from the outside, such gravimeters require a lot of energy, and are therefore not usable everywhere [ROS 09].

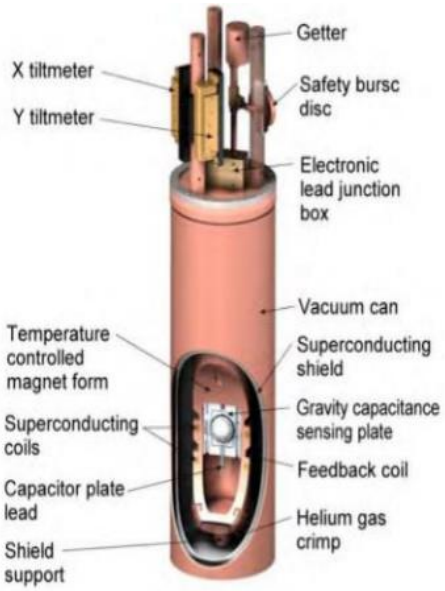



**Figure I.5:***Schematic diagram of the superconducting gravimeter [BAR 53]*

However, when gravity changes, the metal mass will either increase or decrease. An auxiliary coil creates a correction magnetic field so that the sphere returns to its initial position. By measuring the current flowing through the auxiliary coil, the acceleration of gravity is deduced.

### I.1.3. Application

Gravimetry can be used everywhere, in urban areas as well as in areas with uneven terrain. As the gravity field is a global phenomenon, the study scale can be very vast. Therefore, the application of gravimetry lies mainly in the knowledge of the subsoil, Figure I.6. It should be noted that the different types of rocks do not have the same density, and therefore do not have the same gravity on an object placed on the Earth's surface [NAG 66]. This method can therefore be used by geophysical companies [TAL 60], to find areas of land that may contain oil, water, or any other natural resource [PAN 06].

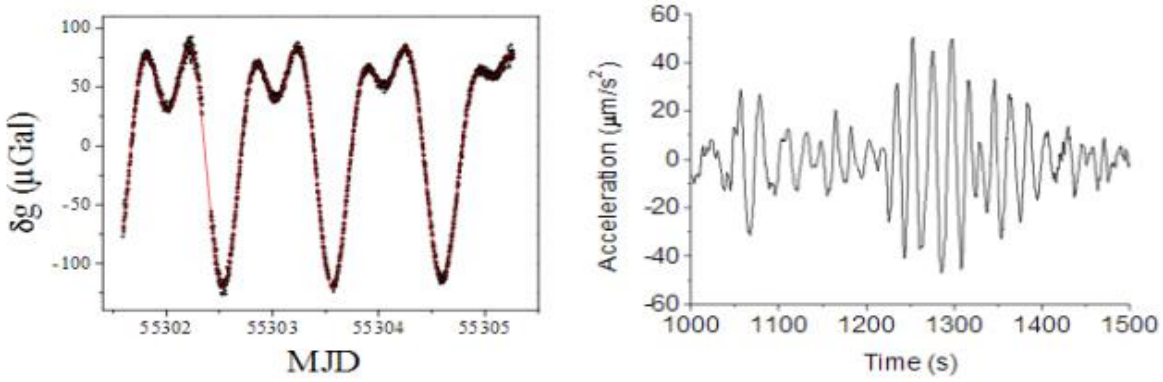



**Figure I.6:***Examples of gravimetry applications.*

*Excellent agreement between measurements & tidal model (lunar-solar tides: ±100 µGal; 1µGal=$10^{-8}$ m.s-2 or ~$10^{-9}$ g), (b) Earthquake, China in 2000 [PAN 06]*

It should be noted that the different types of rocks do not have the same density, and therefore do not have the same gravity on an object placed on the Earth's surface [NAG 66]. This method can therefore be used by geophysical companies [TAL 60] (repeated).

- ✓ **Scientific use:** geoid determination, study of gravity field variations, mass distribution in the earth's crust, post-glacial rebound, etc...
- ✓ **Petroleum prospecting:** search for hydrocarbon slicks that correspond to density diversifications (gravity measurements in prospecting wells or from the surface).
- ✓ **Underwater navigation**: With a gravity field map and a marine gravimeter, it is possible to navigate with precision.

Regardless of the gravimeter used, there are several corrections to be made to the measurements in order to determine the gravity value:

**a) Drift correction of the device**

This correction is intended to eliminate the effect on the measurements by the lunar tide and fatigue of the instrument [HWA 03]. To achieve this correction, it is necessary to carry out the measurements in a loop: the series begins and ends at a given point. For this point the starting and ending values are not similar, this difference is called drift [CHO 99]. It will then be added or subtracted from the measured gravity, Figure I.7.

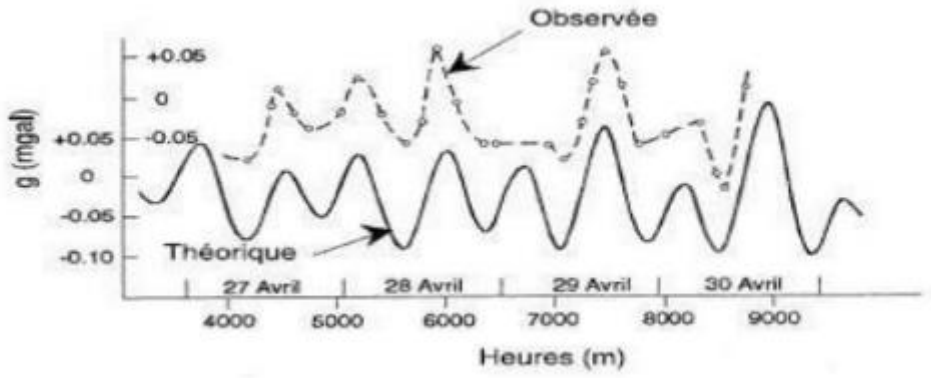



**Figure I.7:***Gravimetric tidal curves, theoretical in dashed lines and observed in solid lines [CHO 99]*

**b) Latitude correction**

This correction takes into account the changes of "g" with the latitude due to the rotation of the Earth and its flattening. The correction is considered positive if passing northward and negative if passing southward [CHO 99], as for example here with an origin point of coordinate (48°44'N), Figure I.8.

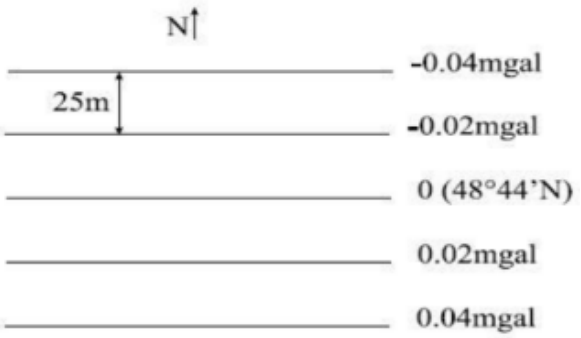



**Figure I.8**: *Example of latitude correction for a reference point with coordinates 48°44'N [CHO 99]*

**c) Altitude correction**

The altitude correction means taking into account the difference in altitude between the measuring station and the reference level [BHA 75], in this case the reference spheroid, Figure I.9.

The value of this correction is:

- ✓ proportional to the elevation (if h increases, Δh increases) ;

- ✓ negative if we are above the reference level;
- ✓ positive if we are below.

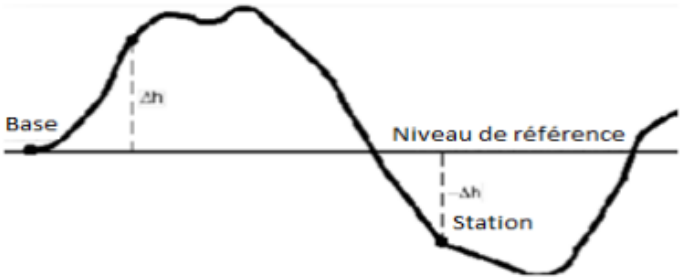



**Figure I.9***: Illustration of altitude correction [CHO 99]*

**d) Plateau correction**

It takes into account the mass between the reference frame and the measuring station [BER 81]. The mass is calculated with the altitude and the assumed density of the earth's crust. We often find a correction called "Bouguer correction" which combines the altitude correction and the plateau correction.

**e) Terrain correction**

The landforms near the measuring stations also have an effect that could be compared to a mini anomaly [BAC 67]. To evaluate this effect, it is necessary to use a chart corresponding to the topographic map of the place where the measurements are made: the HAMMER chart, as shown in Figure I.10.

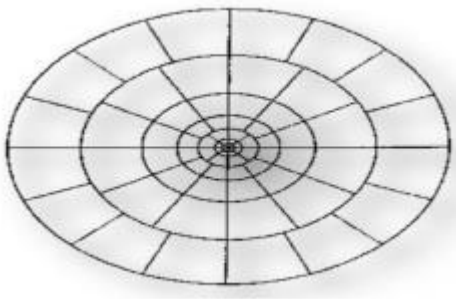


**Figure I.10:** *Hammer reticle in the outer zone [TOU 99]*

## I.2. VOLUME

### I.2.1. Definition

Volumetry is a quantitative analysis technique that allows the determination of different concentrations or chemical activity of a species in solution. It corresponds to the realization of the dosage and the result of such a dosage from a set of measurements for different proportions (table or curve, and not a sole measurement for a dosage) [NEV 87]. This method of analysis is based on, the exact measurement of the volume of a solution, of the concentration and the preparation of a titrated solution. Note that the concentration of a solution is determined by the knowledge of the volumetric dosage.

### I.2.2. Principle

The principle of volumetric analysis is based on the realization of chemical reactions that allow the identification of changes in oxidation state, that is to say, the transfer of insoluble electrons [LEM 03]. According to the type of added agent, we can distinguish two categories of volumetric analysis [PIL 04]:

- ✓ ***Acidimetric volumetry***: this category is characterized by the dropwise operation of an acid in a determined volume of base;
- ✓ ***Alkalimetric volumetry:*** in this second category, the experimenter must perform the operation in a determined volume of acid.

Volumetry entails pouring from a burette a precise volume of a solution of known concentration into a solution containing the substance to be determined, of unknown concentration [PIL 04]. The substance in the titrant reacts continuously with the analyte. The point reached when enough titrant has been added for all the analyte to react is called the "equivalence point" [LEM 03]. This point is often identified by the change in color of a chemical called indicator.

The different conditions for a successful volumetry are [NEV 00].:

- ✓ To know the exact reaction that takes place between the titrant and the substance to be analyzed;
- ✓ The equivalence point must coincide with the turning point of the indicator used;
- ✓ The volume of titrant required to reach the equivalence point must be measured accurately.

### I.2.3. Apparatus

The apparatus of the volumetry consists of a graduated burette when measured manually or in the form of automatic titrimetry when the aim is to improve the repeatability and traceability. The measurement of volumetry therefore consists of taking the volume of a sample using a pipette placed in an Erlenmeyer flask [NEV 87].

It is to be noted that the burette regularly contains a titrating reagent solution allowing to know its concentration [NEV 87]. Consequently, the graduation of the reagent solution indicates the volume poured, which makes it possible to give the equivalence point and the solution to be determined will often be in a clean beaker [PIL 04], in which the volume will be exactly known, Figure I.11.

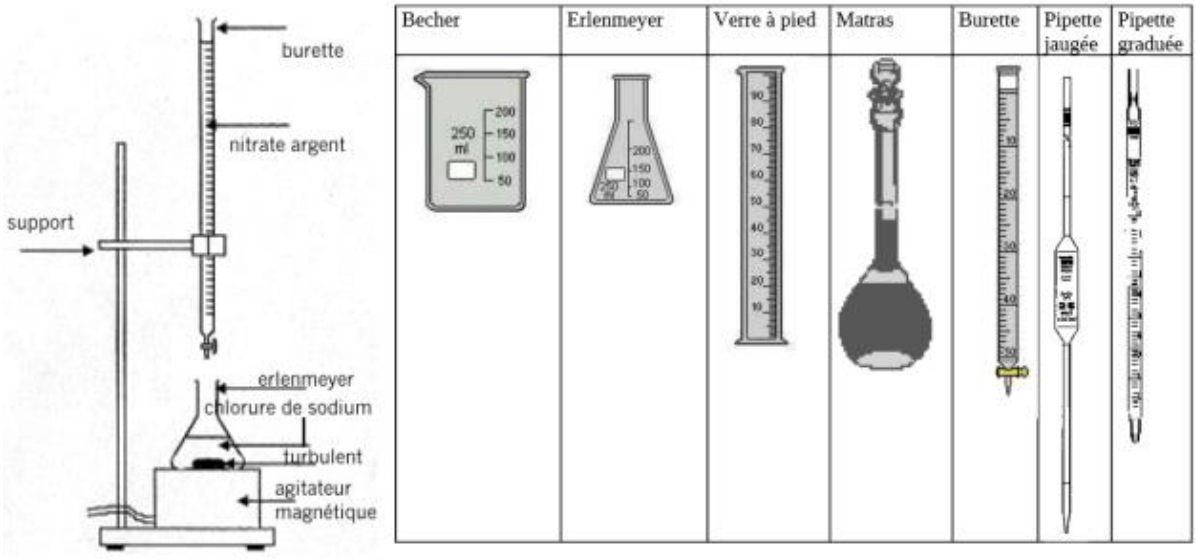



**Figure I.11**: *Instruments used in volumetric analysis that deliver or contain known volumes with precision [NEV 87]*

Volumetric instruments are fundamental instruments which can be made of glass or plastic. In order to meet the ever-increasing demands of volume measurement in the laboratory (serial examinations, test series), new instruments have been developed continuously, for example for dispensing, pipetting and titration [NEV 00].

The devices designed for the same applications by different manufacturers work according to the same principle. Depending on the manufacturer, there are, however, significant differences in the details of the construction and design of the devices [NEV 87]. Each glass volumetric device is individually adjusted. For this purpose, a defined amount of water is precisely dosed and the gauge line is applied to the lowest point of the meniscus. In the case of graduated volumetric instruments, two measuring lines are applied. In fully automated production, computer-controlled systems ensure the highest possible accuracy. Statistical control of the production process allows the production of volumetric instruments with the smallest possible deviation from the nominal value (accuracy) and a small variation of the individual values (coefficient of variation) [PIL 04].

### I.2.4. Application

The measurement of liquid volumes is part of the routine work of every laboratory. Volume measurement is of primary importance in the laboratory. The user has to clarify the accuracy required for the realization of a concrete measurement. Based on this assessment, he can choose the right instrument for his volume measurement [NEV 00].

Volumetric analysis is of great practical interest and has a great advantage in terms of speed of execution. It involves a chemical reaction or physicochemical interaction between the compound to be measured and a known reagent that is added incrementally, a reaction that must be rapid, complete, unambiguous, and measurable [PIL 04]. The final result is the measurement estimated to correspond to an equivalence between these two components, typically a color change, or the inflection point of the measurement curve, or 50% of the variation between their excesses. Accurate measurements require accurate measuring equipment and proper handling. [EBE 76].

## I.3. DIFFERENTIAL THERMAL ANALYSIS

### I.3.1. Definition

The Differential Thermal Analysis (DTA) was devised by Roberts-Austen in 1899. It aims to highlight the transformations that affect the structure of materials when their temperature is altered.

According to ICTAC (International Confederation for Thermal Analysis and Calorimetry) Differential Thermal Analysis (DTA) is a technique in which the difference in temperature between a sample and a reference material is measured as a function of time or temperature while the temperature of the sample is programmed, in a controlled atmosphere.

The graph obtained is called the "differential thermal curve" which comes from the change in chemical composition and the crystal structure of the sample. With the difference in temperature observed between the sample and the reference, we can make a graph of this difference as a function of time or certain difference as a function of time or certain temperatures, Figure I.12

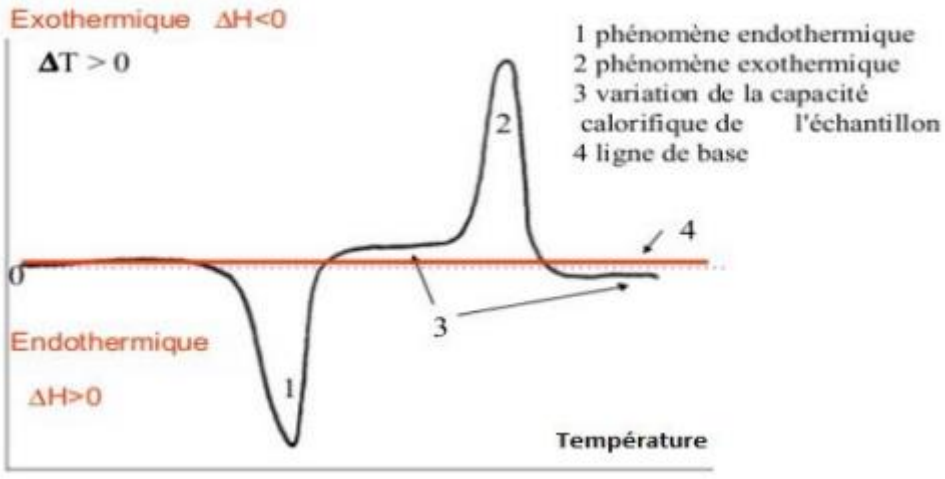



**Figure I.12:***Typical theoretical curve obtained by DTA [BAR 96]*

When the temperature of the sample takes longer to increase compared to the reference, we have a temperature absorption and our peak is therefore endothermic, Figure I.13a. On the contrary, if the temperature of the sample increases faster than that of the reference, we get an exothermic reaction, Figure I.13b

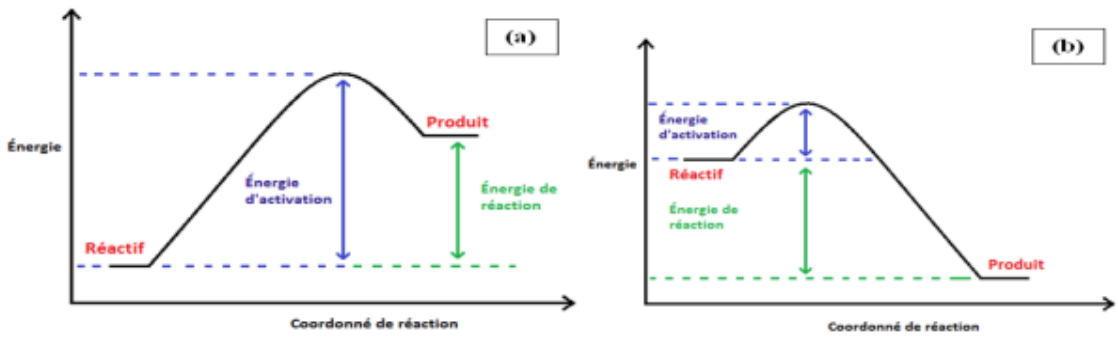



**Figure I.13**: *Recording of temperature as a function of time [BAR 92]*

*(a) endothermic (b) exothermic*

### I.3.2. Principle

The principle of the Differential Thermal Analysis consists in heating under identical conditions, the sample to be measured (S) whose thermal properties are unknown with another sample of identical shape or reference (R) which does not present thermal anomaly in the selected temperature range, or whose specific heat is known (Figure I.14). The differences between the thermal properties of the various samples are reflected by differences in temperature between them [EBE 97].

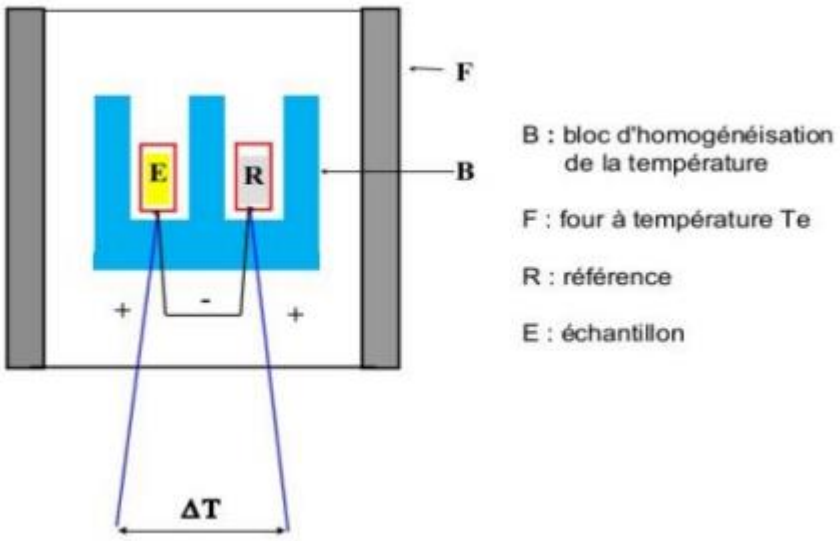



**Figure I.14:***Operating principle of the DTA system [EBE 76]*

It is important to specify that the mineralogical and petrographic procession used by the potter is not found in the final product. Apart from the identification of species, DTA is

useful in the study of the behavior of minerals vis a vis the heat. Structural changes are thus highlighted by cross-checking with other physicochemical methods, notably X-ray Diffraction before and after DTA. generally speaking, the phase transitions and the evaporation of solvents are expressed by endothermic peaks [EBE 97]. On the other hand, crystallization, oxidation and certain decompositions are characterized by exothermic peaks.

### I.3.3. Apparatus

There are different types of DTA devices. The distinction between these apparatuses is made on the quantity of sample which can be brought into play during the analysis as well as the nature of the material.

The devices used for DTA can be divided into several categories [EBE 89]:

devices that allow only a "qualitative analysis": they are characterized by a simple construction, good resolution and good temperature accuracy (Figure I.15a);

devices that allow "quantitative analysis": this type of device is characterized by very reproducible results (Figure I.15b);

devices that allow "calorimetric measurements": this last category represents more complex systems than the devices presented previously, they are based on the same principle of differential measurement (Figure I.15c).

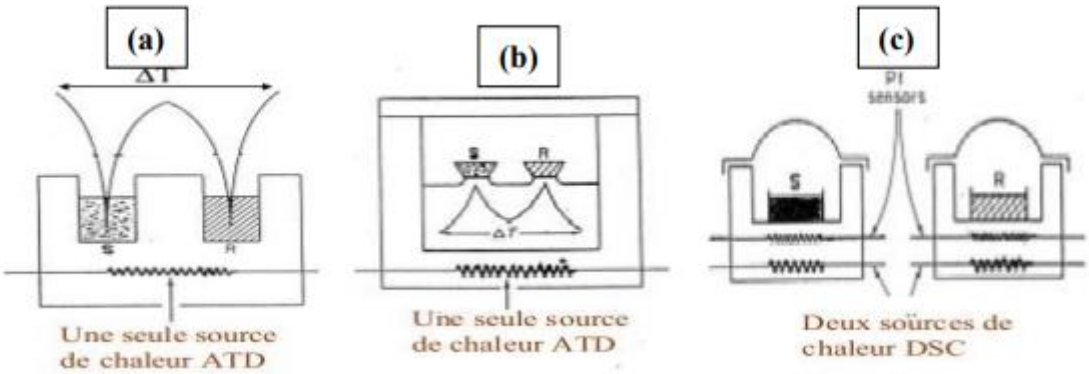



**Figure I.15:***Different types of thermal analyzers [EBE 89]*
*(a) quantitative, (b) qualitative, (c) calorimetric*

To be noted that the multiplicity of materials used allows to highlight the interactions, i.e., the product/walls parameter, which are particularly important in DTA given the low mass of the sample. Among the known interactions we can cite:

- ✓ The reaction between hydrochloric acid and stainless steel;
- ✓ The decomposition of chlorinated organics into hydrochloric acid attacking the stainless steel. Hydrochloric acid and ferric chloride are catalysts of the thermal decomposition of many organic substances;
- ✓ Hydrofluoric acid attacks glass.

### I.3.4. Application

The Differential Thermal Analysis is the first physical method that was used for the search and detection of phase changes during the evolution of a chemical system by temperature variation [LAR 88]. The interest of the DTA concerns three essential points:

- ✓ The identification of phase transition phenomena;
- ✓ The identification of the crystalline phases involved after interpretation of the endothermic or exothermic phenomena recorded at characteristic temperatures;
- ✓ The evaluation of the firing temperature of the studied material according to the phases appeared.

In differential thermal analysis, there are several compounds that can be analyzed. We can analyze organic products, inorganic products, etc. In each different category of compound, there are parts of the apparatus that we must have and that we do not necessarily need for other compounds [EBE 76]. For example, for metals and alloys, the apparatus must have thermocouples made of special ceramics that are chemically inactive so as not to interfere with the analysis in conditions where the temperatures are very high. It is also necessary that the sample holder is made of a special material to prevent that the high temperature and the high pressure distort the results.

The Differential Thermal Analysis is generally associated with a thermogravimetric analysis which allows to measure the variation of mass of a sample according to the temperature of thermal treatment [NEV 00]. This mass variation can be a mass loss or a mass gain, Figure I.16. Note that the DTA is a calorimetric method, since it takes advantage of the thermal effects, positive or negative depending on the case of study, which go along with the tested changes [MUR 03]. For mixtures, a detection of constituents is not always feasible because

many limitations prevent DTA from being a general method of quantitative analysis [NEV 00]:

- ✓ possibility of having characteristic accident overlaps;
- ✓ chemical reactions between the different solid components of the mixture;
- ✓ Effect of certain impurities on the position and shape of the accidents that can be encountered.

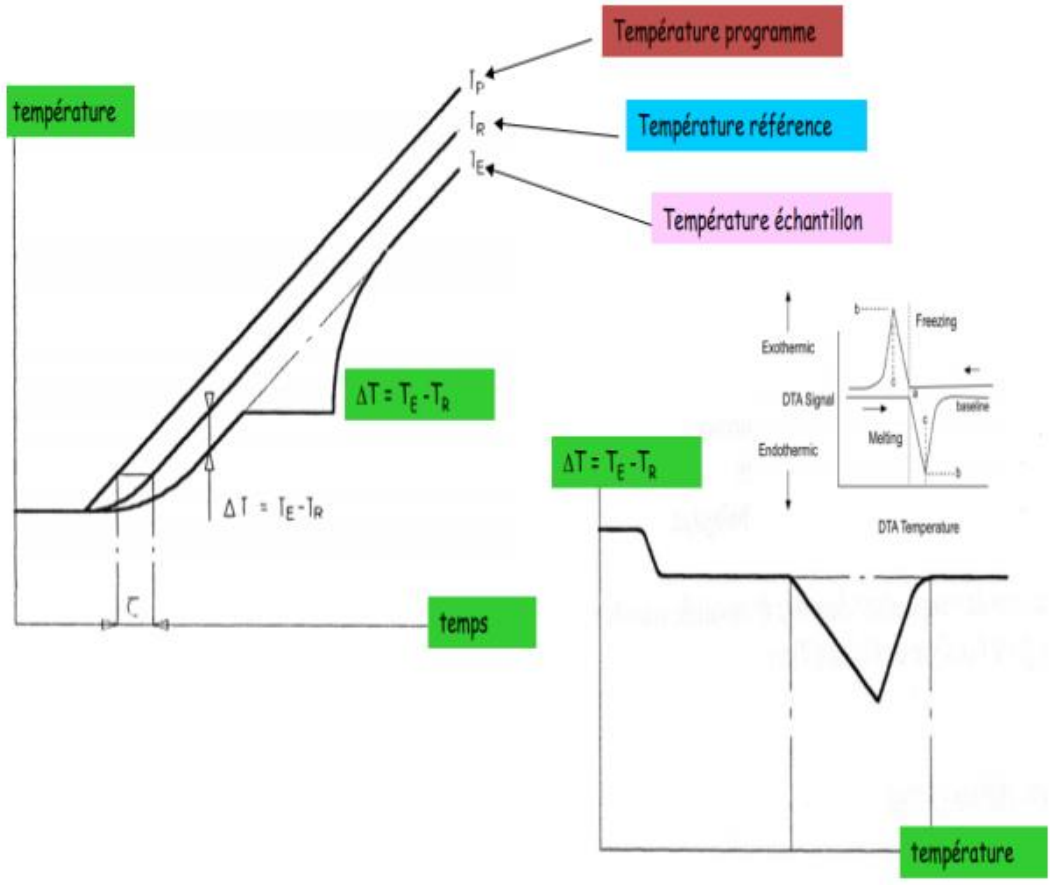



**Figure I.16:***Recording of temperature as a function of time [MUR 03]*

We present hereafter (Figures I.17. and I.18) examples of the differential thermal analysis obtained with different thermal cycles.

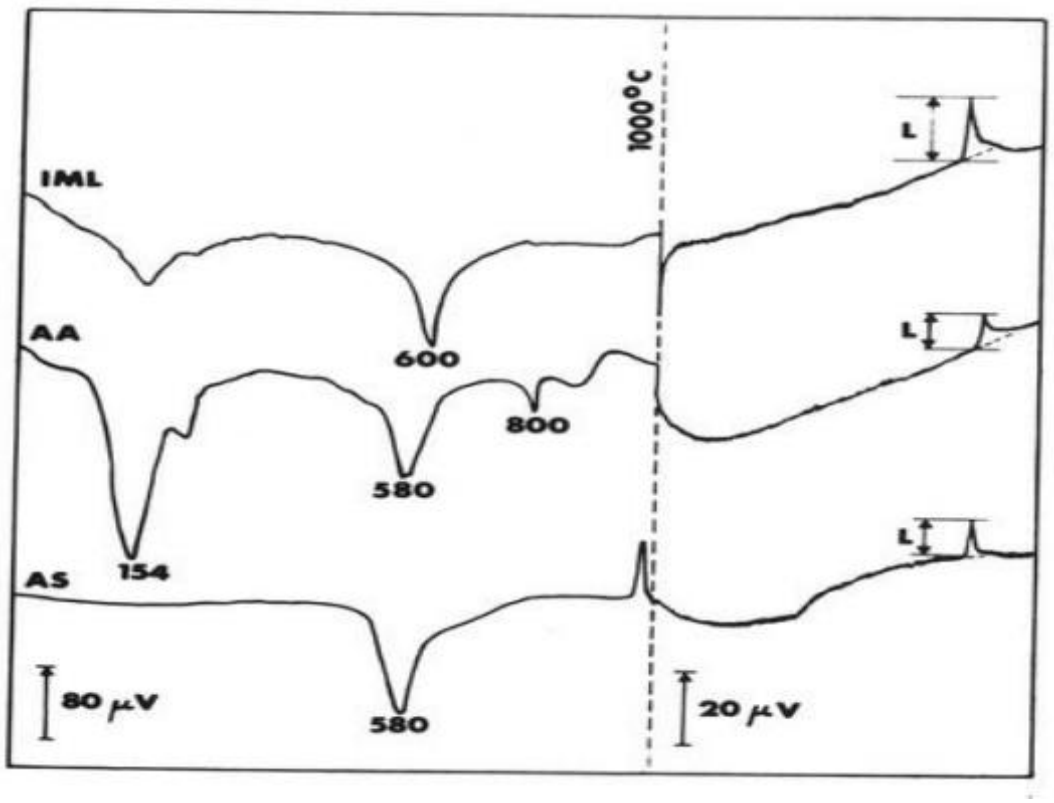



**Figure 17:***Differential thermal analysis curves of clay between 25 and 1000°C, followed by cooling to around 400°C [TOR 96]*

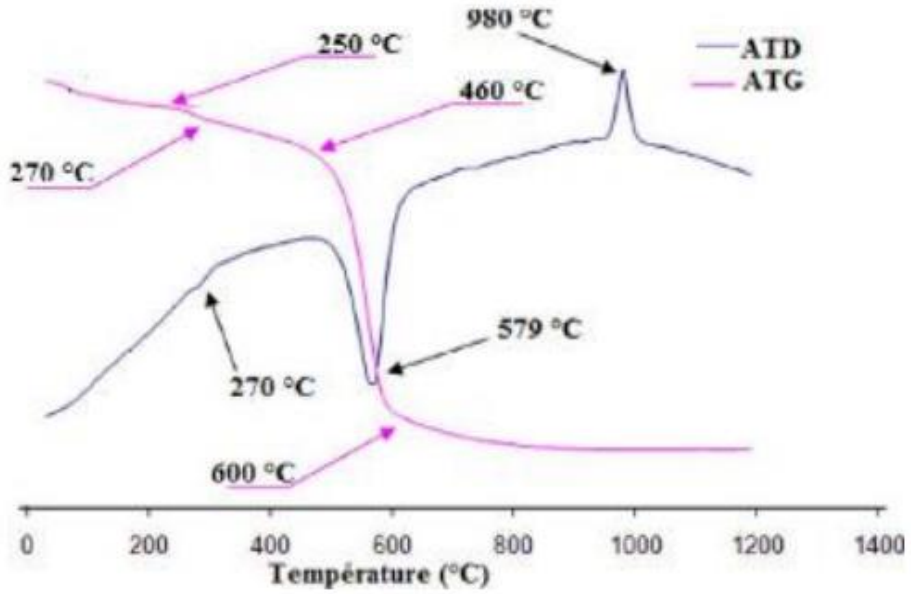



**Figure 18:***DTA and TGA thermograms of the clay fraction [NEV 90]*

## I.4. CALORIMETRIC ANALYSIS

### I.4.1. Definition

Differential Scanning Calorimetry (DSC) is a technique used to study the thermal transitions of a material. It is defined according to ICTAC, as a technique in which the heat flow of a sample is measured as a function of time or temperature while the temperature of the sample is programmed, in a controlled atmosphere [PIL 04].

### I.4.2. Principle

The principle of this method is based on the fact that during a physical transformation, a certain amount of heat is exchanged with the sample to be maintained at the same temperature as the reference [EBE 76]. The direction of this exchange depends mainly on the nature of the transition process, Figure I.19.

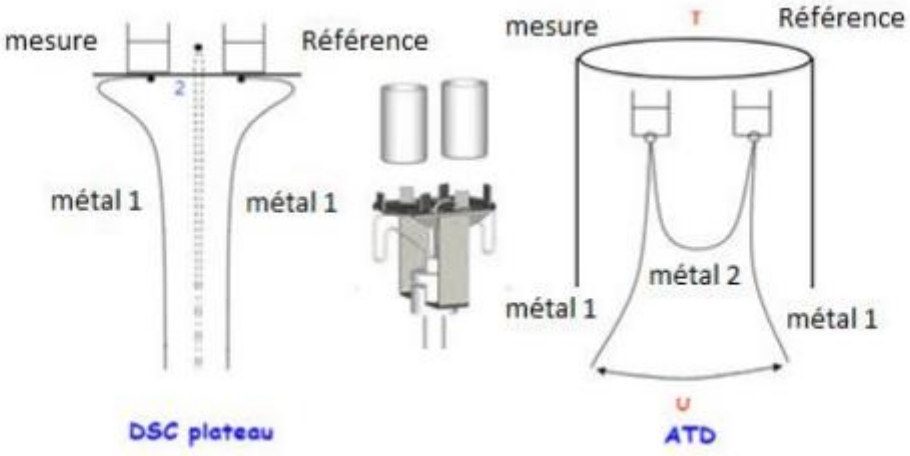



**Figure I.19:***Diagram of a DSC plate [EBE 76]*

The DSC allows to know the temperatures at which appear the different transformations formed and also to provide qualitative and quantitative information on these reactions. Depending on the parameter kept constant, two methods of DSC are known [PIL 04]:

**power-compensated DSC**, keeps the power supply constant;

**heat-flux DSC**, keeps the heat flux constant.

**a) Compensation method**

In this method, which was originally invented by PerkinElmer, the sample and the reference are placed in two different ovens but in the same heat chamber. The temperature variation between the two ovens is done simultaneously by the same quantity of calories. The temperature is always maintained equal in both ovens, and varies linearly [EBE97]. The differences in energy absorbed or released by the sample and the reference are measured. When a transition occurs, depending on the type of reaction, whether endothermic or exothermic, the sample will absorb or release energy. A power generator provides more or less energy compared to the reference. It is this variation of energy that is recorded as a function of time or temperature, as shown in Figure I.20.

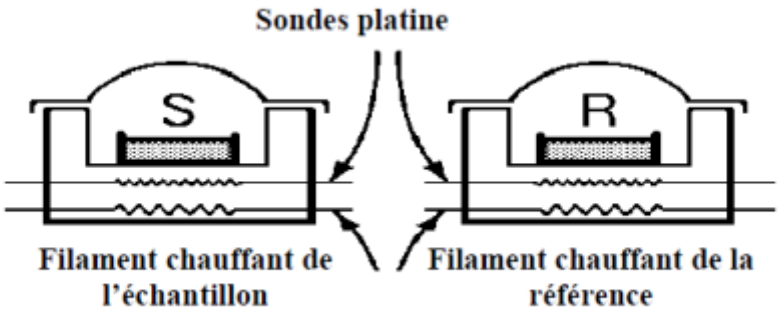



**Figure I.20**: *Principle of power compensation [EBE 97]*

**b) Heat flux method**

For this second method (developed by Du Pont de Nemours-Mettler), the sample and the reference are placed in the same thermal preservation medium (oven). A platinum probe is used to control and record the evolution of the temperature of the device. The temperature signal is then converted into a heat power signal, as shown in Figure I.21

This technique measures the heat flux differences between the sample and the reference during a temperature cycle. The heating temperature, provided by an electrical resistor, varies linearly.

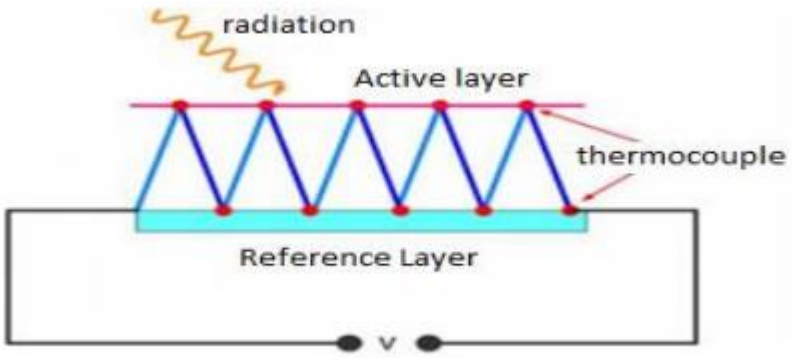



**Figure I.21:** *Principle of heat flux [EBE 76]*

### I.4.3. Apparatus

The measurement involves determining the variation in heat flux emitted or received by a sample when it is subjected to changes in temperature. During heating or cooling, any transformation occurring in a material is accompanied by a heat exchange studied (endothermic or exothermic) [EBE 76]. The knowledge of the course of a decomposition reaction allows the estimation of the domain of stability or instability of a reaction [NEV 87], the estimation of the temperature of the beginning of decomposition and also allows to estimate the consequences in terms of temperature and maximum pressure reached during decomposition, of the quantity of heat released etc... The apparatus used for Differential Calorimetric Analysis can be divided into several categories [EBE97]:

- ✓ **The "pseudo-adiabatic" calorimeters:** this first category allows to practice the technique in a controlled environment so that it is equal to that of the sample or the walls of the test cell to exclude any heat loss. With this technique, the heat released by the sample is used to increase its own temperature as well as to increase the temperature of the test cell [PIL 04].

- ✓ **DEWAR Vase Calorimeters:** The DEWAR pseudo-adiabatic sealed-vase measurement technique is an old method that is used by most process safety laboratories in the chemical industry. It was used in the earlier days to determine the minimum temperature at which thermal runaway would occur under adiabatic conditions. Today, these devices are used to simulate thermal runaway in order to

obtain the necessary experimental information. For this purpose, high-performance devices are characterized by lower adiabaticity coefficients.

- ✓ **"Isothermal" calorimeters:** this type of calorimeter is characterized by the possibility of operating either with a mixture of flows or in batch mode. the sample contained in a test cell is maintained at a constant temperature imposed by the device. When a temperature variation is recorded, the heating system automatically adjusts to maintain the temperature of the chamber constant. The thermal flux released or absorbed by the mixture of reagents is dissipated through an assembly of thermocouples constituting a fluxmeter towards a metallic mass constituting a heat sink.

- ✓ **"Isoperibolic" calorimeters:** this last category of calorimeters concerns the simplest and easiest heat flow devices in terms of equipment and maintenance. With this type of calorimeters, the average heat transfer is brought to a constant temperature and the heat flux measured by the variations of reaction temperatures. This category of calorimeter presents some disadvantages. The measured heat flow is not linear beyond high powers because the temperature is ever increasing, therefore the reaction kinetics and the heat capacity are altered. In addition, it is difficult or impossible to follow the reaction temperature and measure the heat flow. Therefore, this technique cannot be used easily to measure the heat capacity.

It is interesting to note that the analyses of the Differential Scanning Calorimeter are carried out under a sweep of an inert gas (for example, nitrogen or argon) to avoid any reaction of the material to be studied with the furnace atmosphere. The device undergoes significant temperature changes. Consequently, the position of some elements will vary [NEV 87]. All the calculations made by the computer interface are made in relation to the thermocouple positioned between the sample and the reference [PIL 04]. By moving, the results will be distorted. It is therefore necessary to calibrate regularly by checking for example the melting temperatures with indium, zinc or lead [EBE 97].

### I.4.4. Application

The fields of application of the DSC are thus very vast and very varied; this technique allows to [PIL 04]:

- ✓ Measure the purity of the product under test,
- ✓ Determine the thermal capacity,
- ✓ Study the materials considered as non-crystalline solids such as glasses, polymers and rubber;
- ✓ Study the phase diagrams of products considered as mineral and organic;
- ✓ Determine the thermal stability of various organic compounds;
- ✓ Study varieties of reactions such as oxidation, cross-linking...etc.

In addition, this type of analysis allows the determination of phase transitions such as [NEV 00]:

- ✓ the glass transition temperature (Tg) of samples such as polymers, ionic liquids and metallic glasses;
- ✓ the knowledge of the melting and crystallization temperatures;
- ✓ enthalpies of reaction, to know the cross-linking rates of some polymers.

Therefore, the DSC involves researchers and technicians in areas of research and development as well as quality control of a large number of industries (chemical, petrochemical, metallurgical, electronic, mining,) ... It is widely used in industrial quality control because of its applicability in the evaluation of the purity of samples or in the study of the curing of polymers. Among the constraints inherent to the DSC Differential Calorimetric Analysis, we can mention [PIL 04]:

- ✓ Low quantity of sample;
- ✓ Problem of sample representativeness and mass accuracy;
- ✓ the absence of the possibility of having an in-situ mixture;
- ✓ the analysis concerns greater samples.

It is noteworthy that differential scanning calorimetry (DSC) and thermodifferential analysis (TDA) are methods that are often confused. However, there is a fundamental difference between these two methods [NEV 87]. With a DSC apparatus, energy differences are measured, whereas with TDA, temperature differences are measured.

Thermodifferentialanalysis is seldom used for the characterization of polymers since the signals from phase or state changes are characterized by very low amplitudes [PIL 04].

Thus, for example, a solid that melts will absorb more heat to be able to improve its temperature to the same distance as the reference one. In fact, the transition from the solid to the liquid state is considered as an endothermic phase transition. Similarly, the sample under test can undergo exothermic processes when it transmits heat. By measuring the difference in heat flux, a differential scanning calorimeter can measure the amount of heat absorbed or released depending on the measurement conditions. This technique can also be used to observe more subtle phase variations, such as the case of glass transitions. Figure I.22 and I.23 show examples of the calorimetric analysis of mixtures.

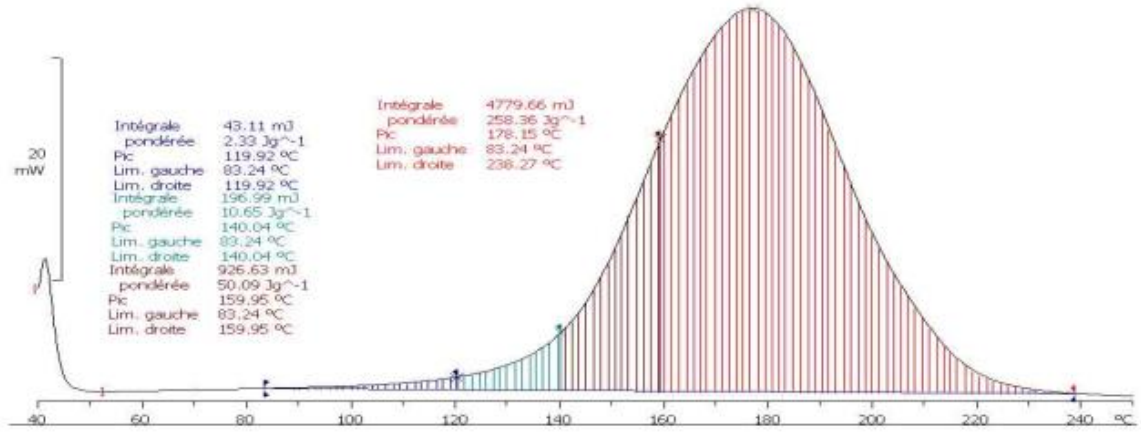



**Figure I.22:***Thermogram of the reaction of a mixture [FER 15]*

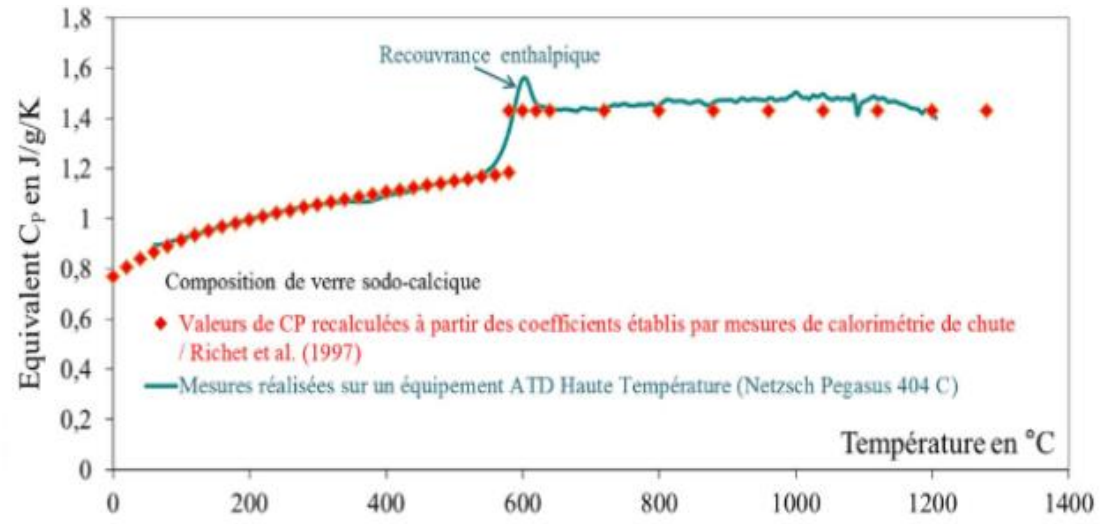



**Figure I.23:***Thermogram of the glass reaction [FRA 95]*

# CHAPTER II

## II.1. EMISSION AND X-RAY FLUORESCENCE SPECTROSCOPY

### II.1.1. Definition

X-ray fluorescence spectrometry (XRF) is a method of chemical analysis using a physical property of the matter. The spectrum of X-rays emitted by the latter is an essential feature defining the composition of the sample, by analyzing its spectrum, we can deduce its elemental composition, i.e., the mass concentrations of elements for the sample studied [BEK 06]. It can be done in two ways:

- ✓ by **"wavelength dispersive analysis"** (WD-XRF, wavelength dispersive X-ray fluorescence spectrometry);
- ✓ by **"energy dispersive analysis"** (ED-XRF, energy dispersive X-ray fluorescence spectrometry).

### II.1.3. Production of rays in X-ray fluorescence spectrometry

In practice, two types of X-ray sources in X-ray fluorescence spectrometry can be used:

- ✓ "X-ray tubes": this type of X-ray source uses electron bombardment;
- ✓ "Radioactive elements": on the other hand, this type of source uses the gamma radiation gamma radiation emitted by radioactive nuclei.

#### II.1.3.1. X-ray tubes

The usual source of X-rays is called the « Coolidge tube», named after its inventor (1917). It is a vacuum tube with two electrodes: a cathode emitting electrons and an anticathode composed of a metal mass held positively at a given potential.

The usual source of X-rays is called the "Coolidge tube", named after its inventor (1917). It is a vacuum tube with two electrodes: a cathode emitting electrons and an anticathode composed of a metal mass held positively at a given potential.

The electrons are accelerated by a high voltage on a metal target which represents a water-cooled anticathode [EBE 76]. These electrons are emitted from a heated filament through a transmitted current. If the voltage source gives a simple transformation, the anticathode is positive [JAC 52].

Two distinct categories of phenomena are based on the knowledge of the nature of the X-rays produced [EBE 97]:

- ✓ ***The emission of the continuous spectrum***: in this first category, the spectrum of emission is constituted by a set of radiations characterized by an intensity variable in a continuous way with the produced wavelength, Figure II.1a.
- ✓ ***The emission of the characteristic lines***: going to this second category, the continuous spectrum is superimposed with a spectrum of lines that are characterized by a wavelength independent of the general conditions of operation of the tube adopted, but depends more on the nature of the anticathode used. therefore, this type of emission is characterized by characteristic lines constituting an anticathode, as shown in Figure II.1b.

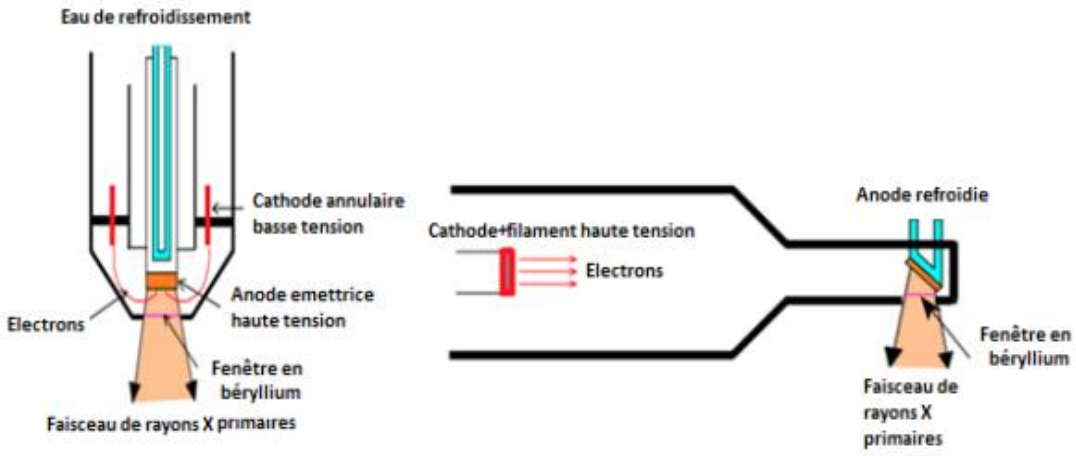



**Figure II.1:***X-ray tubes [EBE 97]*

*(a) continuous spectrum, (b): characteristic lines.*

### II.1.3.2. Radioactive sources

The radioactive sources of type " radionuclide " are commonly used in the case of the need for a radioactive source [HEI 81]. This type of source is very stable and has the advantage of being small. It should be noted that the low intensities produced make it possible to prohibit the use of a dispersion by a given crystal [ELA 02]. For this reason, this type of sources is almost exclusively associated with different techniques of energy dispersion. The emission of radioactive sources is based on the principle of the use of radiation of the "gamma" type which is emitted by radioactive nuclei. For certain atoms, this radiation can also tear

electrons from the layers located in their deep parts, on the one hand, and cause the fluorescence of these atoms, on the other hand [GOL 92].

The specific character of most of the radioactive sources used makes it possible to render several advantages in order to selectively excite certain elements [RON 99]. But it should be mentioned, that this characteristic corresponding to the selectivity does not always present an advantage, especially if we add low intensities of the radioactive rays. Consequently, the application of radioactive sources is limited due to practical constraints [NEV 00].

For this purpose, the use of the secondary fluorescence technique is recommended with such types of radioactive sources. This consists of inducing X-ray emission from a target subjected to gamma radiation from the radioactive source serving in parallel with the characteristic radiation from the atoms in order to induce X-ray fluorescence of the sample under test [RUS 78].

### II.1.4. Description

#### II.1.4.1. Principle

When a material is subjected to a radiation which is characterized by a low wavelength and thus a high energy, as in the case of X-rays, the gamma rays or the electron beam emitted can be used to sufficiently accelerate the formulated ions. As a result, the atoms constituting the material under test may undergo a state called "ionization", i.e., one or more electrons may be ejected from the electronic layers of each of these atoms [RON 99]. The energy source that produces the ionization is called "primary radiation", with the necessity of presenting a primary radiation energetic enough in order to pull the formulated electrons out of the inner layer of the induced atom. More precisely, the energy of the photons or primary particles must be greater than the ionization potential emitted [ELA 02].

The electronic transition emitted by the electrons is called "de-excitation" which indicates the displacement of an electron from a higher level "goes down" to occupy the half-empty quantum bin [BEK 06]. This electronic transition allows the release of the energy

corresponding to the difference in energy of the levels involved, either by the emission of a photon X or by the transmission of the energy through the atomic electrons [PIL 04]. The first case corresponds to "X-ray fluorescence", while the second case corresponds to "Auger emission", Figure II.2a.

The energy of the emitted X photon is equal to the energy difference of the two concerned orbitals. It is therefore characteristic of the atom where the emission was generated [NEV 00]. The distribution of the energies of all the emitted photons is called the "energy spectrum" [EBE 97]. The energy spectrum is therefore characteristic of the composition of the sample. The term fluorescence is applied to phenomena in which the absorption of a radiation produces the re-emission of a less energetic radiation [ELA 02].

Note that the fall of an electron from an upper layer to a lower layer implies that the latter has previously lost one of its electrons [EBE 76]. Secondary electrons can in turn excite atoms, especially light atoms such as carbon or nitrogen, and cause new X-ray or Auger fluorescence emissions [JAC 52]. Each chemical element is characterized by electronic orbitals of a certain energy [EBE 97], as shown in Figure II.2b. Note that the electronic layers are called "K", "L", "M" and "N". The transition produced from the L layer to K is traditionally called Kα, when to the second transition M→K is called Kβ, going to the last transition M→L is called Lα and so on [JAC52].

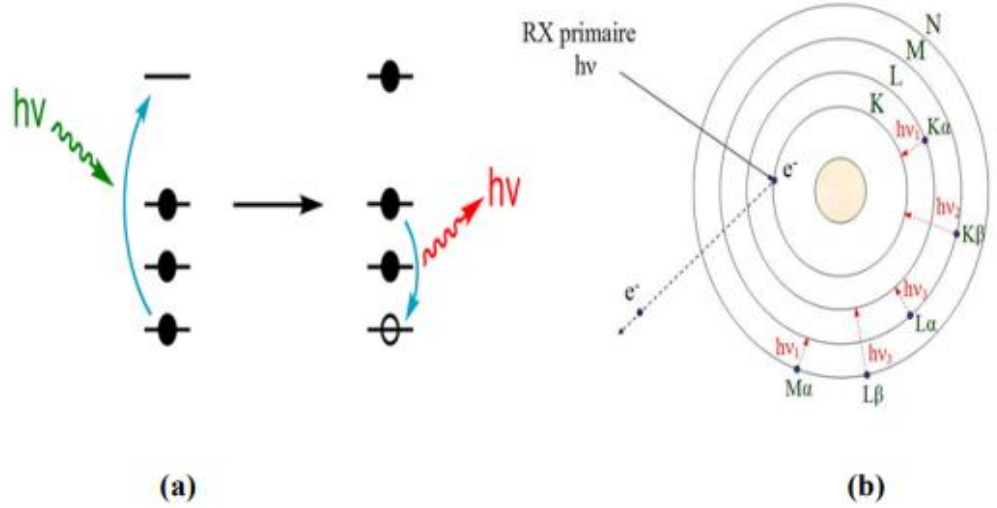



**Figure II.2:** *Schematic diagrams of X-ray fluorescence spectrometry radiation [JAC 52] (a): Schematic representation of X-ray fluorescence, (b): Excitation of fluorescence radiation.*

### II.1.4.2. Methods of measurement

When a photon of sufficient energy, in order to tear an electron from the inner layers of an atom, strikes an atom, there is a certain probability that it will tear off one of its electrons, thus ionizing the atom, which is then in a very unstable state [NEV 87]. The atom will regain its stability by "internal reorganization": electrons from the layers that are more external than those from which the electrons were ejected will come to fill the holes, releasing the energy difference between the layers from which the electron was ejected and those from which it was ejected, in the form of high-energy electromagnetic radiation (X photons).

**a) Primary fluorescence:**

The primary fluorescence results directly from the effect of the incident primary radiation on the element considered. It is the main contribution to the emission of an element in a sample [ELA 02]. It is the only one responsible for the X-ray fluorescence emission for pure element samples, or when the fluorescent element is associated with other elements having fluorescence lines of longer wavelength. A beam of X-rays (usually emitted by an X-ray tube, or by a radioactive source) arrives on the sample to be treated. Only X-ray photons with a certain energy will excite the "deep" layers of the element considered.

**b) Secondary fluorescence:**

In fact, the fluorescence of one of the elements of the sample, excited by the incident beam, can play in turn the role of an "exciting" incident beam on another element of the sample, and thus cause a secondary fluorescence [RON 99]. This last element will emit a total fluorescence which will be the result of the primary fluorescence due to the incident beam and the secondary fluorescence due to the other elements.

**c) Inter-element fluorescence:**

in practice, several elements are present in the sample, which is the usual case. The operation followed consists in introducing matrix coefficients translating the relative absorptions for the different elements. These can be a linear combination of concentrations, or more complicated, which present the case of a secondary fluorescence that is characterized by a reinforcement [JAC 52], i.e., the element that is at the origin of the secondary fluorescence seems to absorb more, unlike the other element, which is strengthened [ELA 02]. This is expressed by a reinforcement factor. The calculation of the emitted fluorescence, is relatively simple in the case of incident radiation, becomes significantly more complicated in practice for polychromatic radiation, but the principle remains the same [GOL 92]. In addition, two major simplifications are possible: in the case of a very thin sample, or on the contrary very thick.

## I.1.5. Apparatus

### I.1.5.1. Primary radiation source

An XRF consists of an X-ray source composed of an X-ray generator and a beam produced by a radioactive source. In the case of a fragile sample, it is often preferable to use indirect radiation: the X-ray tube bombards a target, and it is the fluorescent spectrum of this target that bombards the sample [NF EN ISO 13196].

The sample is excited by a radiation called "quasi monochromatic", which requires an adapted apparatus allowing to modify the treatment of the results since it is necessary to take into account the influence of the target used. This method also greatly simplifies the

calculation of matrix effects, since a radiation called quasi-monochromatic is used [EBE 76]. This is called "polarized X-ray fluorescence".

A microprobe has an electron gun that creates a focused beam. This beam can be deflected and thus point to a specific area of the sample, or it can be scanned, which gives an elementary mapping [GOL 92]. The characteristic energy of the source of the primary radiation, the wavelength of the X-rays for XRF or the acceleration voltage of the electrons for EPMA, will depend on both the depth to which the sample will be likely to give a response and the range of the spectrum on which this response will be given [EBE 97]. The measured spectrum will be limited to energies below the characteristic energy of the primary source.

### I.1.5.2. WDS and EDS spectrometers

The wavelength dispersive analysis is commonly called WDS. The X-ray photons of a single wavelength are selected by Bragg diffraction on a single crystal, downstream of which is a photon detector that can be a proportional gas counter. It is the angle between the path of the secondary radiation and the surface of the crystal that determines the reflected wavelength [ASTM E1621 - 13 and ASTM E2119 - 16].

The detector must be placed on the symmetrical path of the secondary. The basic principle of selecting a single wavelength has its advantages and disadvantages [BEK 06]. The advantage lies in the fact that only the peak of interest is displayed on the detector, which is especially interesting for the measurement of trace elements whose X-rays are not interfered with by the characteristic radiation of an element, which can be a million times more intense than that of the trace element [BEK 06]. To achieve this, it is necessary to vary the angle of inclination of the crystal while repositioning the detector [ELA 02].

This implies the setting in motion of a precision mechanical system. Similarly, if one simply wants to measure several lines of the spectrum, it will be necessary to sequentially adjust the goniometer - this is the name given to this mechanical system - to the different positions of interest [EBE 76]. It is also possible to equip the apparatus with several WDS spectrometers, each of them being adjusted to the positions of interest for the analysis considered; this is called a "multichannel" apparatus, Figure II.3. The figure represents an example of a part of a spectrum obtained by microprobe on a glass [ELA 02].

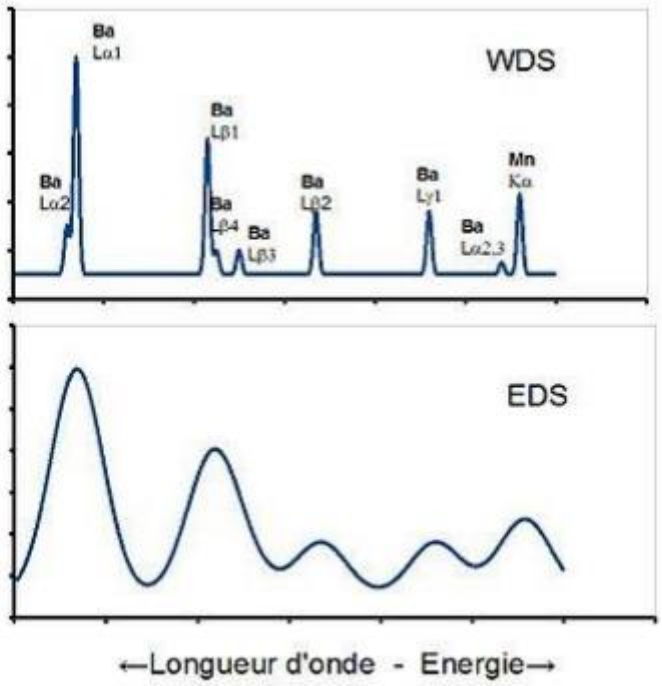



**Figure II.3:***Comparison between spectra obtained by WDS and EDS [ELA 02]*

A Bragg diffraction spectrometer can have an excellent spectral resolution provided that the geometry of the diffraction is respected and that a restriction concerning the emittance is made: the emissive surface or the angle of aperture of the paths must be small [ELA 02]. For XRF, the source is never very small, a plane crystal and a collimator, also called "Soller slits", which is formed by thin parallel copper strips, are used; this ensures that all the secondary rays that strike the analyzer crystal have the same direction [RON 99], as shown in Figure II.4a. With a curved crystal, a thin slit is placed between the sample and the analyzer crystal, the slit, the crystal and the detector being on a circle called "Rowland's circle" [BEK 06].

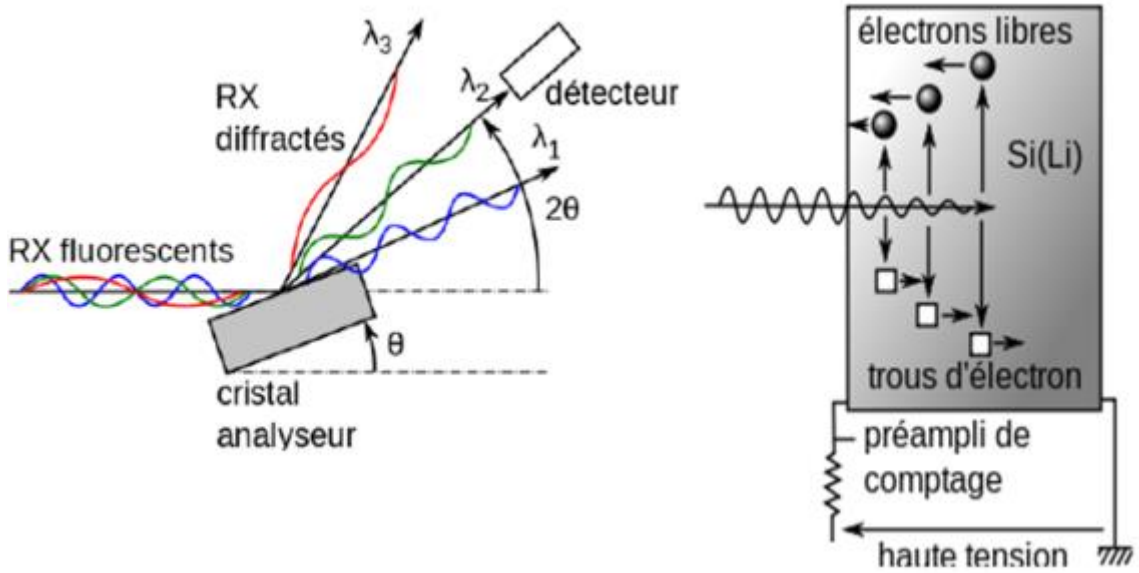



**Figure II.4:***Schematic diagram of spectrum analysis principle [RON 99]*
*(a) Separation of wavelengths by diffraction on a crystal, (b) Principle of the solid detector*

In contrast, in the case of energy dispersive analysis, X-ray photons of all wavelengths of the fluorescent spectrum reach the detector. The latter must convert each photon into a pulse of electric charge that is proportional to the energy of the photon [EBE 97]. It should be mentioned that semiconductor detectors, more precisely of the type SDD Si(Li), are good candidates for this function, since the generation of electron-hole pairs in the dead zone of a P-i-N Junction is proportional to the energy of the photon.

Where WDS spectrometry involves sophisticated mechanics and crystal formation technology, EDS spectrometry will involve better detection electronics to discriminate the levels of the electron charge pulses [GOL 92]. Electronic thermal noise is a priori a limiting factor in spectral resolution, but it is not the fundamental physical limitation of the technique.

The spectral resolution of EDS is therefore much poorer than that of WDS, as shown in Figure II.4b where two EDS and WDS spectra of the same sample are shown [RON 99]. There are many applications that do not require very high spectral resolution. The lower

resolution of EDS is compensated for by the inherent ability to measure the entire spectrum simultaneously [GOL 92].

### I.1.6. Application

If the basic physics of X-ray fluorescence spectrometry is common to all branches of the family, the practical problems of analysis are specific to each branch. For example, the preparation of samples from liquid materials does not make sense in microanalysis which is by definition a local analysis [ELA 02].

X-ray fluorescence spectrometry is an elemental analysis method, thus allowing the determination of pure element concentrations. However, the elements are often present in the form of compounds (molecules, polyatomic crystals) in the initial material [BEK 06].

X-ray fluorescence spectrometry (XRF) allows the determination of the elemental composition of the sample in a qualitative and quantitative way, i.e., to determine the nature and the quantity of the atoms which are present in the sample. This technique is commonly used in the metallurgical [ISO 9516], mining, petroleum [EN ISO 14596] and cement industries [ISO 29581-2], because it has the following advantages:

- ✓ Multi-elemental analysis: almost all elements are analyzed except for very light elements;
- ✓ It is a technique adapted to the analysis of various and complex matrices (powders, minerals, oils, cements, polymers, glasses, ceramics...) to determine the elemental composition.

Despite all these advantages, X-ray fluorescence spectrometry, unlike X-ray diffraction, does not allow access to the crystalline structure of materials (However, in most cases, this parameter is important [EBE 76]. For example, it largely conditions the performance of a cement...)

It may therefore be desirable to present percentages of compounds rather than of elements. For example, concentrations in oxide are often expressed, in particular in geochemistry or for cements [MAS 96]. The concentration of compounds is calculated, in general by the analysis software, from the concentrations of elements and chemical formulas. If the element is not present in the indicated form, then the compound expression is wrong, although the analysis is correct.

X-ray fluorescence spectrometry is an elemental method of analysis, so it is possible to determine the concentrations of pure elements [RUS 78]. However, the elements are often present as compounds (molecules, polyatomic crystals) in the initial material. It may therefore be desirable to present percentages of compounds rather than of elements. One often expresses for example concentrations in oxide, in particular in geochemistry or for cements [BAR 94].

For example, iron can be present in the form of pure iron Fe, or iron oxide [EBE 97]. X-ray fluorescence spectrometry cannot determine the form in which the iron is bound, and the bound elements are generally light and therefore not measurable, so the expression in the form of a single compound may be wrong. This can result in a sum of concentrations less than or greater than 100%. Another example is shown in Figure II.5.

For example, iron can be present in the form of pure iron Fe, or iron oxide: wustiteFeO, hematite Fe2O3, magnetiteFe3O4, but also sulfide: pyrrhotite FeS, marcasite FeS2, or sulfate: ferrous sulfate FeSO4 and ferric sulfate Fe2(SO4)3 [EBE 97]. X-ray fluorescence spectrometry does not allow the determination of the form in which the iron is bound, and the bound elements are in general light and therefore not measurable, so the expression in the form of a single compound may be false. This can result in a sum of concentrations less than or greater than 100%. Another example is shown in Figure II.5 [ELA 02].

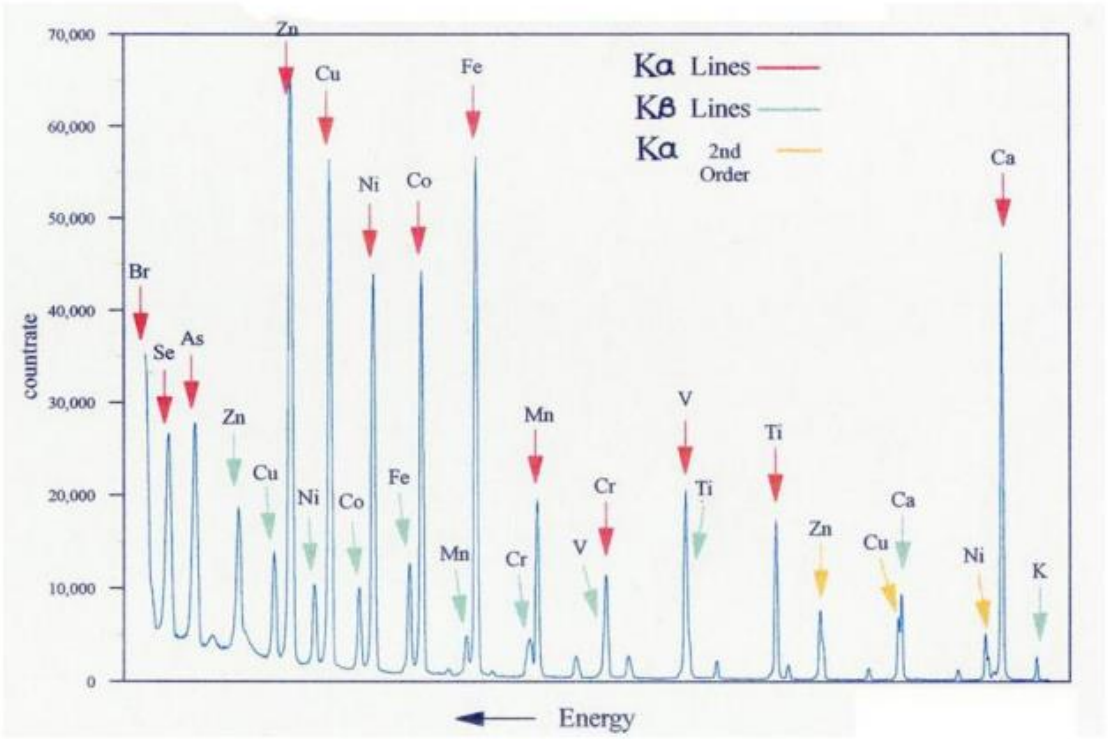



**Figure II.5:** *Typical X-ray fluorescence spectrum by energy dispersion [ELA 02]*

## II.2. X-RAY DIFFRACTION

### II.2.1. Definition

X-ray crystallography or X-ray diffractometry (XRD) is a method of physicochemical analysis based on the diffraction of X-rays on matter [ANR 85]. It is only effective on crystalline matter [PAS 87] (minerals, metals, ceramics, crystallized organic products), but not on amorphous matter (liquids, polymers, glasses); however, amorphous matter scatters X-rays, and it can be partially crystallized, so the technique can be useful in these cases [KEV 89].

### II.2.2. Principle

The technique of X-ray diffraction is based on the fact that a crystal lattice consists of a stack of families of parallel and equidistant lattice planes [HEI 81]. The interaction of a beam of X-rays with matter gives rise to an emission in all directions of radiation of the same wavelength and coherent phase. This scattering phenomenon leads to waves of very

low amplitude in the case of an atom [CUL 56]. On the other hand, scattering by matter on a set of atoms leads to an interference of coherent waves scattered by each atom. This wave, called "diffracted", depends on the atomic structure of the matter [GOL 03].

When a monochromatic X-ray of wavelength "λ" is incident on the lattice planes of a crystal with an angle "θ", diffraction occurs only when the distance covered by the reflected rays of successive planes, differ by an integer number of wavelength [JEN 96]. This is "Bragg's law" (Figure II.6).

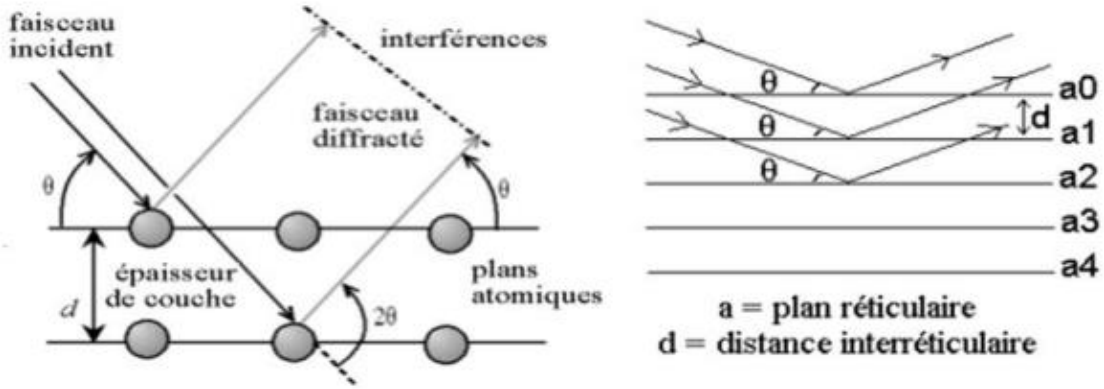



**Figure II.6**: *Principle of Bragg's law [JEN 96]*

Where "d" is the inter-reticular distance separating two planes of the same family (h, k, l), "n" is the diffraction order. By changing the angle "θ", the conditions of Bragg's law are satisfied for different distances d. By plotting the angular positions and intensities of the resulting diffraction peaks, a diagram is obtained which is characteristic of the sample [GUI 80]. The diffractogram is formed from the addition of the individual patterns The diffractogram is formed by the addition of the individual patterns, since it can show a mixture of different phases.

### II.2.3. Description

#### II.2.3.1. Production of X-rays

X-rays are produced by the interaction of electrons with a metallic target. The electrons are emitted by a filament heated by the Joule effect [JOH 65]. These electrons are accelerated by a potential difference and directed towards a metallic target (anode or anticathode) [EBE 76] . The production of X-ray photons is due to the rapid deceleration of the

electrons on impact with the target; Figure II.7. Note that the efficiency of X-ray production is low, typically around 0.2%; the rest of the energy is dissipated as heat.

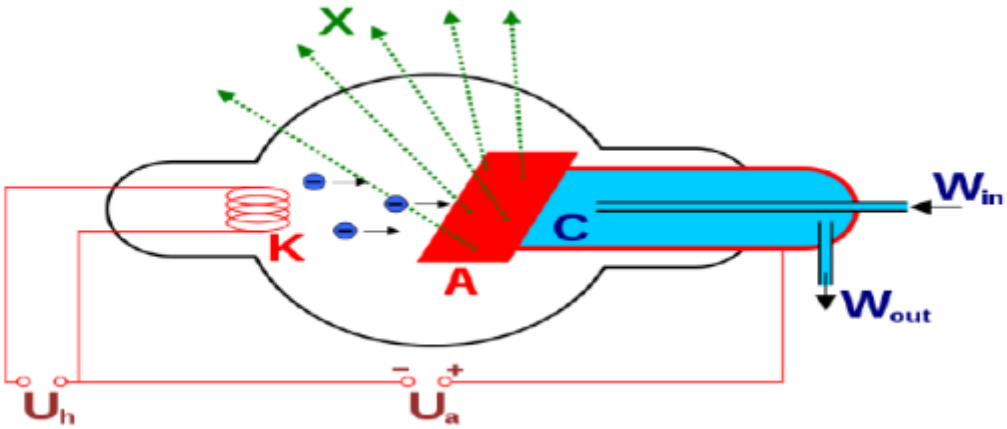



**Figure II.7:***X-rays production [KEV 89]*

It is therefore necessary to evacuate this heat (need for a cooling system) and to use target materials that are good thermal conductors and have a high melting point (refractory metals: tungsten, molybdenum or very good conductors: copper). X-rays are electromagnetic radiation like radio waves [MAU 78], visible light, or infrared. However, they can be produced in three very specific ways:

a) **By orbital changes of electrons coming from the electronic layers;** X-rays are produced by electronic transitions involving the inner layers, close to the nucleus [EBE 89]; the excitation giving the transition can be caused by X-rays or by electron bombardment, this is notably the principle of X-ray fluorescence spectrometry and the Castaing microprobe;

b) **By electron acceleration** (acceleration in the broad sense: braking, change of trajectory); two systems are used [CHI 98]: Braking of electrons on a target in an X-ray tube; electrons are extracted by heating a metal filament, the cathode, and accelerated by an electric voltage in an evacuated tube. This beam is focused so as to bombard a metal target made of tungsten or molybdenum, called the anode or anti-cathode [JOS 92]. The slowing down of the electrons by the atoms of the target causes a continuous braking radiation, called Bremsstrahlung. The curvature of the trajectory in particle gas pedals: this is the so-called "synchrotron" radiation, which is a special case of continuous braking radiation.

c) **By a radioactive source:** the decay of certain isotopes produces X-rays. This makes it possible to have compact sources, but the production of X-rays is not very stable and cannot be controlled, which introduces a dispersion in the measurements.

### II.2.3.2. Line spectrum

When producing X-rays with an X-ray tube, the spectrum is composed of continuous radiation (Bremsstrahlung) on which are superimposed lines specific to the anode used and which are due to the phenomenon of fluorescence [NIC 66]. In contrast to the continuous spectrum, the line spectrum depends essentially on the material in which the X-rays originate, be it the anticathode of an X-ray tube or the absorbing material used in a fluorescence experiment [SCH 82].

The spectral lines are totally independent of the accelerating voltage of the electrons in the first case, or of the frequency of the incident radiation in the second case [CAU 88]. They depend only on the chemical elements that compose the material.

The comparison of these line spectra with the corresponding absorption spectrum allows their interpretation to be completed. The series that make up the spectrum are placed in the vicinity of each absorption discontinuity [LAN 96]. Each series is designated by the same letter as the discontinuity with which it is adjacent. The spectral terms are confused with the wave numbers corresponding to the absorption discontinuities. The frequency of each emission line is equal to the difference of the frequencies of two absorption discontinuities [ALE 02].

### II.2.3.3. Intensity of the lines

The Miller indices (h, k and l) are a way to designate the orientation of the crystal planes in a crystal. They are relative integers, prime between them. The expression of the intensity diffracted by the planes (h k l) of an element of volume of powder $\Delta V$, one must take into account the following particularities [JAC 13]:

- ✓ Have a single-phase product and in sufficient quantity.
- ✓ The crystallite size must be small and homogeneous for this, sieves are used.
- ✓ The surface of the sample must be as flat as possible while avoiding preferential orientations by using a side-filling sample holder.

In a crystal, an atom is bound to the others by various forces. Its equilibrium position is the one that minimizes its energy [GOL 92]. A perturbation results in an oscillation of the atom around this equilibrium position. In particular, thermal agitation modifies the diffractive power of the atom, Figure II.8.

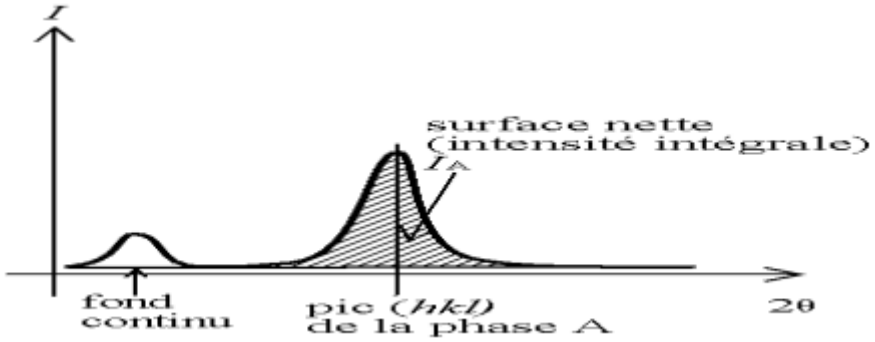



**Figure II.8**: *X-ray intensity [GOL 92]*

### II.2.3.4. Measurement methods

The Bragg equation has two experimentally variable parameters, θ and λ. To realize the diffraction conditions on a family of planes (h k l), only one of the two parameters can be arbitrarily fixed. The choice of the variable parameter determines two groups of X-ray diffraction methods [MIC 97]:

- ✓ **Laue method**: fixed θ, variable λ
- ✓ **Rotating crystal method or powder method**: λ fixed, θ variable

#### II.2.3.4.1. Laue's method

It is applied to single crystals (or large grains). The sample is fixed and the crystal planes have a fixed orientation with respect to the incident X-ray beam. The beam being polychromatic, each crystal plane (h k l) that can give rise to diffraction will give a

diffracted beam (Figure II.9). This method is mainly used for the orientation of crystallographic orientation of single crystals [ERN 80].

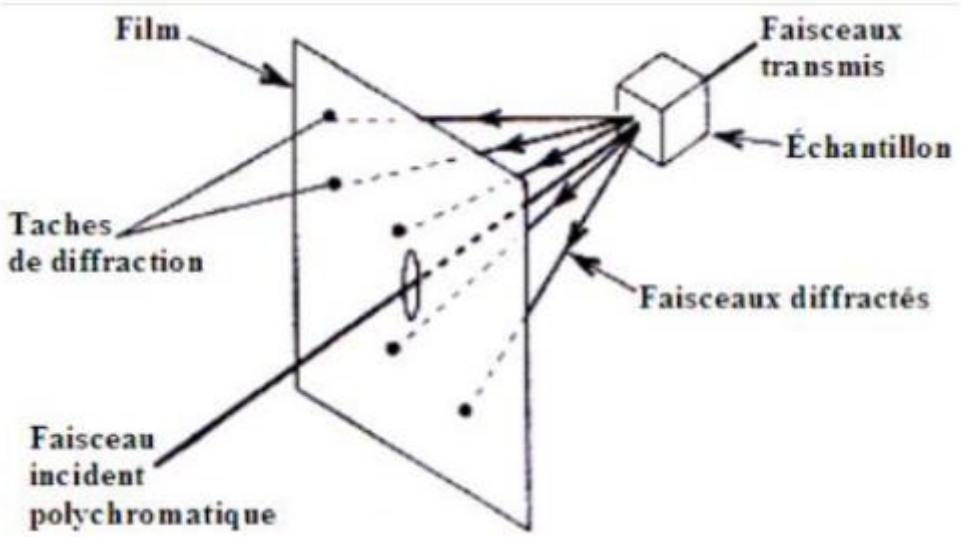



**Figure II.9:***Laue method [ERN 80]*

### II.2.3.4.2. Powder method

This method takes its name from the fact that the examined sample is reduced to a fine powder made of randomly oriented particles. The X-ray beam is monochromatic and diffraction will only occur when this beam makes an angle θ (solution of Bragg's law) with a family of crystal planes that can give rise to diffraction [MIC 97].

The particles being randomly oriented, there will always be a family of {hkl} planes giving rise to di

ffraction, so that all the diffractable beams are obtained simultaneously: these beams form cones of half angle at the apex 2θ (Figure II.10). This method can also be applied to a polycrystalline bulk sample formed of small grains which are equivalent to the particles of the powder.

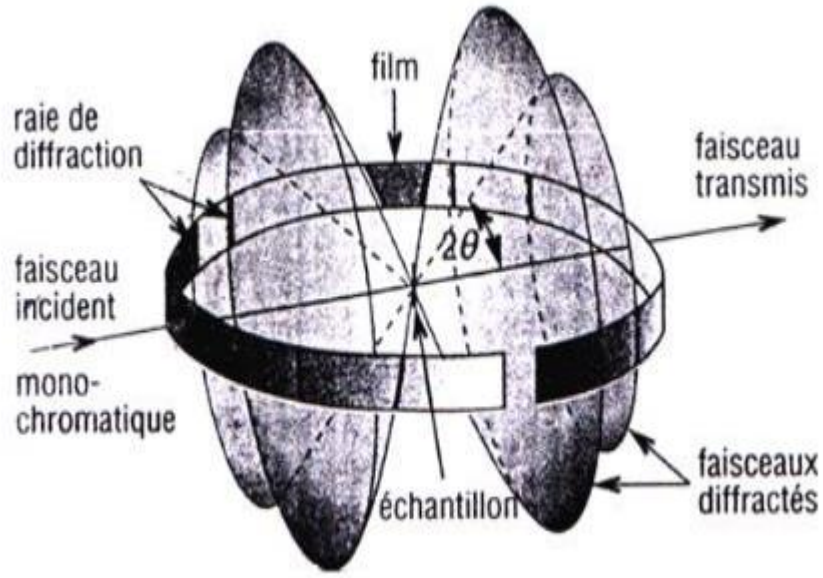



**Figure II.10:***Powder method [MIC 97]*

### II.2.3.5. Structure of minerals and X-ray Diffraction (XRD)

The mineral materials can be constituted of an amorphous or crystallized structure, depending on the degree of order of their atomic arrangement [ERN 80]. Figure II.11.1a illustrates the case of silica, which can be in the vitreous form (silica glass, silicagel, smoke of silica) or crystallized (quartz, cristoballite).

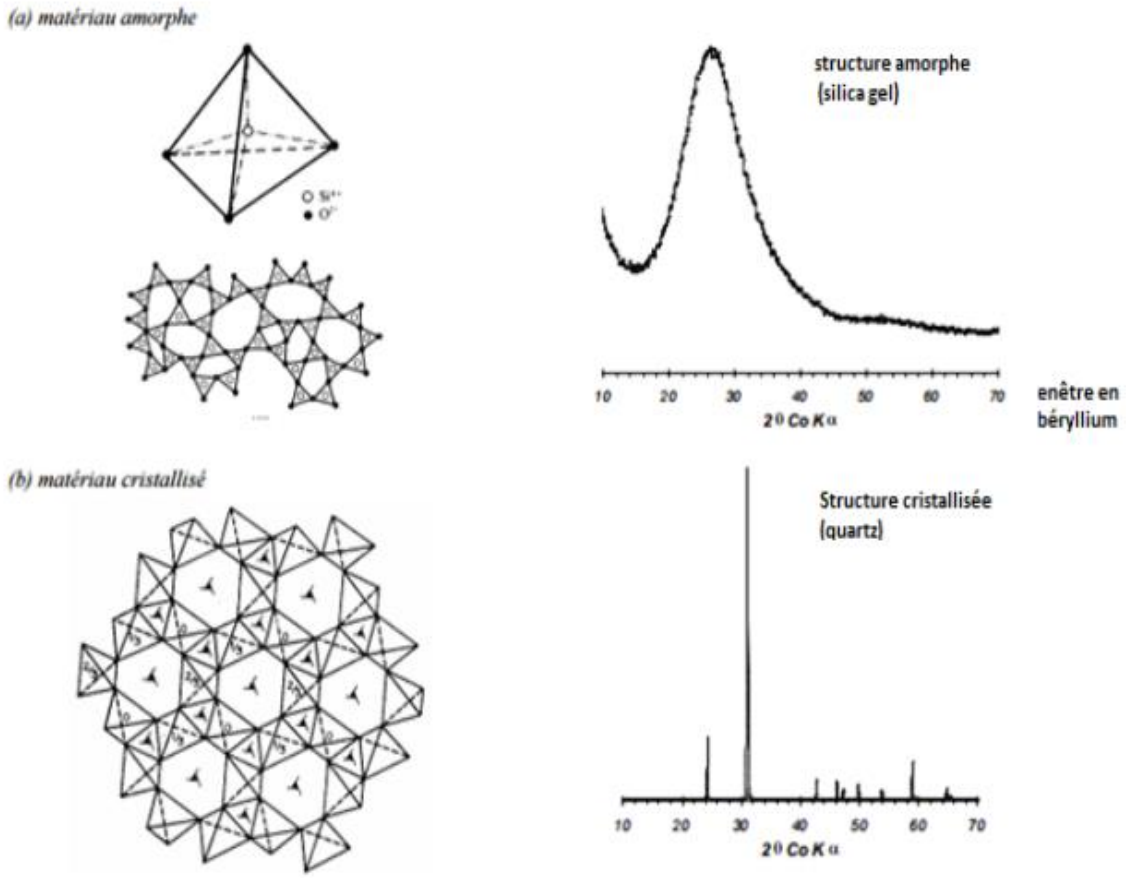



**Figure II.11:***Structures and X-ray diffraction patterns of silica in (a) amorphous [DOR 86] and (b) crystalline [PAS 65] forms*

### II.2.4. Apparatus

The measuring apparatus is called a diffractometer. There are several types of diffractometers, depending on the field of research (materials science, powder diffraction, life sciences, etc.) and on the type of structure: in fact, a distinction must be made between diffractometers dedicated to laboratories equipped with an "X-ray" source, and those dedicated to "synchrotrons", which use much more powerful light sources [CHA 72].

In the laboratory, the diffractometers proposed by the industry are usually complete solutions, i.e., including a diffractometer, an X-ray source, a video-microscope and an X-ray detector in a single secured unit. In synchrotron, diffractometers are available independently from other equipment, unlike laboratory diffractometers. It should be noted that the diffractometers dedicated to synchrotrons are much more complex and the number of light lines to be equipped is much lower than in laboratories [MOU 03]. Nevertheless, it should

be noted that many diffractometers in synchrotrons are designed and assembled by the technical teams of the synchrotrons themselves, which is rarely the case in laboratories.

The recording is done in 2θ varying from 2° to 80°. The X-ray source is fixed and when the sample holder varies by an angle θ, the detector varies by an angle 2 θ (Figure II.12).

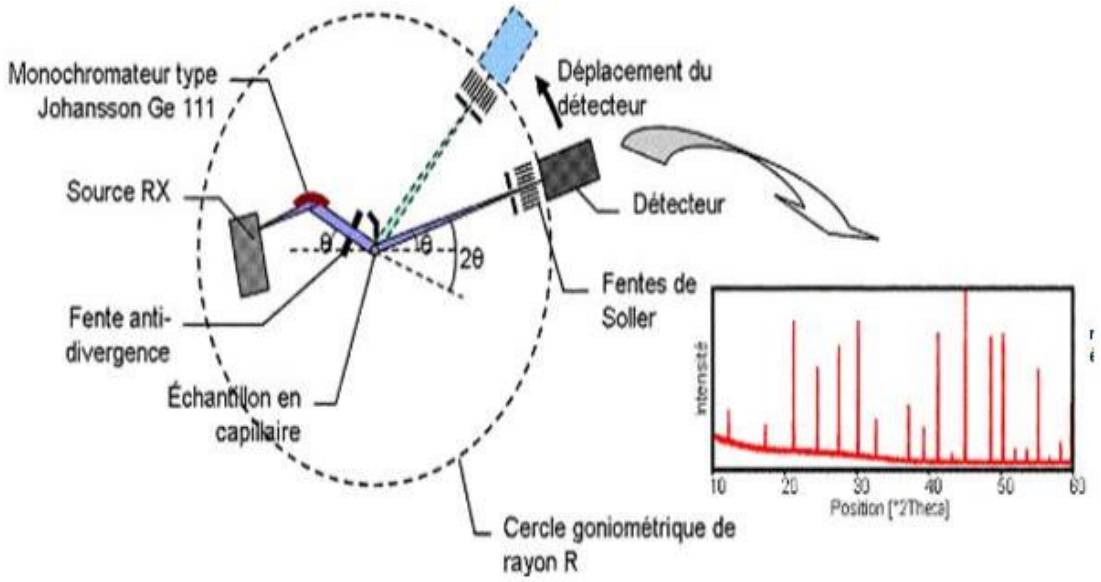



**Figure II.12:***Diagram of a diffractometer [MIC 97]*

The collected data form the diffraction pattern or diffractogram. X-ray radiation, like visible light or γ-radiation, is electromagnetic radiation [JAC 52]. It is characterized by a sinusoidal electromagnetic field which propagates at the speed of light.

### II.2.5. Application

X-ray Diffraction (XRD) allows qualitative control of the elemental composition and crystal structure of materials. It is often used to control the quality of the composition of cements, ceramics, crystalline powders (glass, pigment, clay, metals, carbonates, salts, crystallized organic substance...).

In a mixture, it is possible to determine the nature of each of the crystalline phases present, provided that the signature of each of these phases is known beforehand. The identification procedure is done in two steps, the first is to compare the peaks obtained with those contained in a database that includes several hundred thousand records. The collected data

form the diffraction diagram or diffractogram, Figure III.13. This step is followed by a validation step based on the chemical composition. The validation is carried out by the user who must have a good knowledge of the sample, in order to remove ambiguities and confusions.

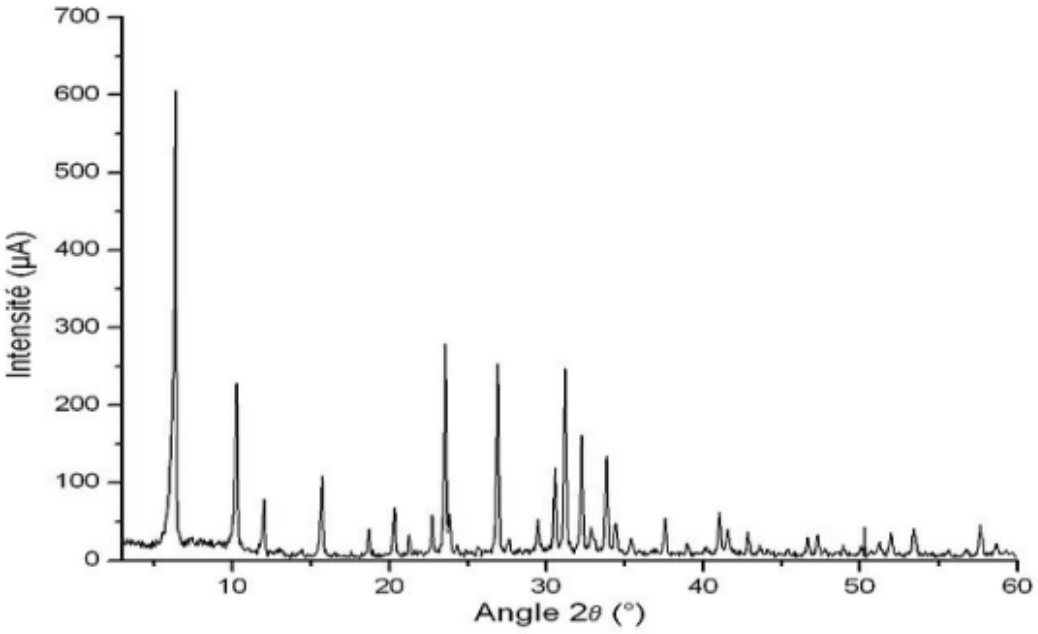



*Figure II.13: Example of powder diffractogram [EBE 89]*

This technique also allows to distinguish products with the same chemical composition but with different atomic arrangements. For example, calcium carbonates such as calcite and aragonite, with the same chemical formula (CaCO3), have different diffraction patterns. On the other hand, phases of different chemical nature but whose atoms are organized according to the same arrangement show great similarities, namely diffraction peaks located at the same angular positions.

To summarize, X-ray diffraction is based on the recording of a diffractogram and on the analysis of the peaks of this diagram, which makes it possible to characterize the crystallites present in the sample from the following elements [NEV 87]:

- ✓ Position of the peaks: qualitative analysis, identification of crystalline phases present
- ✓ Peak width: size and shape of crystallites, internal stresses
- ✓ Peak intensity: estimation of chemical composition, quantitative analysis, preferential orientation

The objects and materials studied in the laboratory by X-ray diffraction are mainly environmental materials (soils, sediments, rocks, suspended matter). The applications concern:

- ✓ Determinative mineralogy: identification of crystallized phases.
- ✓ Identification of clays (phyllosilicates).
- ✓ The follow-up of crystallochemical transformations during reactions (for example drying or hydration).
- ✓ The determination of the structure of the phase (s) contained in the powder by comparing the spectrum obtained with those contained in a database or deducing from an original model proposed. It will be possible to refine with a good precision the crystalline parameters and the atomic positions in a certain number of cases.
- ✓ the detection of the presence of impurities.
- ✓ the test of the crystallinity of materials.

Since diffraction occurs only on crystalline material, it is otherwise known as radio crystallography. For non-crystalline materials, it is called scattering. Diffraction is part of the elastic scattering methods. This method uses a beam of X-rays that meets the crystal causing the dispersion of the light beam in specific directions. By measuring the angles and intensity of the refracted rays, it is possible to obtain a three-dimensional image of the electron density in the crystal. From this density, the average position of the atoms in the crystal can be determined, as well as their chemical bonds, entropy and other information.

## II.3. SCANNING ELECTRON MICROSCOPY

### II.3.1. Definition

Scanning Electron Microscopy (SEM) is an electron microscopy technique capable of producing high resolution images of the surface of a sample using the principle of electron-matter interactions [GOL 92].

### II.3.2. Description and application

#### II.3.2.1. Principle of SEM

The principle of Scanning Electron Microscopy is based on the observation of matter using an electron beam after having put the sample under vacuum [MIC 97]. The primary electrons, coming from the electron gun, strike the surface of the sample; they are scattered in an "elastic" and "inelastic" way, the influenced zone taking the shape the shape of a pear [MIC 97], Figure II.14.

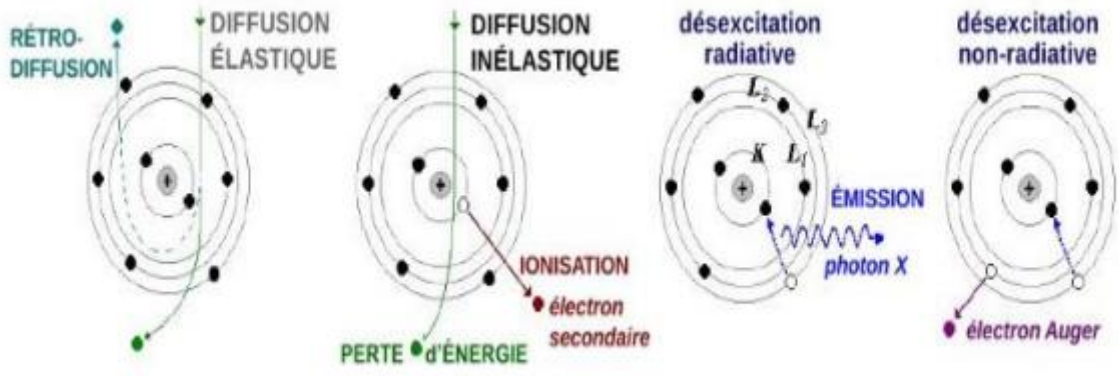



**Figure II.14:***Electron scattering [MIC 97]*

Some electrons are scattered in an elastic way, i.e., by keeping their kinetic energy: they are the electrons known as "retrodiffused". During the encounter with matter, some primary electrons give up part of their kinetic energy to the atoms, causing the ionization of the atom by the ejection of an electron referred to as "secondary" [MIC 97].

The energy of the secondary electrons being low, only the electrons coming from the surface layers come out of the material. The atom thus ionized de-excites, an electron from a higher layer will occupy the empty place, which provokes either the emission of an X proton (secondary emission), or an Auger electron [GOL 92], Figure II.15.

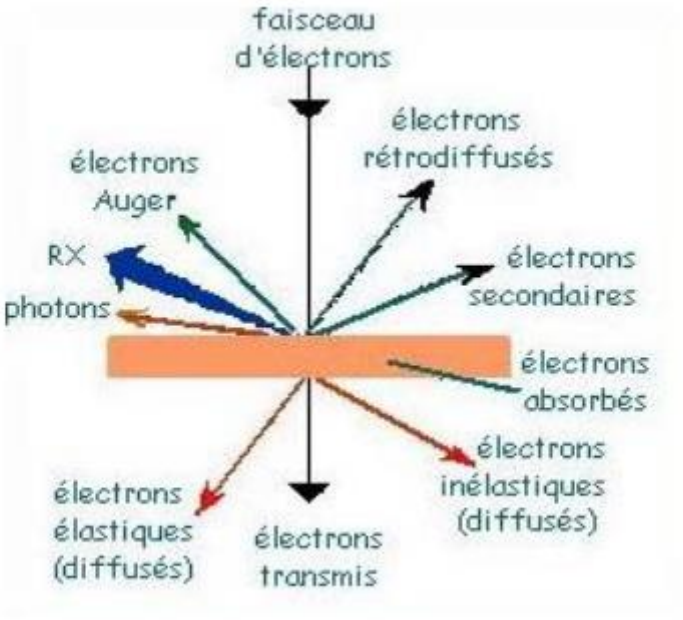


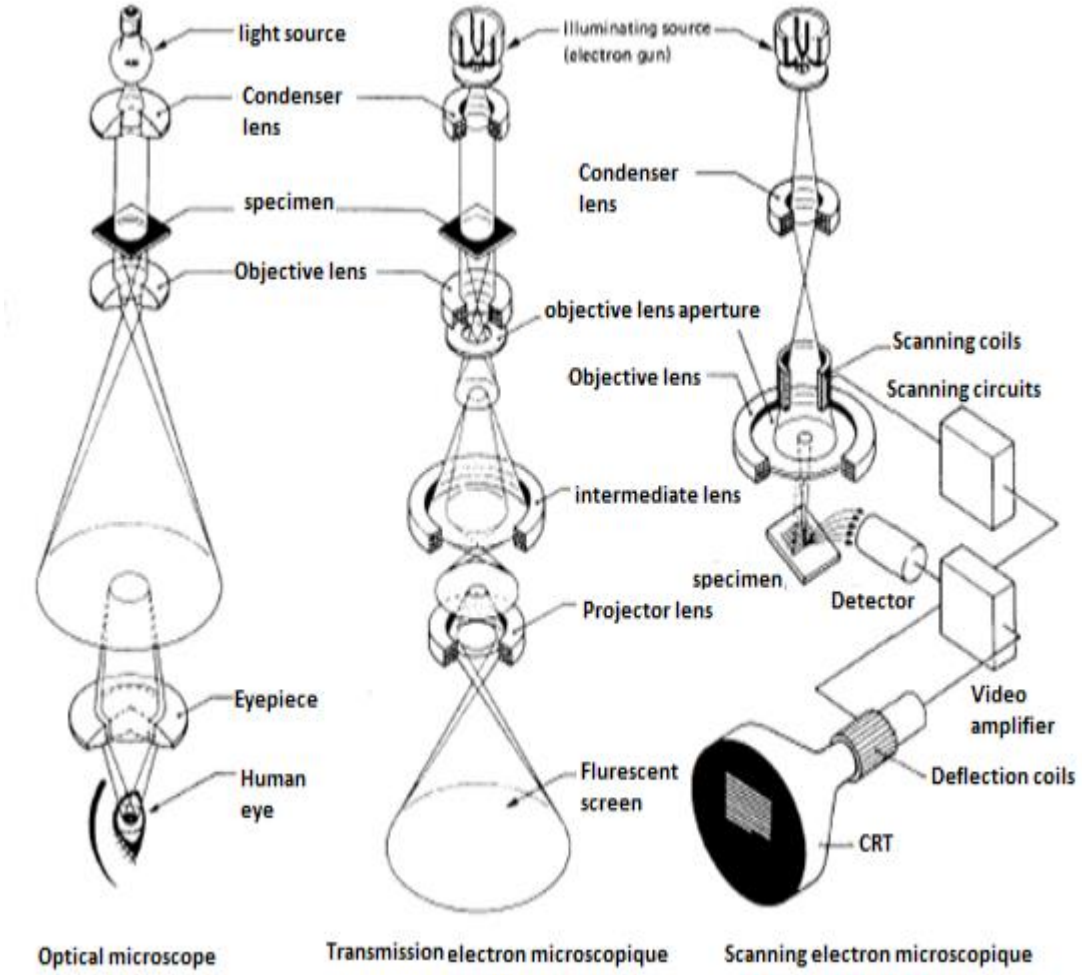

**Figure II.15**: *Set of radiations that can be emitted during the interaction between the beam and the sample [GOL 92]*

### III.3.2.2. Apparatus

The scanning microscope provides a reconstructed image of a probe responsible for exciting the sample, and a panoply of detectors allowing a mapping of the different signals emitted during de-excitation [GOL 03].

The diagram in Figure II.16 shows the general operating principle of the SEM, with the different elements of the column. We note the following in order [JAC 13]:

- ✓ The column formed by: the source, filament + Wehnelt or tip + extracting anode;
- ✓ The accelerating anode;
- ✓ The condenser(s), with their diaphragms.




The objective with its diaphragm and the system of scanning and control of astigmatism consists of adding alignment coils as well as corrective optical elements not represented the sample, which actively participates and determines a lot of information in the formation of the image, with the stage allowing the movements. The detectors and the visualization device, coupled to a column by the scanning system, are shown in Figure II.17 [GOL 03].

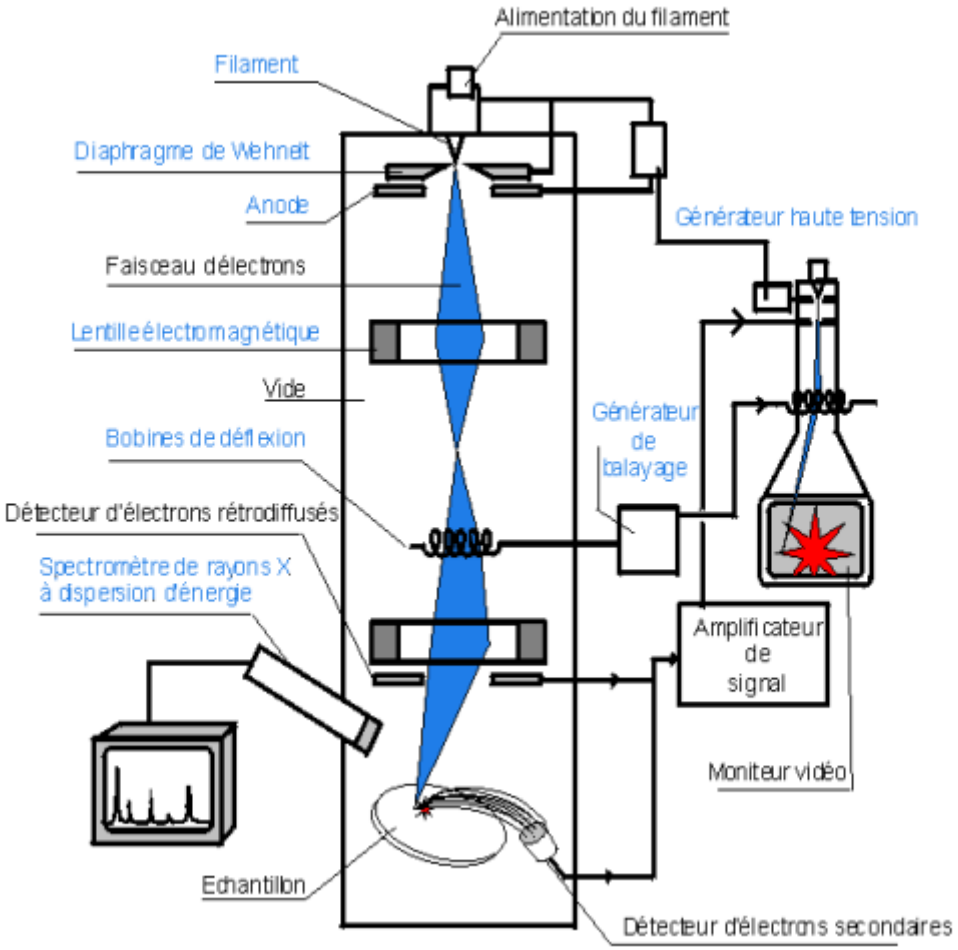



**Figure II.17:***Constituent elements of a SEM [GOL 03]*

### II.3.3.3. Type of electrons

**a) Secondary electron**:

This is the process of interaction or inelastic scattering.The secondary electron can undergo an inelastic interaction with a free electron between two atomic planes of a crystalline material analyzed [CHI 98]. The analysis of these electrons allows to obtain a characteristic image of the surface. These electrons represent a good tool for the observation of tool for the observation of the contours and morphology of the sample. Figure II.18a.

**b) Backscattered electron:**

This is the interaction process or elastic scattering. The incident electron is elastically backscattered. The image obtained is therefore a function of the chemical composition of the sample. II.18b. [CHI 98].

**c) Emission of a photon X:**

A photon of energy equal to the difference between the two emitted electronic energy levels. The vacuum of the superior layer is filled by another electron of a still superior layer with emission of a photon, Figure II.18c. The study of X-ray photons allows a quantitative analysis of the chemical composition of the sample. [CHI 98].

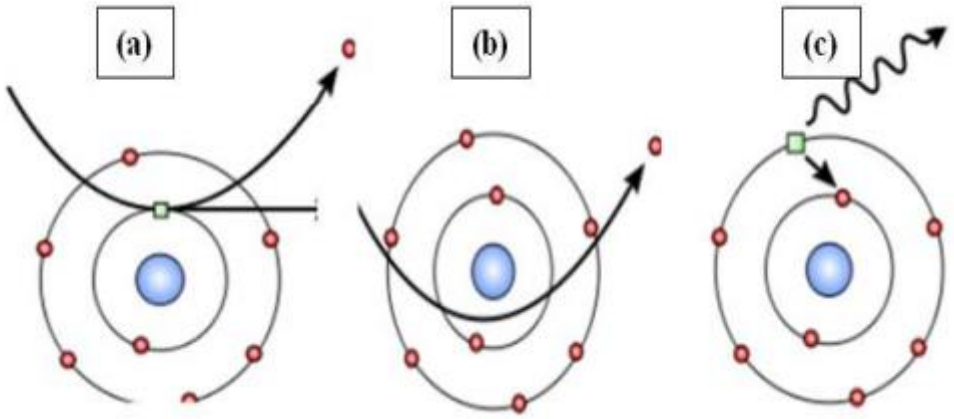



**Figure II.18:** Electron emission [CHI 98]

**(a):** Secondary electron emission, **(b):** Backscattered electron emission,

**(c):** Photon emission

### II.3.3.4 SEM image

The SEM image is a reconstructed image of: a probe, an electron beam, a scan of the surface of the sample [GOL 92], a detector retrieving the signal induced by this probe to form an image of deflection coils to move the beam and thus scan the sample, Figure II.19.

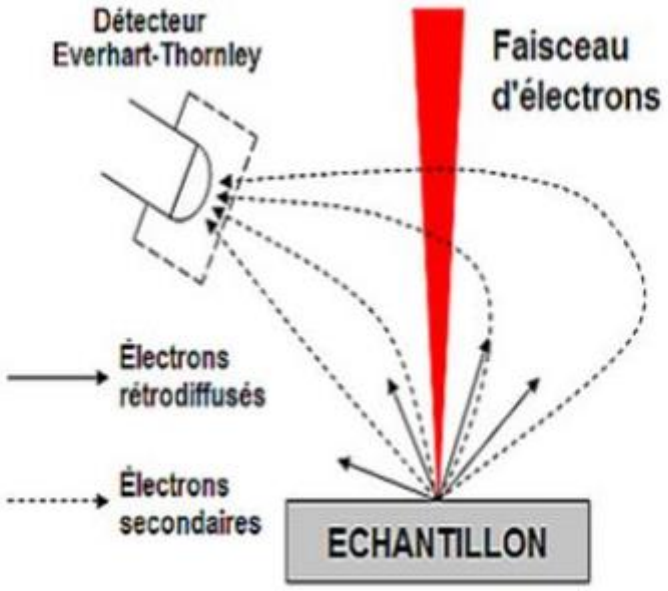



**Figure II.19:** *Composition of a SEM image [GOL 92]*

All recorded signals can be used to form an image. The basic SEM detector (E.T.: Everhart-Thornley) retrieves a mixture in variable proportion of secondary and backscattered electrons, its proportion depending on the primary energy, the sample distance, and the angle of incidence of the primary beam, of the observed material [GOL 92].

The detector is surrounded, for its collecting part, by an electric cage allowing the attraction of the considered electrons: we collect the secondary and backscattered electrons. The detection of secondary electrons is essential and it is this detection that allows us to obtain an image in "relief" with a SEM. This detector is still used today in modern SEM.

Other detectors allow to discriminate secondary electrons from backscattered electrons. The number of secondary and backscattered electrons emitted varies depending on the point of impact of the electron beam on the surface.

### II.3.3.5. SEM Image Contrast

The contrast of the image represents the local variation of the electronic emission. It comes from topographic, chemical and structural effects, governing backscattering and secondary emission [GOL 92].

The topographic aspects are generally the following (Figure II.20):

- ✓ **Tilt effect of the surface with respect to the incident beam (a):** in this first case the secondary emission intensity is minimal with respect to the beam normal, and maximal at grazing incidence [GOL 92], (Figure II.20a).
- ✓ **Edge and tip effect (b,c):** these two cases correspond to the secondary emission that is most intense on fine tips and edges and that appears from a highlight of these [GOL 92], (Figure II.20b).
- ✓ **Shading effect:** this case consists of mounting the detector laterally on the microscope used, which serves to hide all parts of the sample at the same angle. [GOL 92], (Figure II.20c).
- ✓ **Effect of sample composition (d):** this last case is characterized by the presence of elements of high atomic number. The contrast produced is visible if that of the relief is sufficiently soft or if the difference in atomic number is sufficiently large.

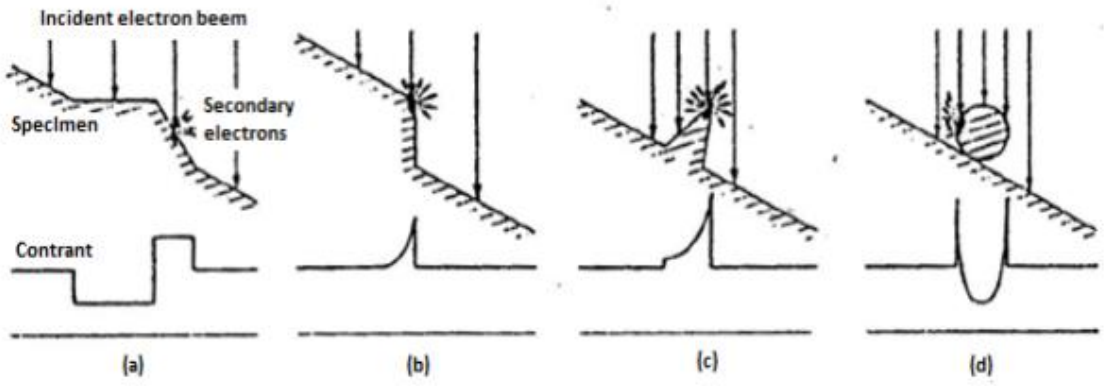



**Figure II.20:** *Contrast of a SEM image [GOL 92]*

*(a): Edge, (b): Tip, (c): Shadowing, (d): Sample composition*

### II.3.3.6. Magnification of the SEM image

The magnification of the SEM image is determined by the fraction between the area scanned on the sample, and the size of the final document on the observation screen.

It is to be noted that it is judicious to speak of the size of the observed field more of magnification [CHI 98].

**The secondary electron** image is characterized by a very good definition, up to magnifications between x 20,000 and x 50,000 in conventional, up to >500,000 in high resolution, with lateral resolution limits of 10 to 200 Å, depending on the sample, the apparatus and the observation parameters. Mounting the detector in the lens eliminates most of the backscatter, and provides a better-defined image of the surface, especially at low primary energy.

The backscattered electron image has a lateral resolution of about 0.1 mm at best. The use of a specialized detector allows to bring out a contrast which is a function of the atomic number of the elements present. A heavy element will give an intense signal and therefore a clear area; a light element will give a weak signal and therefore a dark area, in a way a mapping of atomic number. This mode of operation requires a flat surface to minimize the contrast due to the relief. It is then rich in possibilities, in particular as a basis for an image analysis of a sample of heterogeneous composition. On the other hand, the identification of the elements present requires the use of another method, typically X-ray microanalysis.

The SEM is a compromise device. It is interesting to distinguish between magnification and resolution in order not to confuse these two basic notions. These quantities are a function of several parameters such as the acceleration voltage and the primary current. It will therefore be necessary to determine the set of settings adapted to each sample.

We currently distinguish three types of SEM use [NEV 00]:

- ✓ **Basic imaging:** this type of use covers magnifications between x25 and x10,000 with an occasional maximum of x30,000 to x50,000, this is the case for geology, metallography, polymers and granulometry, etc.
- ✓ **Low vacuum imaging:** this second type is characterized by aqueous, fatty, high outgassing or insulating samples. The recorded magnifications are of the same order as for the case presented before.
- ✓ **The so-called high-resolution imaging:** this last case is characterized by magnifications that are in the range of x50,000 to x400,000. This type of image represents the case of nano-technologies, nano powders and semiconductors. This

type of instrument allows observations with very low primary energy, which gives a great richness of details, already at low magnifications (x2000 to x10.000).

In addition to these basic orientations, analytical (EPMA, CL) and structural (EBSD) aspects can be added to each of these types of instruments [NEV 00]. In spite of a wide range of coverage, these devices are very different, both in performance and in price [GOL 03]. Figure II.20, shows as an example the microstructure of a concrete obtained with SEM.

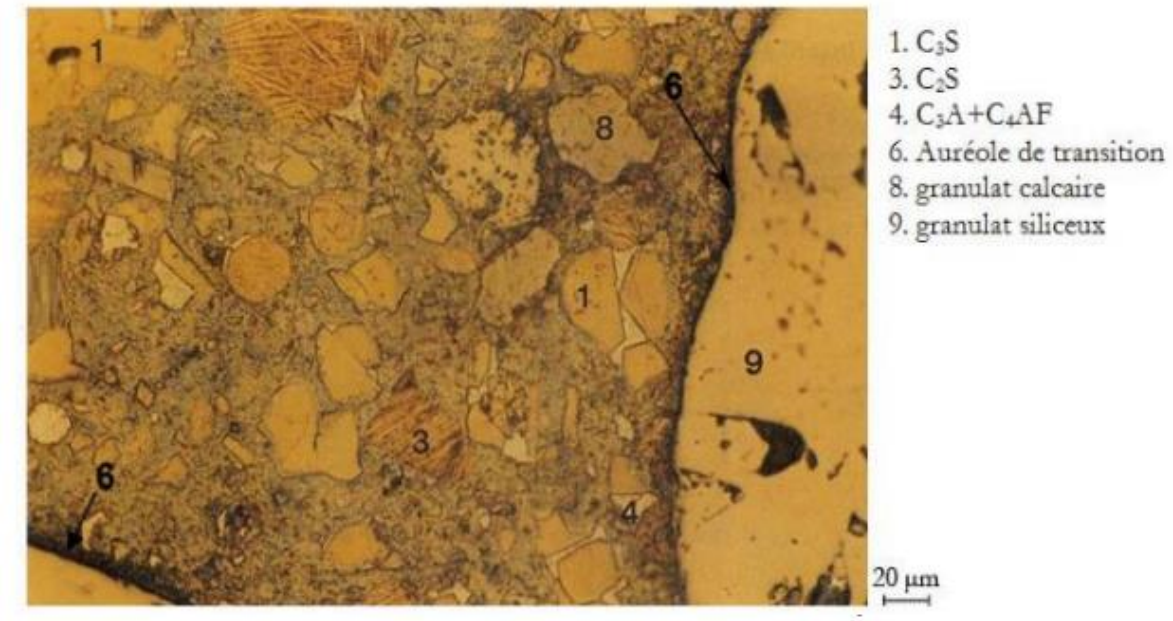



**Figure II.20:***Example of microstructure of a high-performance concrete (SEM) in the fresh state [CAS 04]*

# CHAPTER III

## III.1. Basic concepts

### III.1.1. Representative volume element

Before performing a mechanical test, we must define the characteristic volume element which is called the "representative volume" of the material used. This knowledge allows us to satisfy the assumptions of continuous media. We present in Table III.1, the characteristic volume elements depending on the type and size of heterogeneities and according to the type of material used [NEV 00].

**Table III.1:***Macroscopic volume elements*

| **Type of material** | **Type and size of heterogeneities** | **Characteristic volume elements** |
|---|---|---|
| **Metals and alloys** | Grain: 0.001 to 0.1 mm | 0.5 x 0.5 x 0.5 mm |
| **Polymers** | Molecule: 0.01 to 0.05 mm | 1 x 1 x 1 mm |
| **Wood** | Fiber: 0.1 to 1 mm | 10 x 10 x 10 mm |
| **Concrete** | Aggregates: ≈ 10 mm | 100 x 100 x 100 mm |

It is necessary to mention that the transformation of the "force-displacement" curves obtained into "stress-strain" curves is called "reduction," which gives so-called "rational curves" [ACK 05]. In Figure III.1, we provide examples of rational curves for different types of materials.

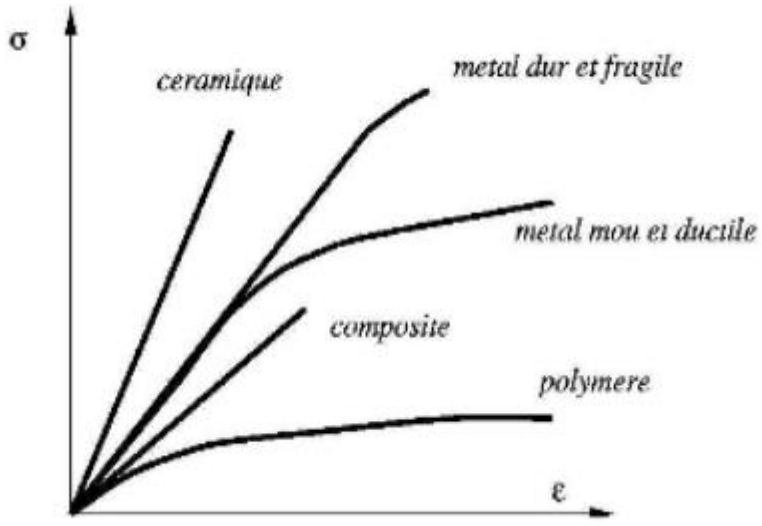



**Figure III.1:** *Typical stress-strain curves of different materials [ACK 05]*

It should be noted here that the rational curve relates two scalars "stress" and "strain", and not two tensors. Therefore, the choice of these two scalars depends mainly on the type of test and the type of material used.

### III.1.2 Strain rate

In fact, the strain rate can have a significant influence on the behavior of tested materials [ACK 04]. Therefore, we must use a strain rate as close as possible to that which will be presented later (see Table III.2) when performing a mechanical test.

For example, if the goal is to validate the creep resistance of a studied structure under the influence of its own weight, the strain rate to be considered will be very low [LIA 06]. On the other hand, if the objective is to validate its resistance to earthquakes, then this same strain rate may take a much higher value, and consequently, the behavior law to be used will not be the same. This leads to the presentation of the different types of mechanical tests, which can be classified according to the strain rate to be considered [ACK 05], Table III.2.

**Table III.2:***Characteristic times and types of tests*

| Characteristic time (s) | Strain rate | Mechanical regime |
|---|---|---|
| $10^{6}$ | $10^{-6}$ | Creep |
| $10^{4}$ à $10^{2}$ | $10^{-4}$ à $10^{-2}$ | Quasi-static |
| 1 | 1 | Intermediate |
| $10^{-2}$ à $10^{-4}$ | $10^{2}$ à $10^{4}$ | Dynamic |
| $10^{-6}$ | $10^{6}$ | Impact |

For example, if we take the case of a uniaxial compression test, the test will be performed in a "quasi-static" regime, meaning that we should not take into consideration the effects of inertia when deducing the equilibrium equations [CHA 92]. This is explained by fixing the specimen on a fixed plate on one side and on a plate that can move at a given speed on the other side, which is relatively slow [ACK 05]. On the other hand, if the machine's plate can no longer reach the required speed, this machine will no longer be sufficient [ALV 03]. Therefore, the compression mechanical test will have to be carried out using another system called "Hopkinson bars," which is characterized by subjecting the specimen to an elastic compression wave that can come from the incident bar [AVI 00]. In this case, if we want to

analyze the test, we must take into consideration the effect of the mechanical inertia of the tested material, which can produce a force peak recorded at the beginning of the solicitation [AND 90]. In the rest of this chapter, we will limit our presentation to mechanical regimes for the following two cases: "quasi-static" and "dynamic."

## III.2 Static tests

### III.2.1 Direct tension test

#### III.2.1.1 Definition

The direct tension test is the most commonly used way of determining the mechanical behavior of a given material under the effect of a progressive solicitation characterized by a low or moderate loading rate [BAR 92]. This test is determined by its ease of implementation and the wealth of information provided during the test. In addition, it allows for the study and identification of the different physical mechanisms of plastic deformation [BAR 82]. This latter allows for the major forming process of various materials used in most of the manufacturing processes in the mechanical industry [BAR 92].

#### III.2.1.2 Principle of the test

The direct tension test consists of subjecting a standardized specimen to a tensile force, usually up to its rupture [NF P18-401]. To be noted that the temperature at which the test is to be performed is at room temperature (20°C), unless otherwise specified. The test specimen is taken from the material to be characterized and machined to standardized dimensions to ensure better comparison of tests performed in different laboratories [ASTM C 490-90].

There is a type of specimen for each type of material to be studied [EN 13286-42]. Specimens of the material in the form of cylindrical or prismatic bars with a calibrated central section of constant cross section "S0" and length "L0" connected at each end to two larger section heads [ACK 04] are fixed in a tensile testing machine, as shown in Figure III.2.

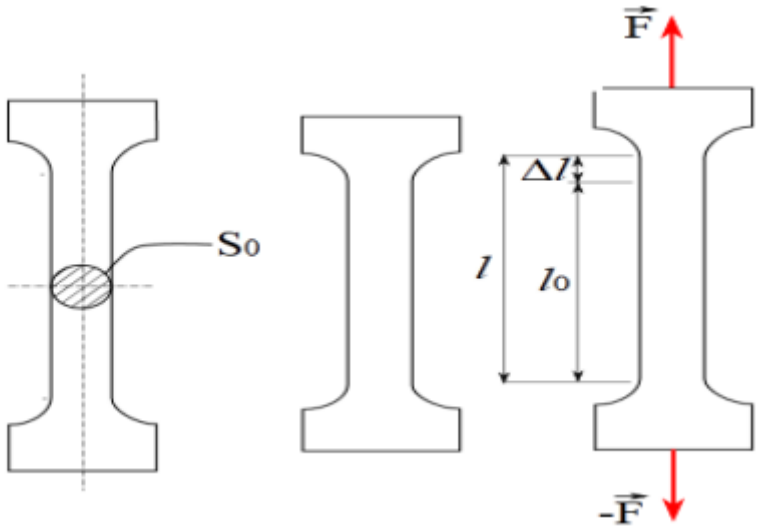



**Figure III.2:** *Operating diagram of a tensile testing machine [ACK 04]*

### III.2.1.3. Apparatus

The test is carried out using a tensile testing machine, Figure III.3, which consists of a rigid frame equipped with a fixed crosshead to which one end of the test specimen is attached; the other end of the specimen is attached to a movable crosshead [BAR 76]. The total movement of the movable crosshead is provided either by a hydraulic control or by worm gears. There are devices that can be attached to the tensile testing machine to measure the tensile force or load and the elongation of the specimen at any given moment [BAR 82].

The test consists of slowly and progressively applying a tensile force to a specimen held tightly between the two jaws of the machine used. This produces a stress-strain diagram, as shown in Figure III.3.

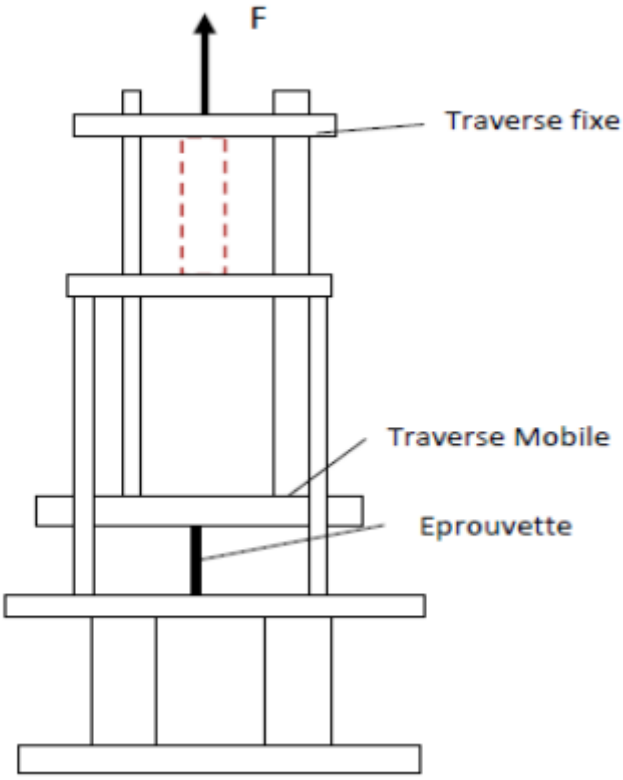



**Figure III.3:** Operating diagram of a tensile testing machine [BAR 76]

The tensile strength "Rm " is defined as the maximum stress reached during the tensile test. The yield strength "Re" corresponds to the stress at which the tested material begins to deform plastically. In practice, although this definition is simple, the limit to be reached is difficult to appreciate because the transition from the elastic domain to the plastic domain of a given material occurs progressively.

The difficulty in reading this limit can lead to erroneous interpretations from one laboratory to another. To address this issue, we must determine another stress called the "conventional yield strength" corresponding to 0.2% (Re 0.2%). The latter defines the stress at which a plastic deformation of 0.2% is measured in order to facilitate reading and obtain more consistent results from one laboratory to another. [NEV 00].

The Young's modulus or longitudinal elastic modulus, denoted "E", defines the measurable constant that relates the tensile (or compressive) stress to the onset of deformation of an elastic material [RIL 72]. The Young's modulus describes the mechanical behavior of the tested material depending on the type of test (tensile or compressive) [PIN 03].

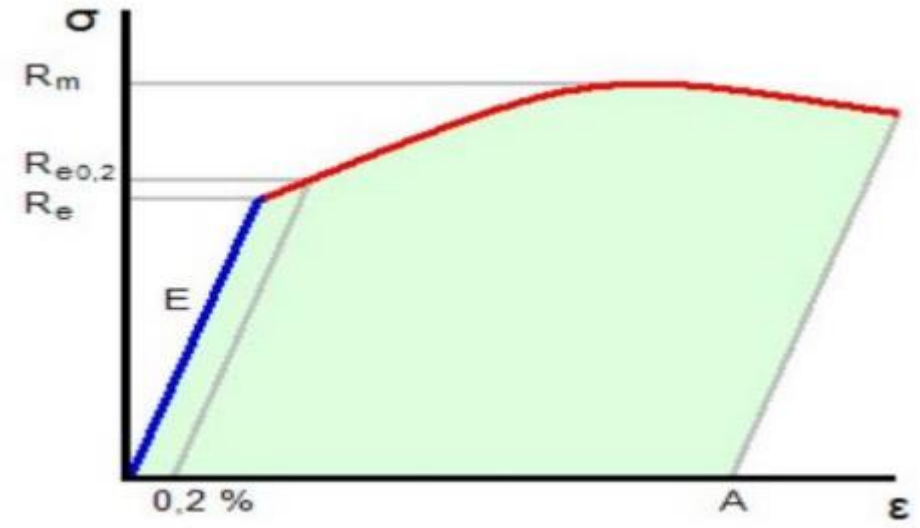



**Figure III.4:***Stress-strain diagram for tensile testing [FRA 95]*

We can also define certain characteristics determined by the tensile test:

- ✓ **Rigidity**: This characteristic is mainly measured by the Young's modulus. The higher the modulus, the stiffer the material, as shown in Figure III.5a. It is determined based on the energy deduced through the bonds between the atoms or molecules that make up the material [NEV 00].

- ✓ **Strength**: This is a characteristic that defines the maximum stress that the tested material can withstand before it breaks. This strength is determined based on the intensity of the bonds formulated, as well as the shape of the tested pieces or its defects [ACK 04].

- ✓ **Ductility**: This is the characteristic that corresponds to the ability of a material to deform permanently before it breaks. In this case, the higher the elongation at rupture, the more ductile the material is considered to be. Conversely, when the permanent deformation is very small or zero, we will speak of a brittle material.

  At this stage, it is necessary to note that we can find a brittle material that can have a very high strength [ACK 05], as shown in Figure III.5a.

- ✓ **Toughness**: This is a property that represents the amount of energy absorbed at the point of rupture by a material. It characterizes the resistance to sudden propagation of cracks [ACK 04]. Its value is equal to the area under the stress-strain curve. This characteristic is important for materials such as ceramics, as shown in Figure III.5b.

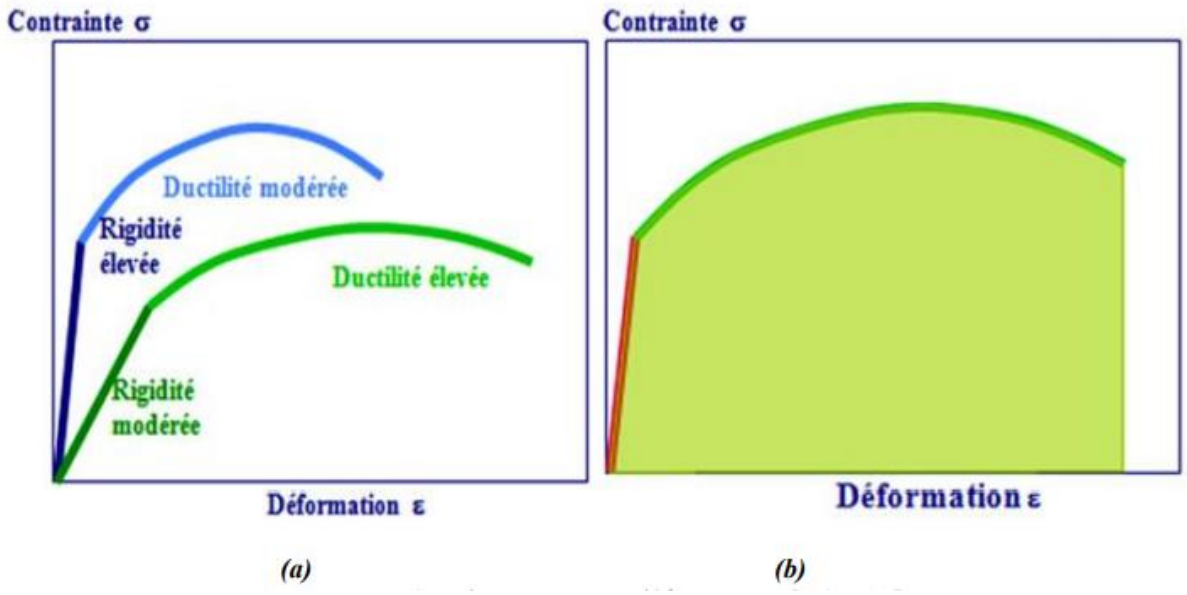



**Figure III.5:***Stress-strain curve [ACK 04]*

*(a) Two materials with different rigidities and ductilities (b) Toughness = area under the curve*

If we analyze the stress-strain curves of a set of materials, we can find three possible behaviors, as shown in Figure III.6.

**a) A brittle behavior:** in this behavior, the material fractures when the deformations are purely elastic. Consequently, the material does not present a plastic deformation domain [NEV 00]. As an example, we present brittle materials such as ceramics, glass, and thermosetting polymers, Figure III.6a.

**b) A ductile behavior:** the ductile behavior is characterized by a plastic deformation called "irreversible" accompanying the elastic deformation, which is reversible. This type of behavior is presented by most metals and alloys and some

thermoplastic polymers [ACK 05]. Note that metals are ductile materials characterized by low elastic limits. Therefore, various procedures are followed in practice to increase the elastic limit of metals, Figure III.6b.

c) **A nonlinear elastic behavior:** this behavior is characterized by an elastic deformation that is not proportional to the load that causes it [ACK 05]. Such behavior represents some materials such as some thermoplastic polymers and elastomers, Figure III.6c.

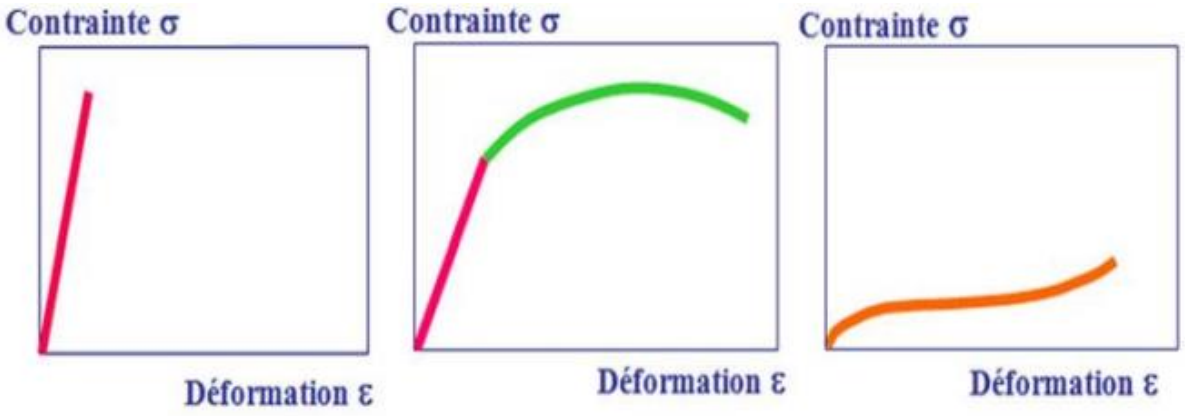



**Figure III.6:** *Stress-strain diagram in tension for different materials [DOM 01] (a): brittle behavior, (b) ductile behavior, (c) non-linear elastic behavior.*

### III.2.2. Compression Test

#### III.2.2.1. Definition

The most common test performed on a material is measuring its compressive strength, partly because this test is easy to execute and also because many characteristics of the material, but not all, can be qualitatively related to its strength [NEV 00]. However, the main reason for this test is the intrinsic importance of compressive strength in structural design [TOR 96]. By definition, compressive strength is the capacity of a material or structure to withstand the loads that tend to reduce its size by compression (crushing), as opposed to tensile strength, which is a resistance to elongation (bursting), and shear strength, which is mainly a resistance to torsion (twisting) [LEM 03].

### III.2.2.2. Test Principle

The compression test consists of subjecting a cylindrical, cubic, or prismatic specimen placed between the plates of a press to two opposing axial forces. If the material under study is ductile, rupture cannot be achieved with this test [VIT 96], Figure III.7. The compression test is mainly used to determine the breaking stress of brittle materials that are difficult to machine for a tensile test [BAR 92].

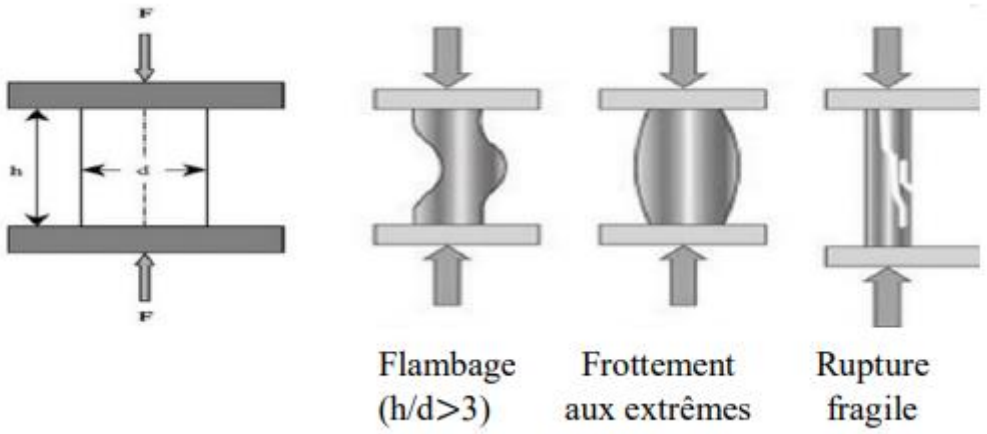



**Figure III.7:***Simple Compression Test [BAR 92]*

The specimen is compressed, and the deformation under various loads is recorded [CHA 99]. The compressive stress and strain are graphically represented in the form of a stress-strain curve, which is then used to determine the elastic limit, the proportionality limit, the elastic-plastic transition point, the stress at the flow threshold, and (for some materials) the compressive strength [CHA 92]. Standard compression tests are described in standards [ASTM C-773] (high-strength ceramics), [ASTM E-9] (metals), [ASTM E-209] (high-temperature metals), and [ASTM D-695] (plastic materials).

Although compression tests are simple and easy to perform, they are rarely conducted in comparison to tension tests [NEV 90]. This can be explained as follows: for ductile materials, both tests (tension and compression) provide almost the same values for the Young's modulus, yield strength, and flow limit [REY 05]. In addition, it is impossible to obtain the ultimate strength for these materials, since the specimen hardly breaks, but rather widens as the compressive force increases, taking on the shape of a barrel. The same applies to the ductility properties that cannot be obtained for ductile materials. Therefore, compression tests are often performed on brittle materials for which the compressive strength is much more important than the tensile strength [LAR 88].

### III.2.2.3. Apparatus

The machines used for compression testing are similar to those used for tensile testing; often the same machine can be used to perform both tests. The compression machine must allow at all times to evaluate the distance between two determined points of the specimen or the distance between the contact surfaces of the machine, which consists of:

- ✓ a fixed platen;
- ✓ a movable platen;
- ✓ a mechanism for driving uniform motion at controlled speed;
- ✓ a force indicator;
- ✓ a deformation indicator.

During compression testing, the sample shortens and widens. The relative deformation is "negative" in the sense that the length of the sample decreases [NEV 87]. Compression also tends to amplify the lateral irregularities of the sample and, beyond a critical stress, the sample may buckle and the deflection may increase until buckling [BAR 82]. Note that the faces of the specimen (in contact with the machine plates) must be well polished, flat, parallel, and perpendicular to its axis, so that the loading is axial and friction forces are minimized, Figure III.8.

The results of the compression test can be affected by different parameters: the type and dimensions of the specimen, the type of mold, the preparation of the test faces on which compression is exerted, the rigidity of the press, and the speed of application of the load [BAR 96]. For all these reasons, the tests must be carried out according to a precise procedure without deviating from the imposed specifications [NEV 95].

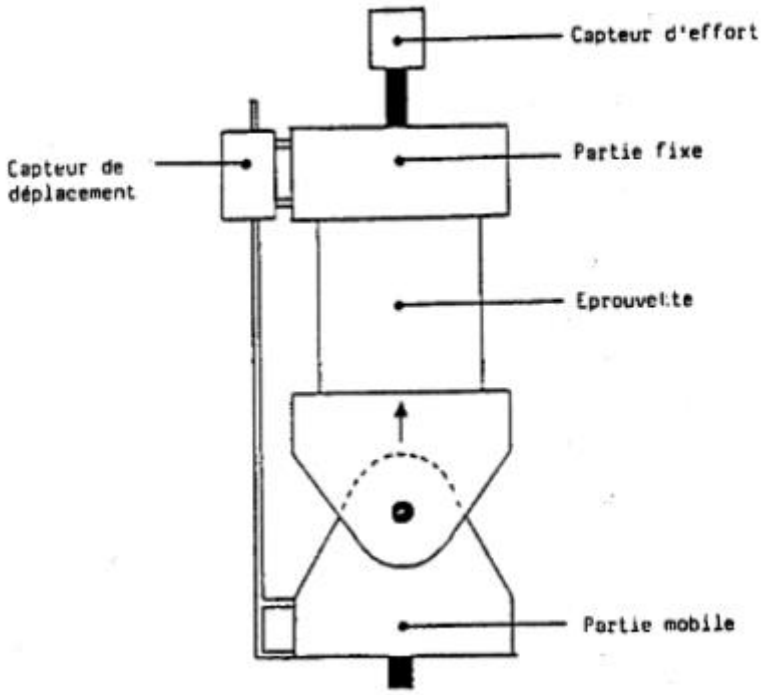



**Figure III.8:***Simple Compression Test [BAR 82*]

In practice, professionals calculate stress relative to the initial surface area of the cross-section (at the beginning of the experiment, before deformation), neglecting the fact that stress actually applies to the deformed cross-section (widened, laterally bulged before rupture) under the effect of the load [LAR 82].

As we have already seen, the shape of the curve describing the behavior of the material under compression depends on the nature of the material itself. For example, ductile metals never break, so for such materials, we must end the test after obtaining significant plastic deformations or once the maximum load developed by the machine is reached. For semi-ductile metals and brittle metals, the ultimate strength can be reached and therefore calculated. It should be mentioned that this latter category, which includes semi-ductile metals, may or may not present a yield plateau, depending on the conditions of processing of the metal of the tested specimen, while brittle materials do not present one, for which only the ultimate stress can be calculated under compression.

Concrete and ceramics generally have much higher compressive strengths than tensile strengths [NF EN 12390-3]. Conversely, composite materials [ISO 14126], such as epoxy

matrix composites reinforced with glass fibers, tend to have lower compressive strength than tensile strength. Metals [EN ISO 6892-1], on the other hand, often have similar strengths in tension and compression. The behavior of plastics in compression [ISO 604] is quite complex from a molecular standpoint, and general laws of behavior cannot be stated [ISO 527-1]. Therefore, compression tests will be performed to simulate real-life cases, and the results obtained will be used with great caution.

## III.3. DYNAMIC TESTS

Dynamic tests are experimental techniques that allow a material to be subjected to a high rate of deformation [ALV 03]. This category of tests is used in various applications such as seismic protection, deformation calculation in automobile crash tests, various processes related to metal shaping, particularly machining [AND 90], or practical problems related to armor piercing, which involve high rates of deformation (from 1 to $10^7$ s−1). In this chapter, we limit our presentation to the deformation range between 1 to $10^5$ s−1 [AVI 00].

For the conventional uniaxial tensile tests presented in the previous section, thedeformation rates reached range from $10^{-4}$ to 0.1 $s^{-1}$, which is clearly insufficient for some real-world deformation rates [LAL 86], such as those mentioned above. Therefore, it is necessary to have experimental techniques that allow the mechanical behavior of materials to be characterized under well-controlled conditions [BER 09]. We will present high-speed tensile tests here, which allow deformation rates to be varied from 1 to 400 s−1, approximately, and Hopkinson tests, whose deformation rate range is typically from $10^2$ to $10^5$ s−1[CAR 03].

### III.3.1. High-speed tensile test

#### III.3.1.1. Definition

High-speed tensile tests differ from quasi-static tests, firstly by the need to achieve very high cylinder speeds (up to 25 m/s), and secondly by the use of specific measurement techniques, both for load measurement and for elongation measurement of the sample [ROC 71, CHA 99]. For the latter, the non-contact extensometer is one of the most commonly used techniques.

### III.3.1.2. Principle of the test

The principle of this test is to illuminate the surface of the sample with two laser beams from the same source that interfere on the surface of the sample, thus forming fringes whose distance depends on their wavelength and the angle formed by the lasers [MON 94]. When the surface of the specimen moves within the interference volume of the two beams, the roughness provides a natural population of scattering sources that scatter the light with intensity modulated by the local intensity contrast. The frequency is then proportional to the surface displacement speed. By simultaneously measuring two points on the surface, their relative motion can be deduced, allowing the strain to be determined [ISO 17025].

### III.3.1.3. Apparatus

To achieve high displacement speeds (around 20 m/s), it is necessary to use systems called "servo-hydraulics". In addition, in order to obtain deformation speeds that are as constant as possible, it is necessary to start the movement of the mobile part without moving the specimen before the desired speed has been reached. This can be achieved by systems that allow the jaws to be tightened "on the fly" or by a device that drives the crosshead with a sufficient delay [PIR 01].

With such deformation speeds, it is necessary to have acquisition systems working at very high frequencies, of the order of a few MHz [NEC 04]. In addition, the force cell mounted on the frame will receive a strongly noisy signal from the mechanical waves propagating in the experimental device. This may require measuring stress directly on the specimen, using strain gauges applied to parts of the specimen that are deformed purely elastically (outside the useful section) [OWO 02]. Strain measurements must also be made directly on the specimen, using an appropriate extensometer system. Laser Doppler extensometers can be used in this case [PIR 93].


**Figure III.9**: *High-Speed Tensile Testing Device [GAR 08]*
*1) Doppler-effect laser cells for strain measurement 2) Sample*

The principle of this method consists of illuminating the surface of the sample with two laser beams from the same source that interfere on the surface of the sample, thus forming fringes whose distance depends on the wavelength of the beam and the angle formed by the lasers between them [THY 91]. When the surface of the specimen moves in the interference volume of the two beams, the roughness provides a natural population of scattering sources that will scatter light with an intensity modulated by the local intensity contrast [THO 97].

The modulation frequency is then proportional to the surface displacement speed. By simultaneously measuring two points on the surface, their relative motion can be deduced, allowing for determination of deformation. High-speed tensile tests require taking into account the propagation of elastic waves in the system, and therefore modeling each of the tests [EN ISO 6892-1].

## III.3.2. Hopkinson Test

### III.3.2.1. Definition

The Hopkinson test bar is a proven experimental test method in mechanical materials testing. Unlike quasi-static testing machines, the Hopkinson test allows for the determination of material properties under dynamic conditions. The method, involving the use of a Hopkinson bar, has shown its effectiveness in many applications thanks to increasingly powerful testing and measurement technology [COM 09].

### III.3.2.2. Test Principle

The principle of Hopkinson bars is to measure the elastic deformations on the incident bar and the transmission bar, both in contact on either side of the specimen to be deformed [DUP 07], as shown in Figure III.10.

The test consists of impacting an impactor to set it in motion, which allows it to strike an incoming bar. A compression wave is then created and transmitted along this bar [GMH 97]. This incident wave has the appearance of a square wave whose duration is equal to the time it takes to make a round trip in the impactor. It should be noted that it is preferable to use long bars in order to allow for one-dimensional elastic approximation as the situation becomes too complex to be usable in the case of two dimensions (2D) [TIM 69].

For Hopkinson tests, the assumption of stress homogeneity is not always tenable - especially at higher speeds - and it is necessary to take into account the propagation of waves in the sample when dealing with transient regimes [CAR 03].

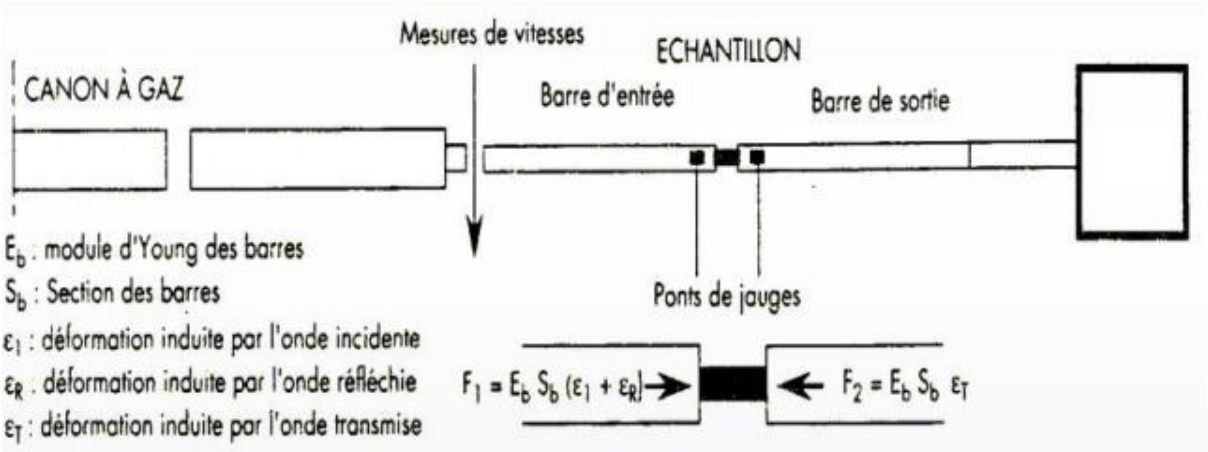



**Figure III.10**: *Schematic of the Hopkinson compression test principle [DUP 07]*

### III.3.2.3. Apparatus

Hopkinson bars (compression bars), also called SHPB (*Split Hopkinson Pressure Bar*), are mainly used to characterize the dynamic behavior of materials, that is, their behavior in case of impact. Indeed, many materials, such as metals and plastics, resist more strongly when deformed more quickly, as can occur, for example, in accidental situations.

The device must therefore allow very brief tests, typically of a duration of less than one millisecond, on samples of the material under study. A strain gauge is glued to the middle of each of the bars, which are made up of an input bar and an output bar. These gauges record the longitudinal deformations associated with the mechanical waves that propagate in the bars [COR06]. The measurements made at the level of the gauges must be virtually transported to the faces of the bars to deduce the forces and displacements at the level of the sample faces, by integrating the velocities, Figure III.11.

The shortening of the sample is calculated by time integration of the velocity [BER 09]. To do this, we measure the waves using strain gauge points placed:

- ✓ at the middle of the input bar, to separate the incident and reflected waves well. It is necessary to assume that the waves do not disperse, or to predict the dispersion while knowing that the dispersion equation for an infinite elastic cylinder has been known since the 19th century.
- ✓ a few diameters after the sample for the output bar so that the wave is well established.

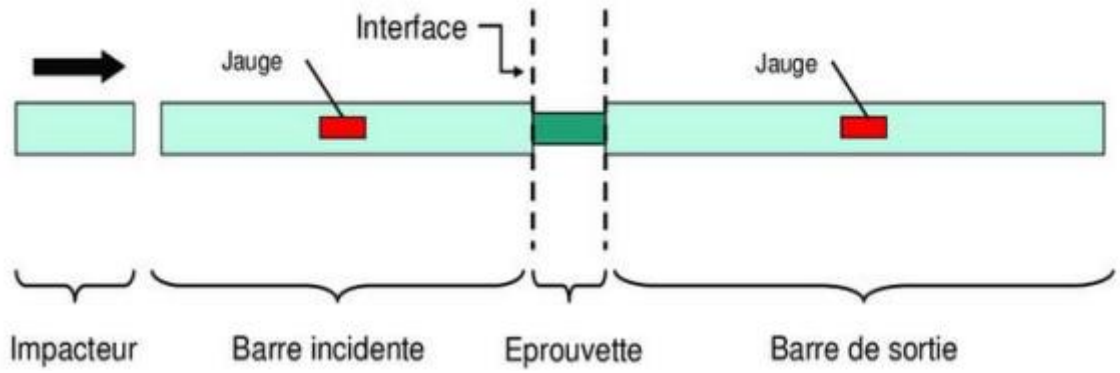



**Figure III.11:***Hopkinson test [COR 06]*

## III.4. DEFORMATION MEASUREMENTS

Extensometry is a method that includes different techniques for measuring deformations, which are necessary for obtaining certain physical quantities such as Young's modulus, elongation at rupture, and elastic limit, or for comparing experimental results with numerical results [BER 88]. Extensometry is also widely used for the active monitoring of infrastructure such as bridges, dams, or large buildings, as it allows for anticipating their aging and thus planning necessary maintenance operations for safety purposes [BUR 97].

There are two types of measurement: contact extensometry and non-contact extensometry.

### III.4.1. Measurements with contact

#### III.4.1.1. Strain gauges

##### III.4.1.1.1. Definition

A strain gauge is a small resistive element that is bonded to a test specimen at the point where deformation measurement is desired [GIB 97]. It consists of a fine wire wound in a preferred direction and bonded to the specimen through an insulating carrier, as shown in Figure III.12. When the specimen is subjected to loading, its deformation is transmitted through the adhesive and carrier to the gauge [JEA 01]. A change in resistance results, and the strain gauge is used to measure the deformation of a structural element by translating it into an electrical resistance change [GIB 97].

It should be noted that a strain gauge measures strain, while a stress gauge measures stress. Strain gauges are mistakenly referred to as stress gauges because strain is measured to determine stress using elasticity theory [JEA 01].

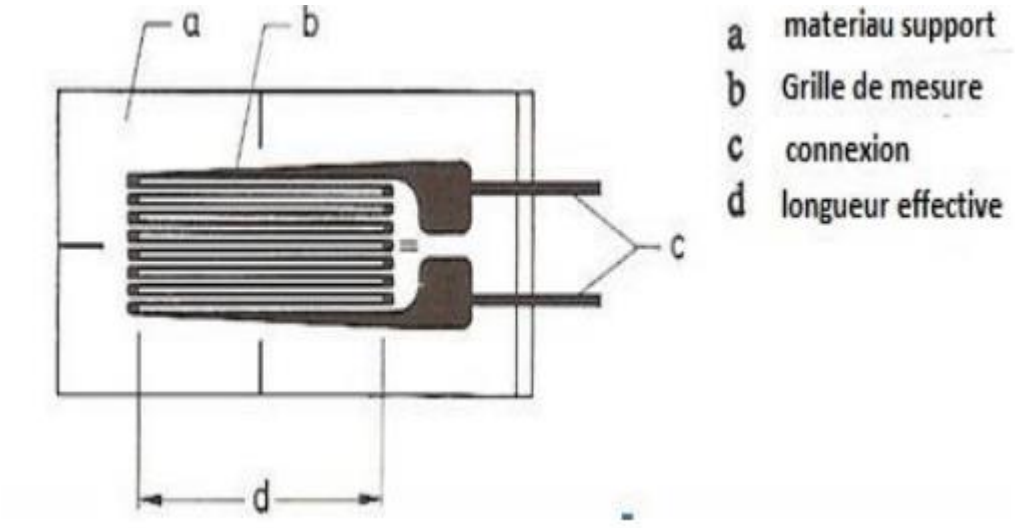



**Figure III.12:** *Strain gauge [JEA 01]*

**III.4.1.1.2. Principle**

The strain gauge is based on the principle of elongation or shortening of a resistive wire attached to the support that deforms [ISO 376]. When the deformation field is relatively uniform and uniaxial, the gauge is placed along the main orientation of this field, as shown in Figure III.13.a. When the deformation field is planar, it is necessary to place at least three gauges (Figure III.13.b.).

To measure the principal components of the deformations and their orientations by acting on the wire through tension or compression. This elongation or contraction of the wire leads to a change in its electrical resistance. We then measure this change in resistance between the resting state and the state under stress [DES 80]. Therefore, the wire is arranged in coils to act on the deformation simultaneously on several sections of the wire in order to amplify the change in electrical resistance [ISO 376].

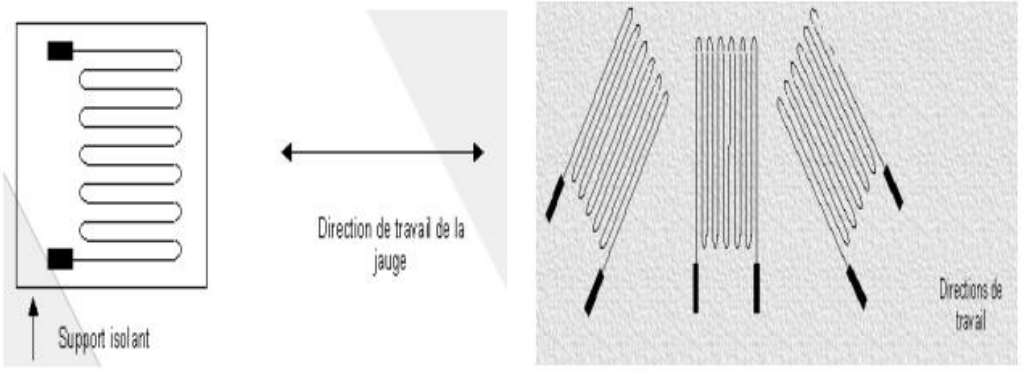



**Figure III.13:** Strain gauge arrangement [ISO 376]

(a) Principale orientation, (b) rosette

#### III.4.1.1.3. Scope of application

In general, strain gauges are bonded to the entire material that is considered relatively homogeneous (such as steel, concrete, wood, composites, aluminum, etc.), with gauges adapted to the type of material being tested. In the case of concrete structures, several ways of bonding to passive and active reinforcement of concrete are possible. [LEM 03]

The fields of application of strain gauges are vast, but the main ones are:

- ✓ Measurement of stress or strain states on various structures, on-site or in the laboratory. These measurements can be performed under static or dynamic conditions; Behavior under loading/fatigue verification (metallic and prestressed structures)/monitoring of structures;
- ✓ Determination of the strain diagram;
- ✓ Determination of the modulus of elasticity of a concrete or metal specimen under compression;
- ✓ Verification of the functioning of critical sections (under controlled loads or traffic);
- ✓ Detection of cracks on metal and concrete, in the case of cracks in prestressed concrete in association with a displacement sensor (notion of Strain gauge/sensor coupling);
- ✓ Release of stress on metal and concrete.

### III.4.1.2. Vibrating Wire Extensometer

#### III.4.1.2.1. Definition

The vibrating wire extensometer, also called a sonic gauge, is a contact-type extensometer used to measure the deformation of a structural element by determining the frequency variation of a wire stretched inside the sensor body. Reading vibrating wire gauges is done using a portable station or a data acquisition center. It can be performed in "damped" or "sustained" mode depending on the manufacturer [ALO 03].

This type of extensometer is frequently used for instrumentation: it offers a longer lifespan and metrological stability superior to strain gauges, and is less expensive than comparators

[NEV 00]. Note that the constant relating elongation to frequency strongly depends on the ambient temperature. For this reason, vibrating wires are often equipped with integrated temperature sensors that allow for measurement compensation.

**III.4.1.2.2. Principle**

The principle of the vibrating wire extensometer is based on the variation of the fundamental vibration frequency (often in the audible spectrum) of a stretched wire, which depends on the tension according to a determined law, Figure III.14. An elongation or a contraction result in an alteration of the wire tension, and therefore of the fundamental frequency.

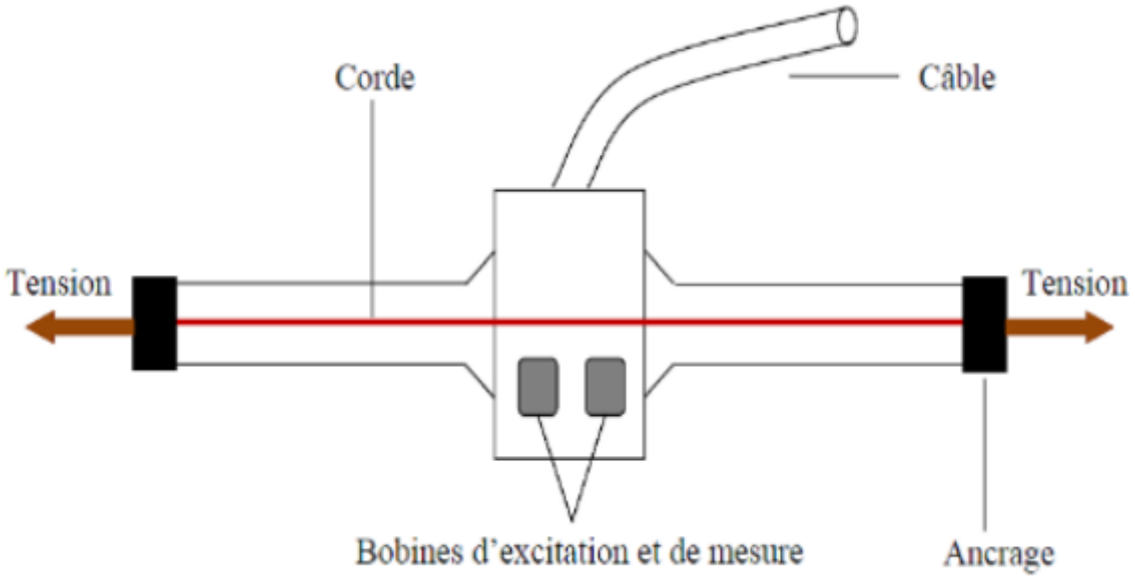



**Figure III.14:***Strain gauge [MEI 16]*

This physical principle allows for the measurement of the deformation of the structure to which the sensor is fixed at both ends. This type of extensometer has an electromagnet that both excites the oscillator and acts as a microphone for determining the frequency.

Therefore, when the distance between these two anchors varies over time due to deformation of the structure, the tension of the cord will change and cause a variation in its resonance frequency. If the variation in the oscillation frequency of the wire is known, it is possible to calculate the variation in deformation by combining the equations describing the oscillations of bodies with those of wave propagation. The oscillation of the cord is

achieved using the excitation coil, and the resonance frequency measurement is performed by the measurement coil [MEI 16].

### III.4.1.2.3. apparatus

The vibrating wire extensometers consist of a cylindrical tube with a head at each end. The vibrating cord slides freely inside the tube, is tensioned, and fixed to the two heads. In the usual case, there are two electromagnets in the middle of the tube: one allows for the excitation of the cord while the other measures its vibration frequency [ALO 03]. In the case of sensors embedded in concrete, the heads that anchor the sensor within the material follow the elongation or shortening of the concrete; this leads to an elongation or shortening of the cord whose vibration frequency is thus modified, as shown in Figure III.15.

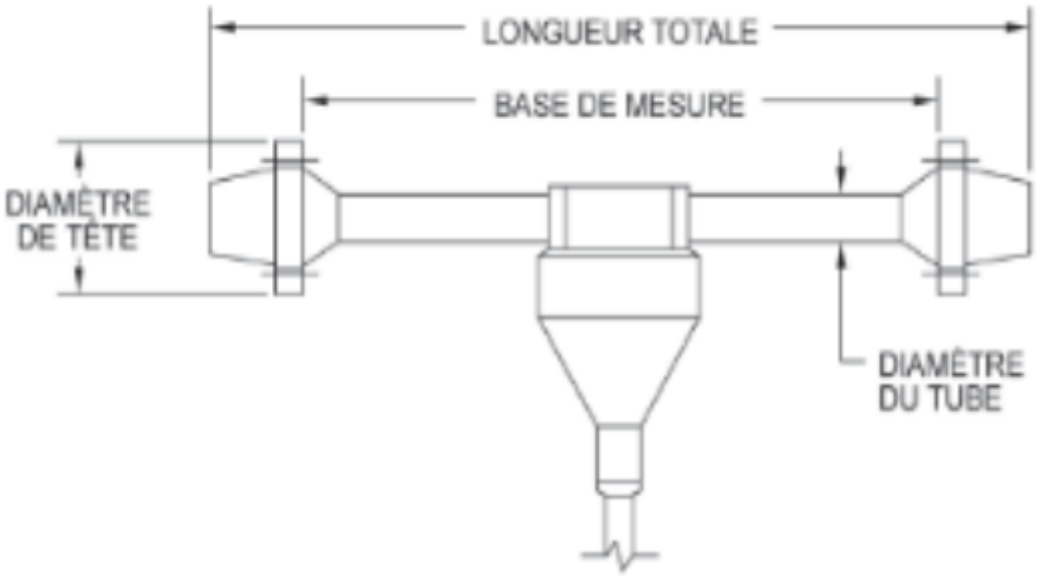



**Figure III.15:***Representations of a vibrating wire extensometer [MEI 16].*

### III.4.1.2.4. Application

The advantages of this type of extensometer are numerous for its fields of application:

- ✓ The electrical signal corresponding to the measurement of the resonance frequency of the vibrating rope can be transmitted without disturbance over a hundred meters of cable;
- ✓ Vibrating rope sensors have proven to be very reliable and robust over time (experience feedback over more than fifty years);
- ✓ Measurement frequencies can hardly be higher than one hertz due to the excitation of the rope, but this is not a problem in the field of civil engineering or hydrology, where the measured phenomena are slow.

## III.4.2. Non-contact measurements

### III.4.2.1. Optical fiber

#### III.4.2.1.1. Definition

The use of optical fiber sensors has become commonplace in civil engineering, typically for measuring strains at the core or surface of structural materials [FRA 86]. They are also designed for measuring temperatures, pressures, deformations, pH, fluid opacity, gas concentration, etc. The fiber optic measurement system consists of two elements: an interrogator and an optical fiber [LEC 17].

#### III.4.2.1.2. Principle of operation

The operation of the distributed fiber optic measurement system is as follows: the interrogator emits a light wave into the optical fiber [G652]. As the wave travels through the fiber, it is partially absorbed or diffused by the glass fiber. The diffused part of the wave is then analyzed by the interrogator. The signal received by the interrogator is a backscatter spectrum [DUC 15]. It includes several characteristic lines (Figure III.16):

- ✓ The Rayleigh line which represents the different density and composition fluctuations of silica;
- ✓ The Brillouin lines which are related to acousto-optic modes;
- ✓ The Raman lines which are related to molecular vibrations of silica.

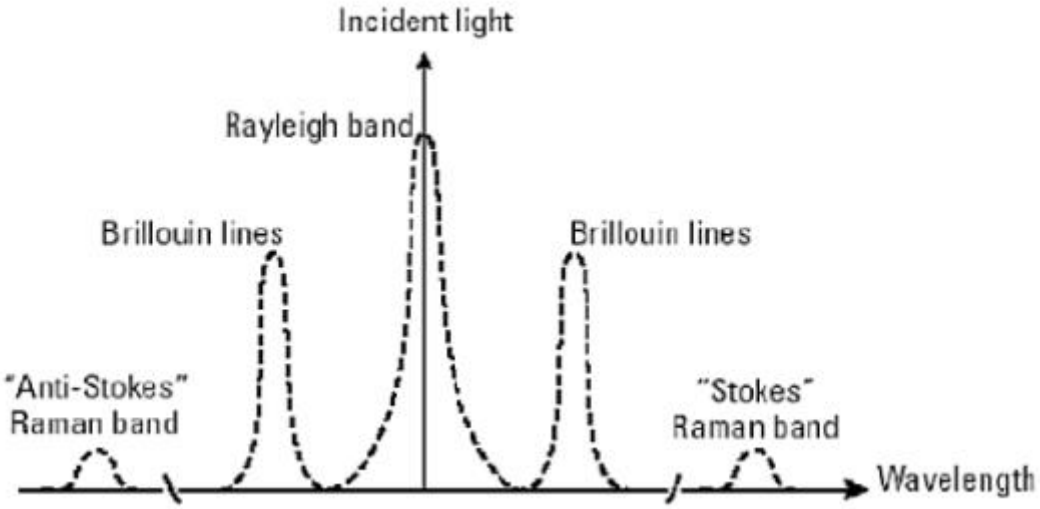



**Figure III.16:** *Backscattering spectrum of silica [DUC 15]*

### III.4.2.1.3. Apparatus

An optical fiber extensometer is a device comprising one or more fibers, allowing the collection of information representative of measured quantities without any energy input other than that of the observed phenomena and/or the light waves circulating in the fiber(s) [NF C 93-800-1991]. The deformation induced by the structural element on the sensor that is attached to it modifies the length of a reflective cavity called "Fabry-Pérot," and this length variation is measured by interferometry [QUA 98]. The tracking of the phase of the interfering signals allows for the determination of the deformation, as shown in Figure III.17.

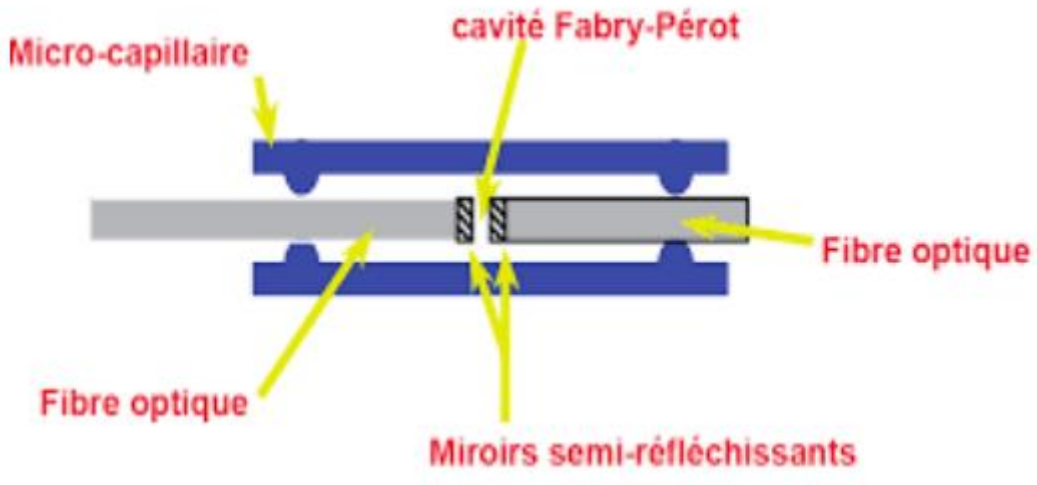
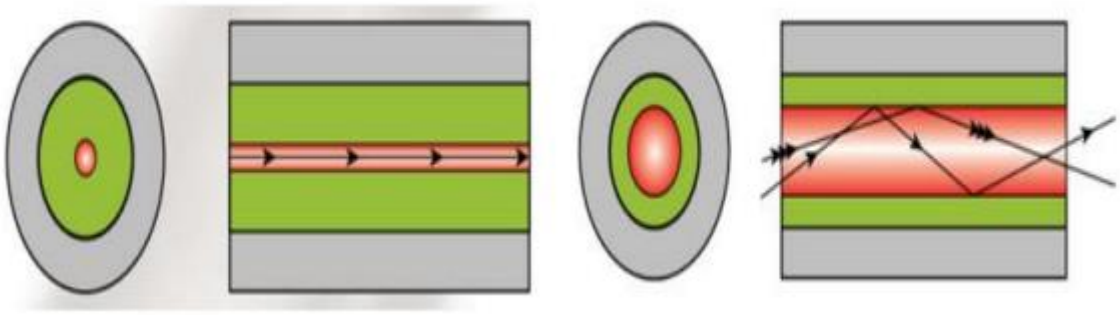



**Figure III.17:** *Special device for absolute measurement [QUI 00]*

The same optical fiber performs the point measurements and ensures the connection with the acquisition system. This technology is suitable for short-base deformation measurements [FER 15]. It does not offer multiplexing of measurement points on the same fiber [TOR 08]. There are two types of optical fibers (Figure III.18):

- ✓ **Single-mode fiber:** in this case, the fiber is called "single-mode" because, due to the very small size of the core (9 µm), there is only one mode of propagation of light. These devices are characterized by the use of a laser [DUP 02] and have only one possible path, Figure III.18.a.
- ✓ **Multimode fiber**: this type of fiber is called "multimode" because light propagates along several modes, meaning that it can follow multiple paths inside the core [TOR 08]. These devices use LEDs and have multiple possible paths for light rays within the fiber, Figure III.18.b.



**(a)** **(b)**

**Figure III.18:** *Types of optical fibers [TOR 08]*
*(a) Single-mode fiber; (b) Multimode fiber*

### III.4.2.1.4. Application

These sensors can be embedded in c Figure III.18: Types of optical fibers [TOR 08] (a) Single-mode fiber; (b) Multimode fiberoncrete or attached to the surface of structures made of concrete, masonry, wood, metal (in the latter case, welding is often prohibited and bolts or adhesives must be used, etc.) [PIL 04].

They allow to:

- ✓ measure the behavior under loading and monitor the structures;
- ✓ determine the deformation diagram;
- ✓ verify the operation of critical sections (under controlled loads or traffic).

### III.4.2.2. Laser Interferometry

#### III.4.2.2.1. Definition

One of the applications of lasers (Light Amplification by Stimulated Emission of Radiation) is in interferometry for measuring deformations with great precision [PUB 94, PIL 04]. Interferometry is a measurement technique that uses the phenomenon of interference of waves [QUA 98], as shown in Figure III.19.

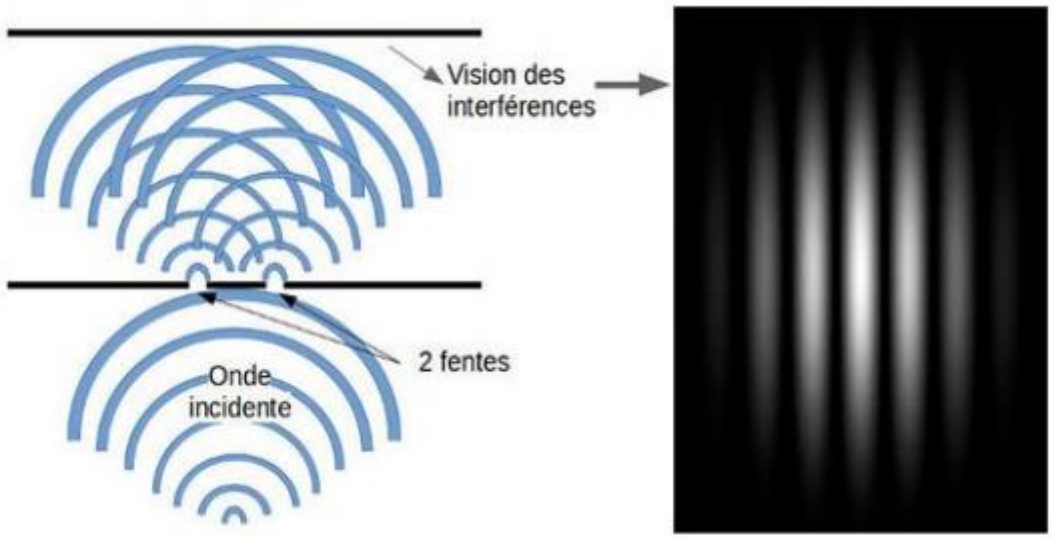



**Figure III.19:***the phenomenon of wave interference [QUA 98].*

Measurements can concern certain characteristics of the waves themselves (Figure III.20) and materials with which they interact [ISO, VDI 3441].

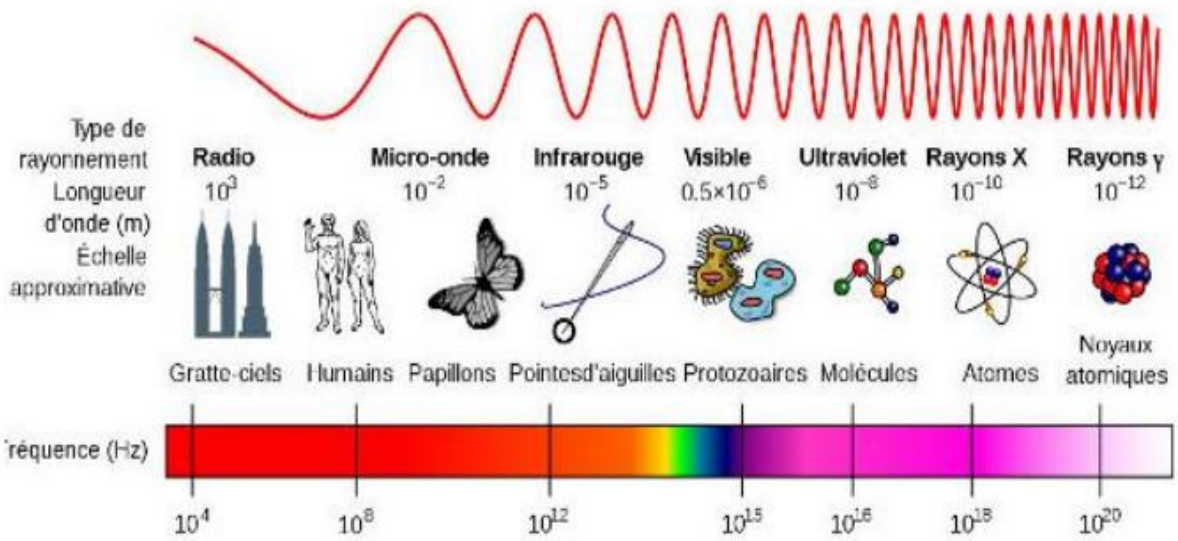



**Figure III.20:***Types of waves [FRA 86]*

Moreover, interferometry describes techniques that use light waves for studying deformation variations [DES 80]. This measurement interferometry is widely used for calibration and mechanical control in precision machining [PIL 04].

### III.4.2.2.2. Principle

Laser interferometry uses the principle of superposition to combine waves in such a way that the result of their combination represents a significant property that is the diagnosis of the original state of the waves [PUB 94]. This works because when two waves of the same frequency combine, the resulting intensity pattern is determined by the phase difference between the two waves: in-phase waves undergo constructive interference while out-of-phase waves undergo destructive interference, as shown in Figure III.21.

Note that waves that are not completely in-phase or completely out-of-phase will have an intermediate intensity pattern, which can be used to determine their relative phase difference [BAU 00]. Most interferometers use light or another form of electromagnetic wave.

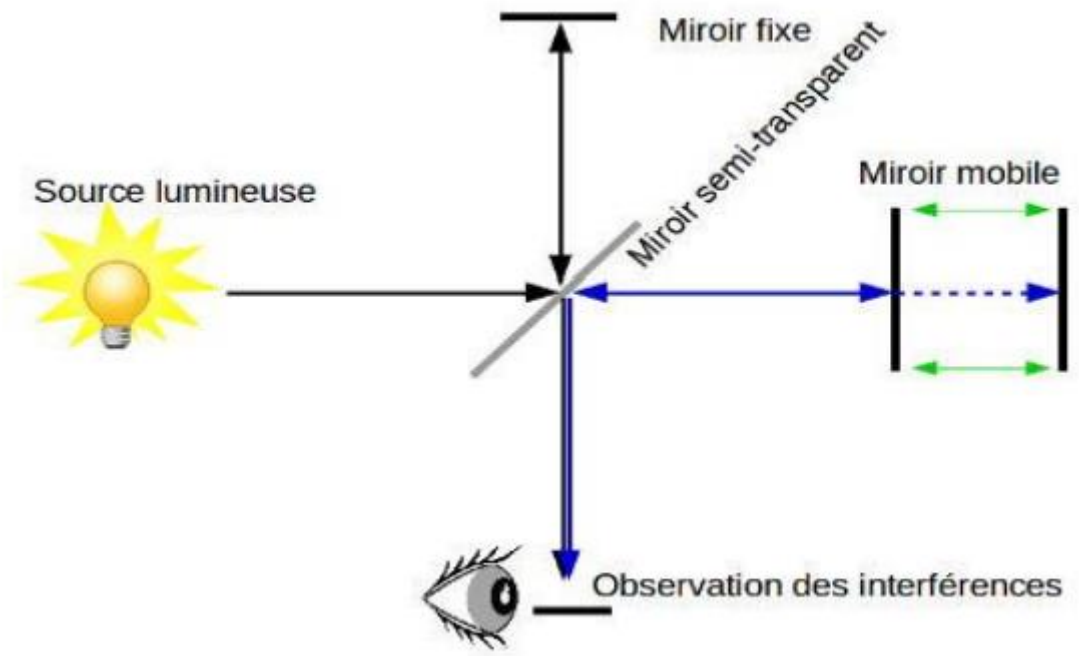



**Figure III.21:***Schematic diagram of laser interferometry [PUB 94]*

Typically, a single incoming beam of coherent light will be split into two identical beams by a beam splitter (a partially reflecting mirror) [GRE 99 and PUB 94]. Each of these beams travels on a different path, called a path, and they are recombined before arriving at a detector [BAU 00]. The path difference, the difference in distance traveled by each beam, creates a phase difference between them. It is this introduced phase difference that creates the interference pattern between the initially identical waves. If a single beam has been split along two paths, then the phase difference is the diagnostic for anything that changes the phase along the paths [PIL 04]. This could be a physical change in the length of the path itself or a change in the refractive index along the path.

### III.4.2.2.3. Apparatus

The laser interferometer is an apparatus that combines light waves (or non-visible waves) and analyzes the pattern of the combination of these coherent waves (same wavelength, same polarization) [QUA 98]. The test bench is given by Figure III.22. The interference fringes observed on the receiving plane come from the difference in distance that the light travels along the black or blue path [GRE 99]. In the case of a laser beam, we can notice that the spacing of the fringes is half the wavelength of the laser [QUA 98]. Furthermore,

during a displacement of the movable mirror, the fringes also move, so it is sufficient to count them to deduce the displacement of the mirror.

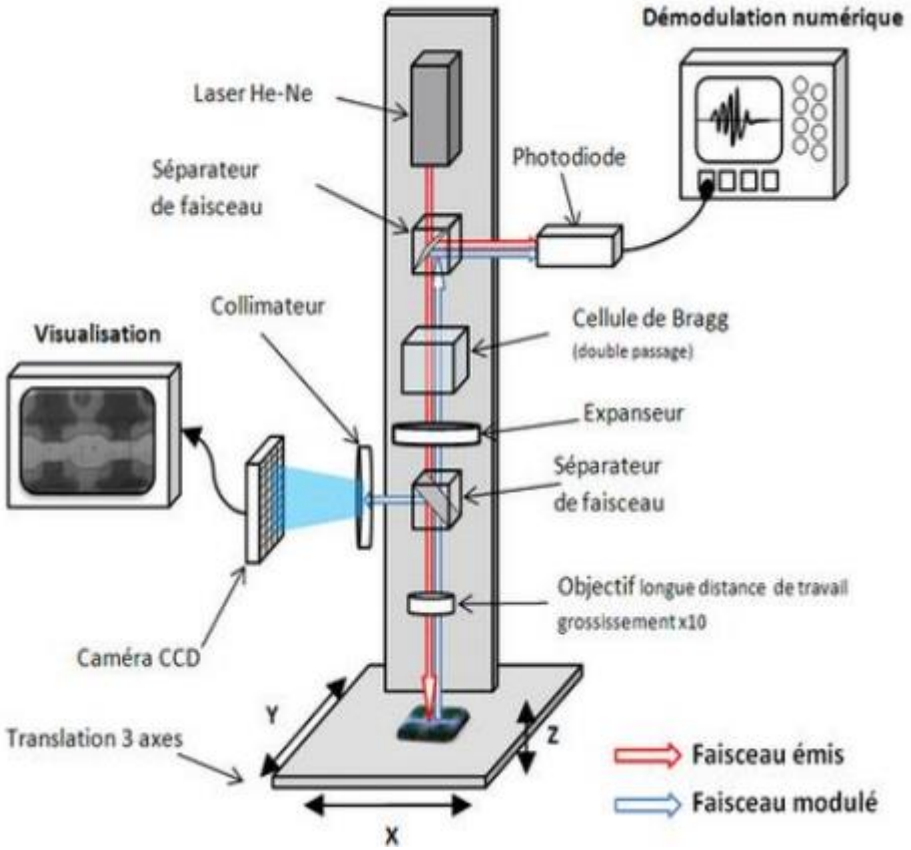



**Figure III.22:***Laser interferometry setup [PIL 04]*

# BIBLIOGRAPHIC REFERENCES

**[ACK 04] ACKER, P., J. TORRENTI, ET F. ULM,** «Comportement du béton au jeune âge», Traité Mécanique et ingénierie des matériaux, Hermès, 2004.

**[ACK 05] ACKER, P., TORRENTI, J., ULM, F** « Comportement du béton au jeune âge»,LAVOISIER, ISBN 2-7462-0980-2, 2005.

**[ALE 02] Alexander M. Korsunsky, Steve P. Collins, R. Alexander Owen, Mark R.Daymond, SaïdaAchtioui et Karen E.** «Fast residual stress mapping using energydispersivesynchrotron X-ray diffraction» , James 2002.

**[ALO 03] E. E. Alonso, A. LEDESMA,** "Andvances in Understanding Engineered ClayBarriers», ISBN 0415365449, 2003.

**[ALV 03] ALVANDI A.,** « Contribution à l'utilisation pratique de l'évaluation dynamiquepour la detection d'endommagement dans les ponts ». Doctoral thesis, National School of Bridges and Roads, Paris, 2003.

**[AND 90] ANDRE P**., « Vibrationsaléatoires et analyses spectrale», Polytechnical and University Presses of Romandy, Lausanne, 1990

**[ANR 89] A.N.R.T.** «Pratique du microscope électronique à balayage», Microanalyse parsonde électronique, Spectrométrie des rayons X» Microanalyse par sonde électronique»Quantitative aspects, 1989.

**[AVI 00] AVITABILE P.,** « Experimental modal analysis: a simple non-mathematicalpresentation », Sound and Vibration Magazine, 2000.

**[BAC 67] BACKUS G. GILBERT F.** « Numerical application of a formalism forgéophysical inverse problems », Geophysics.J.R.astr.Soc.,13, p:247 -276, 1967.

**[BAR 94] BAROGHEL-BOUNY, V.,** « Caractérisation des pâtes de ciment et des bétons.Méthodes, analyse, interprétation », Doctoral thesis, National School of Bridges and Roads, 1994.

**[BAR 92] BARON, J., J. OLLIVIER,** « La durabilité des bétons », National School of Bridges and Roads, Paris, 1992.

**[BAR 76] BARRON J., LESAGE R.,** « La composition du béton hydraulique, dulaboratoire au chantier», research report LPC, LCPC, Paris, No.64, december 1976.

**[BAR 82] BARON, J., ET R. SAUTEREY**, « Le béton hydraulique », The National School of Bridges and Roads, Paris, 1982

**[BAR 96] BARON, J., OLLIVIER, J.P,** « Les bétons, basses et données pour leurfabrication», Paris : Eyrolles Edition, 1996.

**[BAR 57] V. BARANOV.V,** « A New method for interpretationaeromagnetic maps andPseudo-gravimetric », Geophysical anomalies, XXII p:359-383, 1957.

**[BAR 53] BARANOV.V,** « Calcul du gradient vertical du champ de gravité ou du champmagnétique mesuré à la surface », soilGeophysicalProspectingp :171-191, 1953.

**[BAU 00]Baur Dirk R.,** « Pratique des lasers : mesure-holographie-expérimentation », 2000.

**[BEK 06] Beckhoff.B.**, Kanngießer.B, Langhoff.N, RWedell. N et Wolff. H, « Handbookof Practical X-Ray Fluorescence Analysis», Springer, ISBN 3-540-28603-9 ,2006

**[BER 09] BERTRAND C.,** «Vibrations des structures pour l'ingénieur et le technicien :théorie et applications», Ellipses, Lausanne, 2009.

**[BER 81]BERNARD-VIGNON Ph.** «Technique inverse appliquée à trois problèmes engéophysique»,Thesis, University of Montpellier, 1981.

**[BER 88] BERTHAUD Y.** « Mesure de l'endommagement du béton par une méthodeultrasonore ». Civil Engineering Thesis: University of Paris 6, 1988.

**[BHA 75]BHATTACHARYYA B.K., NAVOLIO M.E** «Digital convolution for computinggravity and magnetic anomalies due to arbitrary bodies», Geophysics,40 p:981-992-1975.

**[BHA 80]BHATTACHARYYA B.K** «A generalized multibody model for inversion ofmagnetic anomalies»,Geophysics 45, p:255-270 1980.

**[BUR 97] BURLION N.** « Compaction des bétons - Eléments de modélisation etcaractérisation expérimentale ». Civil Engineering Thesis: University of Paris 6, Cachan Normal Superior School, 1997.

**[CHA 92] CHABOCHE J.L.** «Une nouvelle condition unilatérale pour décrire lecomportement des matériaux avec dommage anisotrope. Paris : Comptes Rendus del'Académie des Sciences», T314, series II, 1992.

**[CHA 99] CHANVILLARD, G.,** « Le matériau béton : Connaissances générales», ALEASEDITEUR, ISBN 2-84301-012-8, 1999.

**[CHA 72] W.O.Charles ,** «The Scanning electron microscope. Pt 1, Theinstrument», Cambridge University Press, London, ISBN 0-521-08531-4, 1972.

**[CAR 03] CARACCIOLO R., GASPARETTO A., GIOVAGNONI M**., «An experimentaltechnique for complete dynamic characterization of an elastic material». Journal of Sound andVibration, 272:1013–1032, 2003.

**[CAU 88] P. Caussin, J. Nusinovici, D. W. Beard,** «Using digitized X-ray powderdiffraction scans as input for a new PC-AT Search/match program», Adv. X-Ray Anal. vol.31, pp 423–430, 1988.

**[COM 09] COMBES, B.,** « Vibrations des structures pour l'ingénieur et le technicien : Théorie et applications», Ellipses Edition Marketing S.A, ISBN 978-2-7298-2079-1, 2009.

**[COR 98] CORN S.,** « Simplification de modèles éléments finis de structures à comportement dynamique de poutre ». Doctoral thesis, Faculty of Science and Technology of the University of Franche-Comté, 1998

**[COR 06] A. Cornet and F. Hlawka.**« Propriétés et comportement des matériaux ».Ellipses, 2006.

**[CAS 04]CASTELLOTEA,M., ALONSOA, C., ANDRADEA, C., TURRILLASA, X.,**

**CAMPOC, J.,** «Composition and microstructural changes of cement pastes upon heating, asstudied by neutron diffraction», Cement and Concrete Research 34, 1633–1644. 2004

**[CHE 19] A. W. Hull, J. Am.** «A New Method of Chemical Analysis», Chem. Soc. 41, pp1168–1195, 1919.

**[CUL 56] B. D. Cullity,**« Elements of X-ray Diffraction», ed. Addison-Wesley, 1956

**[CHI 98] C. CHRISTIAN,** «La Microscopie électronique», 1998.

**[DES 80]Desmeules. E et Roynar.D,** « Optique ondulatoire cours avec exercices résolus »,Le Hir/Maruani series, Paris, Masson, 1997.

**[DOM 01] F. Dominique**. « Essais mécaniques et lois de comportement ». Hermès science,2001.

**[DUC 15] Jean Bernard Duchez. Etude du noircissement dans les fibres optiques dopées Ytterbium** : interaction entre photo -et radio-noircissement ». Optics, Nice Sophia Antipolis University, 2015.

**[DUP 04] DRURY J.C.,** «Ndt fundamentals: Ultrasonics, part 1. Basic principles of sound»,46:650–652, 2004.

**[DUP 07] DUPUY J., CORN S., IENNY P., DARIDON L**., « Comparaison de méthodesdynamiquesd'analyse du comportementmécanique de matériaux », 18th French Congress of Mechanics, Grenoble, 2007.

**[EBE 76] Eberhart J.-P.,** «Méthodes physiques d'étude des minéraux et des matériauxsolides», Paris, Doin, 1976.

**[EBE 97]J.-P. Eberhart,** «Analyse structurale et chimique des matériau» 2nd edition.,Dunod, ISBN 978-2100033676, 1997.

**[ELA 02]W. T. Elam, B. D. Ravel et J. R. Sieber,** « A new atomic database for X-rayspectroscopic calculations », Radiation Physics and Chemistry, vol. 63, p. 121–128, 2002.

**[EBE 76] J.-P. Eberhart,** « Méthodes physiques d'étude des minéraux et des matériaux », edition.Doineditors, 1976.

**[EBE 89] J.P.EBERHART:** «Analyse structurale et chimique des matériaux». Dunod 1989.

**[ERN 80] R.Ernst ,** «The early Development of Electron Lenses and ElectronMicroscopy», 1980 .

**[FER 15] P. Ferdinand et al.,DISCOMS**, « Distributed Sensing for Corium Monitoringand Safety», CANSMART 2015: International Conference on Smart Materials and Structures,2015.

**[FRA 95] D. Francois, A. Pineau, and A. Zaoui.** «Comportement Mécanique desMatériaux».Hermès, 1995.

**[FRA 86] Françon M.,** « Thèmes actuels en optique, Collection physique fondamentale etappliquée », Paris, Masson, 1986.

**[GAR 08] G. Gary.** «Comportement des métaux à grande vitesse de déformation».Modeling. In Techniques de l'Ingénieur, Mechanical Engineering Handbook, 2008.

**[GAR 02] GARNIER V., CHAIX J.F., CORNELOUP G**., « Caractérisation nondestructive du béton par analyse d'ondes ultrasonores », The Bulletin of the Laboratories of Bridges and Roads239 :105–118, 2002.

[GIB 72] GIBSON, «Network adjustment by least square alternative formulation andsolution by iteration». Geophysicsp:168-179 AL CHALABI M, 1972.

**[GMH 97] GMUR, T.,** « Dynamique des structures: Analyse modale numérique », Polytechnic and University Presses of Romandy(ISBN) 2-88074-333-8, 1997.

**[GOL 03] J.I. GOLDSTEIN; D.E. NEWBURY;P. ECHLIN; P.C. JOY;C. FIORI;E. LIFSHIN;** «Scanning electron microscopy an X-Ray microanalysis». Plenum Press 1981Kluwer Academic/Plenum Publisher 2003 (3rdedition)

**[GOL 92] J. Golstein, D.E. Newbury, D.C. Joy, C.E. Lyman, P. Echlin, E. Lifshin,L. Sawyer et J.R. Michael,** «Scanning electron Microscopy and X-Ray Microanalysis»,Plenum press, ISBN 0-306-44175-6, 1992.

**[GRE 99]Grenier Jean-Claude,** « Au rayon des superlasers », Géo. Hartmann Francis, Leslasers, « que sais-je ? », n° 1565, Paris, University Presses of France, 1974. Eugène Hecht, Physics, Paris, DeBoeckUniversity, 1999.

**[GUI 80] GUILLAUMIN :** « La pratique du microscope électronique à balayage enbiologie ». Masson, 1980.

**[HAN 38] J. D. Hannawalt, H. W. Rinn et L. K. Fevel,** «Chemical Analysis by X-RayDiffraction», Ind. Eng. Chem., Anal. Ed. 10, pp 457–512, 1938.

**[HEI 81] K.F.J. HEINRICH:** «Electron Beam X-Ray Microanalysis Van NostrandReinhold» 1981.

**[HEI 81] Kurt F. J. Heinrich,** « The physics of X-Ray », In Electron Beam X-Ray Microanalysis, Van Nostrand Reinhold Company, ISBN 978-0442232863, 1981.

**[HWA 03] HWANG H.Y., KIM C.,** «Damage detection in structures using a few frequencyresponse measurements», Journal of Sound and Vibration, 270:1–14, 2003.

**[LAN 96] J. Ian Ianford, D. Louër, «Powder diffraction»,** Rep. Prog. Phys. vol. 59, pp131–234, 1996.

**[LEC 17] Pierre LECOMTE,** Mesure haute température en environnement irradié par fibreOptique utilisant l'effet Raman », EDF R&D (Sylvain BLAIRON, Jose VEAU & ArnaudBARTHET), 2017

**[LEM 03] C. Lemaignan**. «La rupture des matériaux». EDP Sciences, 2003.

**[JAC 52] Jacques Despujols,** « Application de la spectrométrie des rayons X au dosage desfaibles teneurs de métaux dans les minerais », The Journal of Physics and Radium. Applied Physics, vol. 13, p. 31A-41A, 1952.

**[JAC 13] R. JACKY**, «Microscopieélectronique à balayage», Techniques of Engineering, in Analysis and Characterization.2013.

**[JEN 96] R. Jenkins, R. L. Snyder,** « Introduction to X-ray Powder Diffraction», ed. WileyInterscience, 1996.

**[JOH 65] G. G. Johnson Jr., V. Vand,** «A computerized powder identification system», Ind.Eng. Chem vol. 59, p19, 1965.

**[JOS 92] J.Goldstein, D. E. Newbury, P.Echlin, E.L.Charles, D.C. Joy, Eric Lifshin, L.C. Sawyer, J. R. Michael,** «Scanning Electron Microscopy and X-ray Microanalysis »,Springer Verlag, ISBN 978-0306472923 ,1992.

**[KEV 89] X.Kevex:** «Energy Dispersive X-Ray Microanalysis: An Introduction». KevexInstrument ; San Carlos U.S.A. 1989.

**[LAL 86] LALANNE M., BERTHIER P., DER HAGOPIAN J.,** «Mécanique desvibrations linéaires», Masson, Paris, 1986.

**[LAR.82] LAROIX R., FUENTES A., THONIER H.,** «Traité de béton armé», Eyrolles Edition, Paris, 1982.

**[LAR 88] LAROZE S.,** « Mécanique des structures, Tome 2, Poutres », EyrollesMassonEdition, 1988.

**[LIA 06] LIAO Y., WELLS V.,** «Estimation of complex modulus using wave coefficients»Journal of Sound and Vibration, 295:165–193, 2006.

**[MAS 96]Maso, J.,** «Interfacial transition zone in cement», Report of RILEM TC, E&FNSpon., London. 1996

**[MAU 78] F.MAURICE, L.MENY, R.TIXIER**: «Microanalyse et microscopie à balayage».EDP Sciences 1978.

**[MAZ 84] MAZARS J.,** «Application de la mécanique de l'endommagement aucomportement non linéaire et à la rupture du béton de structure », Doctoral thesis, Sciences : University of Paris 6 E.N.Sof Cachan, 1984."

**[MED 10]Meddah, M. S., S. Zitouni, et S. Belâabes**, «Effect of content and particle sizedistribution of coarse aggregate on the compressive strength of concrete», Construction andBuilding Materials, 24, no. 4(4), 505-512, 2010.

**[MEI 16] Bingqing MEI**, « Optimisation des couplages magnéto-mécaniquesd'extensomètres à corde vibrante pour le suivi du vieillissement de constructionsstratégiques». Doctoral thesis: Mechanics, Acoustics, Electronics and Robotics Sciences of Paris: Pierre and Marie Curie University. 156 p. 2016

**[MEN 99] MENICHETTI.V et GUILLEN.A,** « Techniques inverses engravimétrie etmagnétismeApplication to the southern termination of the Paris Basin anomaly, 1999.

**[MIC 97] Michael T. Postek,** «The Scanning Electron Microscope in Handbook of ChargedParticle Optics», CRC Press, Université du Maryland, ISBN 0-8493-2513-7, 1997.

**[MON 94] P. Montmintonnet.** « Le laminage, aspects mécaniques ». ENSMP/CEMEF,1993-1994.

**[MOU 03] MOUNANGA P.,** «Etude expérimentale du comportement de pâtes de ciment autrès jeune âge : hydratation, retraits, propriétés thermophysiques », Doctoral thesis, University of Nantes, 2003.

**[MUR 03] M.MURATet G.DIDIER ,** « Application de l'analyse thermique différentielle audosage semi-quantitatif du quartz dans les argiles»,Bull, French company,pp 131-151, 2003.

**[NAG 66]NAGY. D** «The gravitational attraction of a right rectangular prism»,Geophysics31, p :362-371 ,1966.

**[NEC 04] NECHAD H.,** « Evaluation de l'endommagement et de la rupture de matériauxhétérogènes par ultrasons et émission acoustique : estimation de la durée de vie restante »,Doctoral thesis, National Institute of Applied Sciences of Lyon, 2004.

[NF EN 13284] NF EN 13284-1 Novembre 2017 Émissions de sources fixes – Déterminationde faibles concentrations en masse de poussières - Partie 1 : méthode gravimétrique manuelle.

[NF X44-052] NF X44-052 Mai 2002 Émissions de sources fixes - Détermination de fortesconcentrations massiques de poussières - Méthode gravimétrique manuelle.

[NF EN 13284-2] NF EN 13284-2 Novembre 2017 Émissions de sources fixes - Détermination de faibles concentrations en masse de poussières - Partie 2 : assurance qualitédes systèmes de mesurage automatisés.

**[NIC 66] M. C. Nichols,** «A Fortran II program for the identification of X-ray powderdiffraction patterns», UCRL-70078, 1966

**[NEV 87] NEVILLE A.M., BROOKS J.J.,** « Concrete of Technology», London: LongmanEdition, 1987.

**[NEV 90] NEVILLE A.M.** Properties of concrete. London: Longman scientific andtechnical, 1990.

**[NEV 95] NEVILLE A.M.,** « Properties of Concrete», London: LongmanEdition, 4th,1995.

**[NEV 00] NEVILLE A.M,** «Propriétés des bétons », Interuniversity Research Centre on Concrete, Eyrolles, 2000.

**[OWO 02] OWOLABI G.M., SWAMIDAS A.S.J. et SESHADRI R.,** «Crack detection inbeams using changes in frequencies and amplitude of frequency response functions», Journal of Sound and Vibration, 265:1–22, 2002.

**[PAN 06] PANET.I, DIAMENT.M**, « La gravité observée depuis l'espace », forSciences, november 2006, p. 46-53.

**[PAS 87] M. PASSEREL :** «Analyse X en dispersion d'énergie. Microscopie à balayage».Fac.Des Sc. & Tech., Marseille 1987.

**[PED 77] PEDERSON.B,** «Interprétation of potentiel field data A Généralized Inverse»,Approch Geophysical prospecting, june 1977.

**[PIL 04] Pilloud. J,** « Cours de physique, optique, » Lycée Denis-de-Rougemont, 2004

**[PIN 03] PINTELON R., GUILLAUME P., VANLANDUIT S., BELDER K.D.,ROLAIN Y.**, «Identification of young's modulus from broadband modal analysisexperiments», Mechanical systems and signal processing, 18:699–726, 2003.

**[PIR 93] PIRANDA J.**, « Cours des Dynamiques des structure », ENSMM, Besançon, 1993.

**[PIR 01] PIRANDA J.,** « Analyse modale expérimentale », Mesures et Contrôle, Techniquesde l'ingénieur, 2001.

**[QUA 98]Quaranta Lucien,** Introduction à l'optique cours avec exercices corrigés,Enseignement de la physique, Paris, Masson, 1998.

**[REY 05] REYNOUARD J-M., PIJAUDIER-CABOT G.,** «Comportementmécanique dubéton », Mechanics and Materials Engineering, Germ Sciences, 2005.

**[PUB 94]Publitronic/Elektor. Dändliker R.,** Les lasers, principe et fonctionnement, Lausanne,Presses polytechniques et universitaires romandes, 1996. De La Taille Renaud, Comment çamarche, le laser, Science & Vie, n°919, 1994.

**[RIL 72] RILEM**, «Recommandations RILEM CPC8, Modulus of elasticity of concrete incompression», Materials and Structures, 6(30), 507-512.,1972.

**[ROC 71] ROCARD Y.,** «Dynamique générale des vibrations », Masson, 4$^{th}$edition, 1971.

**[ROS 84] ROSSLER M.,** «Vibrations des Systems mécaniques: méthodes analytiques etapplications », Masson, Paris,1984.

**[ROS 09] ROSEA, M.,** « Vibrations des systèmes mécaniques : méthodes analytiques etapplications», MASSON editor, ISBN 2-225-80205-X, 1984.

**[SCH 82] W. N. Schreiner, C. Surdukowski, R. Jenkins, J. Appl. Crystallogr,** «A newminicomputer search/match/identify program for qualitative phase analysis with the powderdiffractometer», vol. 15, pp 513–523, 1982

**[SCI 02] SCIM.A,** « Magnetic and gravity model of the Morokweng impact structure », onDirect Sciences, 2002.

**[TAL 60]TALWANI .M , EWING .M** «Rapid computation of gravitational attraction ofthree dimensional bodies of arbitrary shape»,Geophysics 25,p:203-225, 1960.

**[RON 99] J.Ron ,** «Fluorescence spectrometry» 2$^{nd}$ ed., New York, WileyIntersciences, 1999.

**[RUS 78] J. Ruste,** « Spectromètre de rayons X », Microanalysis and Scanning Microscopy, Les Éditions de la Physique, 1978.

**[THO 97] THOMAS G.,** «Dynamique des structures: analysemodale numérique», Polytechnic and University Press of Lausanne, 1997.

**[THY 97] THYAGARAJAN S. K., SCHULZ M.J., PAI P.F**., «Detecting structural damageusing frequency response functions», Journal of Sound and Vibration, 210:162–170, 1997.

**[TIM 69] TIMOSHENKO S., YOUNG D.H.,** « Dynamique Supérieure », PolytechnicBookstore Ch. Bérenger, 1969.

**[TOR 96] TORRENTI j.-M.,** «Comportement mécanique du béton : bilan de six années derecherche», LPC research report OA23, 1996.

**[TOR 08] B.Tortech. ,** « Effets des radiations sur des fibres optiques dopées erbium :influence de la composition ». Physics, Jean Monnet University - Saint-Etienne, 2008.

**[DUP 02] Dupont J.,** « Comportements de Capteurs à Fibres Optiques, noyés ou fixés ensurface d'ouvrages en béton », ENPCthesis, 2002.

**[QUI 00] QUIRION M., BALLIVY G.,** « Application des capteurs à fibre optique FabryPérot pour le suivi à long terme de structures en béton », Bridges and RoadsLaboratories Bulletin - No. 225 - Reference 4296, March-April. pp. 75-88, 2000.

**[BEL 93] BELLEVILLE C., DUPLAIN G. (1993),** White-light interferometric multimodefiber-optic strain sensor, Optics letters – Volume 18 – N° 1, pp. 78-80, 1993.