

# Materials Characterization Techniques: Applications and Features

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*(Received 17 April 2009; accepted 26 June 2009)*

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After the second half of the twentieth century there was a great growth in the understanding of the microstructure of materials, and it was only possible through the discovery of new techniques for characterization of materials. Today there is a number of direct and indirect techniques to characterize samples such as x-ray diffraction, atomic absorption, thermal analysis, electron microscopy and heating. This article presents the characteristics of these techniques, the types, extent and applications.

Keywords: characterianon od materials, chemical analysis, mineralogical analysis, thermal analysis.

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## 1. INTRODUCTION

The evolution of the seventeenth century scientific and technological evolution of industry in the late nineteenth century not reflected in changes in understanding the structure of materials. Only in 1911/12 that the techniques of X-ray diffraction allowed the determination of the crystalline structure of various materials and other indirect techniques such as dilatometry and thermal analysis. The metallurgist could later characterize the changes of phases and microstructure of metals and the correlation with the properties came naturally. Electron microscopy also allowed better resolution of the microstructures and the observation of details which were not observed in optical microscopes.

In this paper, firstly, the traditional techniques of thermal analysis are decrypting, such as: (i) thermogravimetry (TG/DTG), (ii) differential scanning calorimetry (DSC), (iii) differential thermal analysis (DTA), (iv) mechanical dynamical analysis (DMA) (iv) termomechanical analysis (TMA), (v) evolved gas detection and evolved gas analysis. The next step is the study of mineralogical analysis by x-ray diffraction. Finally, it will be presented chemical analysis techniques: (i) x-ray fluorescence (FRX) and (ii) analytical atomic absorption spectroscopy.

## 2. METHODOLOGY

The methodology used in this paper consisted in a planning to obtain the better layout for the text disposition. Afterwards, it was realized a bibliography research about analytical chemical instruments and techniques for materials characterization. The objective was chiefly the study of the application and features of the methods to characterize materials without entering in details about the equipments. The study was made in interdisciplinary way covering materials engineering, chemical analysis and mineralogy.

## 3. RESULTS AND DISCUSSIONS

### 3.1 Thermal Analysis

The term thermal analysis (TA) is frequently used to describe analytical experimental techniques which investigate the behavior of a sample as a function of temperature [1].

The increasing use of thermal analysis both in academic as industrial applications has promoted in different areas: organic, inorganic, petrochemical, pharmaceutical, natural products, construction materials, coatings, catalysis, glass, ceramic, food, grease, surfactants, and composites polymers [2].

Below are described the most common thermal analysis, that include: Differential thermal analysis (DTA), differential scanning calorimetry (DSC), thermogravimetry (TG/DTG), Thermomechanical analysis (TMA), evolved gas detection and evolved gas analysis (EGD/EGA).

### 3.1.2 *Differential thermal Analysis (DTA)*

Differential thermal analysis (DTA) is a thermal technique in which the temperature of a sample, compared with the temperature of a thermally inert material, is recorded as a function of the sample, inert material or furnace temperature as the sample is heated or cooled at a uniform temperature [3]

The sample holder assembly is placed in the centre of the furnace. One holder is filled with the sample and the other with an inert referential material, such as alfa-alumina. Thermocouples inserted in each holder measure the temperature difference between the sample and the reference as the temperature of the furnace is controlled by a temperature programmer [1].

A DTA curve plots the temperature difference as a function of temperature (scanning mode) or time (isothermal mode). During a phase transition the programmed temperature ramp cannot be maintained owing to heat absorption or emission by the sample [1].

Through the (DTA) curves is possible to obtain the information about the structure and ordination of that. Then, any changes give precious information about the material [2].

Temperatures changes of the sample are occasioned by transitions or enthalpy reactions (endothermic or exothermic) due phase changes, melting, change of crystalline structure, vaporization, decomposition reactions, oxidation, reduction and others chemical reactions [2].

Generally phase transitions, reductions and some decomposition reactions generate endothermic effects while crystallization, oxidation and others reactions of decomposition generate exothermic effects [2].

The numbers and form of the peaks endothermic or exothermic in function of the temperature identify quantitatively and qualitatively a determined substance. As the area of the peak is proportional heat change involved, the technique is useful to semi quantitative determinations, and some cases, quantitative of heat reaction.

This method has been applied with success, for example, to determination for maximum nucleation in glasses and has several advantages compared to conventional method. In complement, important parameters like enthalpy and activation energy for crystallization are investigated using (DTA) [4].

### 3.1.3 *Differential scanning calorimetry (DSC)*

Differential scanning calorimetry, (DSC), is a technique which combines the ease of measurements of heating and cooling curves, with the quantitative features of calorimetry. Temperature is measurement continuously, and a differential technique is used to assess the heat flow into the sample and to equalize incidental heat gains and losses between reference and sample. Calorimetry is never a direct determination of the heat content [3].

A (DSC) curve is similar to a (DTA) curve, except in the Y-axis nomenclature (heat flux instead of temperature difference). As the (DTA) technical, the (DSC) curve is directly proportional to enthalpy variation [2].

The degradation, the oxidation and crystallization of polymers can be studied using (DSC) in order exothermal. The transition from a solid to a brittle rubber and glass transition are some examples beyond the determination of specific heat, degree of crystallization, melting temperature of crystal, etc. [2, 3].

### 3.1.4 Thermogravimetry (TG)

Thermogravimetry (TG) is the branch of thermal analysis which examines the mass change of a sample as a function of temperature in the scanning mode or as a function of time in the isothermal mode. Not all thermal events bring about a change in the mass of a sample (for example melting, crystallization or glass transition), but there are some very important exceptions which include desorption, absorption, sublimation, vaporization, oxidation, reduction and decomposition [1].

(TG) is used to characterize the decomposition and thermal stability of materials under a variety of conditions and to examine the kinetics of the physicochemical processes occurring in the sample [1].

The sample is placed in a thermal microbalance. It established a program of heating at a rate pre-determined then the change in weight of the sample is detected.

Mass changes occur when the sample loses material in one of several different ways or reacts with surrounding atmosphere. This produces steps in the (TGA) curve or peaks in the (DTG) curve. Different effects can cause a sample to lose, or even gain, mass and so produce steps in the (TGA) curve [5].

Nowadays, mainly compensation balances are used [5]. The principal elements of a microbalance are an electronic microbalance, a furnace, a temperature programmer and an instrument for simultaneously recording the outputs from these devices [1].

(TG) curves are from empiric nature, cause depends mainly of the parameter sample and type of heating used. Then, the difficulties to made comparisons between the laboratories diverse. But, this effect have been circumvented with new thermobalances [2].

### 3.1.5 Thermomechanical Analysis (TMA)

The variables of state for thermomechanical analysis are deformation (strain) and stress. Temperature is controlled through a heater and coolant at the bottom. Atmosphere control is possible through the sample tube. The heavy black probe measures the position of the sample surface with a linearly variable differential transformer, (LVDT) [6].

(TMA) is frequently used to measure temperature of glass transition (T<sub>g</sub>) through expansion mode or penetration. The expansion mode also provides dates of thermal expansion linear under and above of the (TG) [2].

This technique is similar to a (DSC) method. Is possible to obtain through (TMA) information about the expansion or thermal contraction which the sample undergoes when is submitted of a time variation or temperature in the constant stain [3].

### 3.1.6 Evolved gas detection and evolved gas analysis (EGD) and (EGA)

The detection or analysis of the gases evolved during a chemical reaction, as a function of the temperature, constitute the techniques of thermal analysis called evolved gas detection (EGD) and evolved gas analysis (EGA) respectively [3].

(EGA) was preceded by evolved gas detection (EGD), which merely detected the presence of evolved gases [1].

The international confederation of thermal analysis (ICTA) nomenclature committee (17) defined (EGA) and (EGD) as:

*Evolved gas detection (EGD):* Evolved gas detection is a technique of detecting whether or not a volatile product is formed during thermal analysis.

*Evolved gas analysis (EGA):* Evolved gas analysis is a technique of determine the nature and amount of volatile product or products formed during thermal analysis.

This techniques are almost always used in conjunction with other thermal analysis or multiple techniques such as: (TG)-(EGD), (TG)-(DTA)-(EGD)-(MS), (EGD)-EGA, and others [1, 3].

Evolved gases can be sampled either continuously or intermittently. The most common methods of EGA, mass spectroscopy (MS) and Fourier transform infrared (FTIR) spectroscopy, continuously monitor the purge gas is collected over a given time or temperature interval and subsequently analyzed [1].

### 3.2 Microscopy Analysis

Microscopy analysis usually are performed by optical microscopic or electron microscopic through transmitted light or reflected light.

It's used microscopy analysis both for the qualitative determination and quantitative determination. Therefore, actually, there is emphasis decreasing in the light transmitted techniques.

#### 3.2.1 Optical Microscopy

The optical microscope is the primary toll for the morphological characterization of microstructure in science, engineering and medicine [7].

The geologist works primarily in transmission, polishing his mineralogical specimens down to a thickness of less than 50  $\mu\text{m}$  and mounting them on transparent glass slides using *polarized light* is the most frequent source of contrast, and provides information in the optical properties and spatial orientation of the crystalline phases which are present in the sample [7].

Metallurgical samples for metallographic examination were originally prepared as thin slices, by using the methodology developed earlier for mineralogical specimens, in 1964 by Henry Sorby. However, the presence of conduction electrons renders metals opaque to visible light and all metallurgical samples must be examined in reflection [7].

The contrast of the image is resulted of the light reflective light in several regions of the microstructure, since the system consists basically for the illumination system and lenses.

Polymers and plastics can be imaged in either reflection or transmission, but the amorphous, glassy phases give poor contrast. Filled plastics and polymer matrix composites can be examined in reflection, crystalline polymer phases, etc.

However, this technique depends generally of any previous knowledge about the phases that can to be finds or final composition or structural previous analysis as x-ray diffraction. Another limitation is the maximum increase achieved that is about 2000 times.

#### 3.2.2 Electron Microscopy

The electron microscope has extended the resolution available for morphological studies from that dictated by wavelength of visible light to dimensions which are well into the range to image the lattice planes in any crystal structure that is from of the order of 0.3  $\mu\text{m}$  to of order of 0.15 nm [7].

The transmission electron microscope is in many ways analogous to a transmission optical microscope – but usually “upside down”, in the sense that source of the electron beam is at the top the microscope “column” while the recording system is at the bottom [7].

In practice way, the surface of a sample can be: broken, polished, polished and attacked depending of the researcher intention.

The principal advantage of the electronic microscopy is the opportunity to see sub-micrometric details of the microstructure. On the other hand, electronic microscopy not is applied thick sections, excepted to obtain silhouettes of the material geometry. Thus, for the examination by electronic transmission, is necessary to prepare very thin sections ( $< 1\mu$ ).

### 3.3. Mineralogical Analysis

#### 3.3.1 X-ray spectroscopy (DRX)

X-ray diffraction is a tool for the investigation of the fine structure of matter. At first, x-ray diffraction was used only for the determination of crystal structure. Later on, however, other uses were developed, and today the method is applied not only to structure determination, but to such diverse problems as chemical analysis and stress measurement to the study of phase equilibria and measurement of particle size, to the determination of the orientation of one crystal or the ensemble of orientations in polycrystalline aggregate [8].

The x-ray spectroscopy was, for several years, used chiefly for determination of the heavier elements, particularly those such as niobium, tantalum and the rare earths whose determination by chemical or other methods was a difficult and unreliable venture [9].

Nowadays, is used several techniques for diffraction as; x-rays diffraction, diffraction of electrons and neutrons. The X-ray diffraction is suitable for the study of solids and liquid crystals and electron diffraction is used in the study of gases and vapors and surface coatings. X-rays, as well as light and ultraviolet radiation are electromagnetic waves whose wave length ranges from 0.1 to 100 Å and are usually as a form of radiation possession of great power of penetration [8].

About 70 elements are easily identified by spectral methods. Those that are more difficult to identify include the gases and few nonmetals, primarily because sensitive lines lie in the short ultraviolet portion of the spectrum that is difficult to observe [10].

The qualitative sample can be placed in the electrode in several different forms. If the sample is a powder, it can be conveniently placed in the cup electrode without weighting. Small metal filings also can be placed in the cup and the solvent evaporated [10].

In the powder method, the crystal to be examined is reduced to a very fine powder and placed in a beam of monochromatic X-rays. Each particle of the powder is a tiny crystal, or assemblage of smaller crystals, oriented at random with respect to the incident beam [8].

Usually measurements about 0.05 to 0.10 Å suffice, since the spectroscopist usually relies on the identification of three or four spectral lines to prove the presence of an element in the analytical sample. Use also is made of unknown spectra of elements to compare with the unknown sample. This technique does not require measurements of wave lengths of spectral lines. If it necessary to measure line wavelength adopted in 1960, is the red line of krypton-84, with a wavelength assignment of 6057.802106 Å. This standard is not especially useful for qualitative analysis but is the basis for wavelength tables for iron lines, which can serve as a secondary standard [7].

In practice, for example, among de the various techniques for materials characterization (DRX) is the most appropriate for the determination of crystalline phases present in ceramics. It is possible because in most solids (crystals), the atoms are ordered in crystalline planes separated by distances itself from the same order of the x-ray wavelength magnitude [11].

### 3.4 Chemical Analysis

#### 3.4.1 X-ray fluorescence spectroscopy (FRX)

(FRX) is used to determine the chemical composition of materials in non destructive form. This method is based in the principle of the absorption for the material which causes atomic excitation that premises secondary radiation called x-ray fluorescence [12].

The emissive rays have a characteristic wavelength well defined for each chemical element that constitute the irradiated material (chemical qualitative analysis), being possible through comparison to patterns sample, to establish proportion of the chemical elements presents (chemical quantitative and qualitative analysis).

This technique intended for analysis samples, solids, liquids or gaseous, detecting elements with higher atomic number 11 until urane (last natural element present in the periodic table). [12].

In this method a sample suitable prepared is irradiated by an x-ray bunch. The bunch can to dislocate a electron of the layer more interns of the atom; to substitute the lost electron, one other electron can to jump from one of the external layer and, this madding to release energy in x-ray fluorescence, emissived in wavelengths which are characteristics each element.

#### 3.4.2 Analytical atomic absorption spectroscopy (AAS)

The absorption of energy by ground state atoms in the gaseous state is the basis of atomic absorption spectroscopy. The absorption is specific to each element, another element absorbs this wavelength.

(AAS) used this phenomenon for the quantitative determination of metals, semimetals and some non-metals in a wide range of the samples as; biological samples, metallurgical, pharmaceutical, geological and weather.

The most common detector for atomic absorption spectroscopy is the photomultiplier tube, although other photosensitive devices have been used, such as the barrier layer photocell, the vacuum phototube, and photographic emulsions [7].

The sample is usually in liquid form, although recent developments with carbon rods and tantalum boats have possible the use of solid samples and the method consists in determinate the presence and amount of metals in a chemical solution.

#### 4. FINAL CONSIDERATIONS

The present review showed the most common analytics techniques applied in the materials characterization. However, there wasn't how to explore more the theme because the limited space available, nevertheless it was possible remembers the features and applications of each technique.

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